



BDG SYNTHESIS

Certificate of Analysis

This material is a research-grade material prepared by custom synthesis. The quantity available is limited, 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 research-grade materials. Research 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.

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

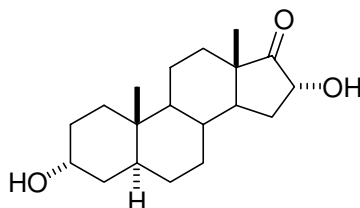
Barry Dent

Barry R. Dent, PhD, Director
7 August 2003

Name: 16 α -Hydroxyandrosterone

CAS Number: 14167-49-8

Structure:



Molecular Weight: C₁₉H₃₀O₃ = 306.44

Lot Number: BDG 4388.5

Appearance: White, crystalline solid

Purity by HPLC: 98.9 %

Expiry Date: 7 August 2004

Because of the small amount of material available it is not possible to perform formal storage stability studies. This expiry date is assigned from experience gained with the material in the laboratory and/or on storage.

Storage and Handling:

Temperature: ambient laboratory temperature; may be refrigerated.

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:**Source of Material**

The material was made by an unambiguous synthetic route, using literature procedures where possible; starting materials were purchased from reputable sources and all intermediates were checked for identity by NMR.

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 ethyl acetate 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.

High-resolution mass spectrum (FAB+): found m/z 307.2268. $C_{19}H_{31}O_3$ $[M+H]^+$ requires m/z 307.2273. The deviation of 1.8 ppm is within normally accepted limits for the establishment of identity by HRMS.

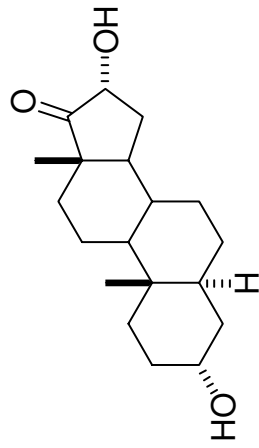
HPLC: A sharp, symmetrical peak is observed (98.9 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis: Found: C 74.42, H 9.85 %
 $C_{19}H_{30}O_3$ requires: C 74.47, H 9.87 %

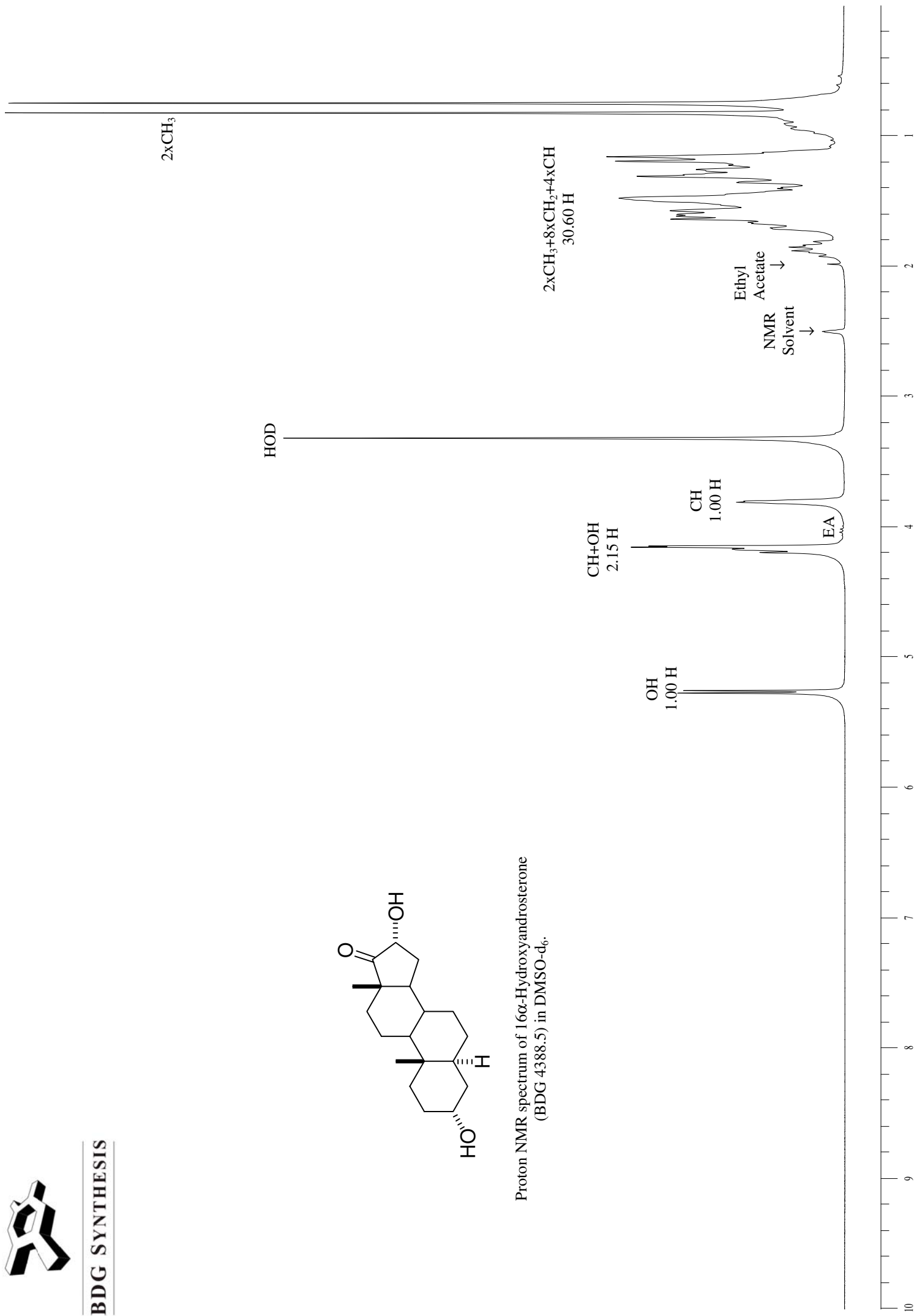
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).



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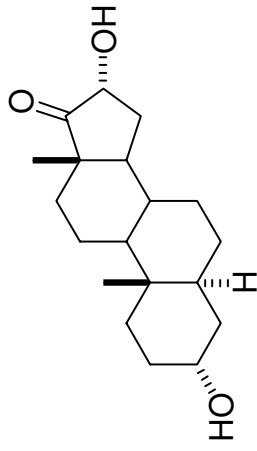


Proton NMR spectrum of 16 α -Hydroxyandrosterone (BDG 4388.5) in DMSO-d₆.





BDG SYNTHESIS



Carbon-13 NMR spectrum of 16 α -Hydroxyandrosterone (BDG 4388.5) in DMSO-d₆.

NMR
Solvent

4xCH₂+CH+C

CH

2xCH₃

3xCH₂

CH₂

CH

CH

C

CH

CH

C=O

225

200

175

150

125

100

75

50

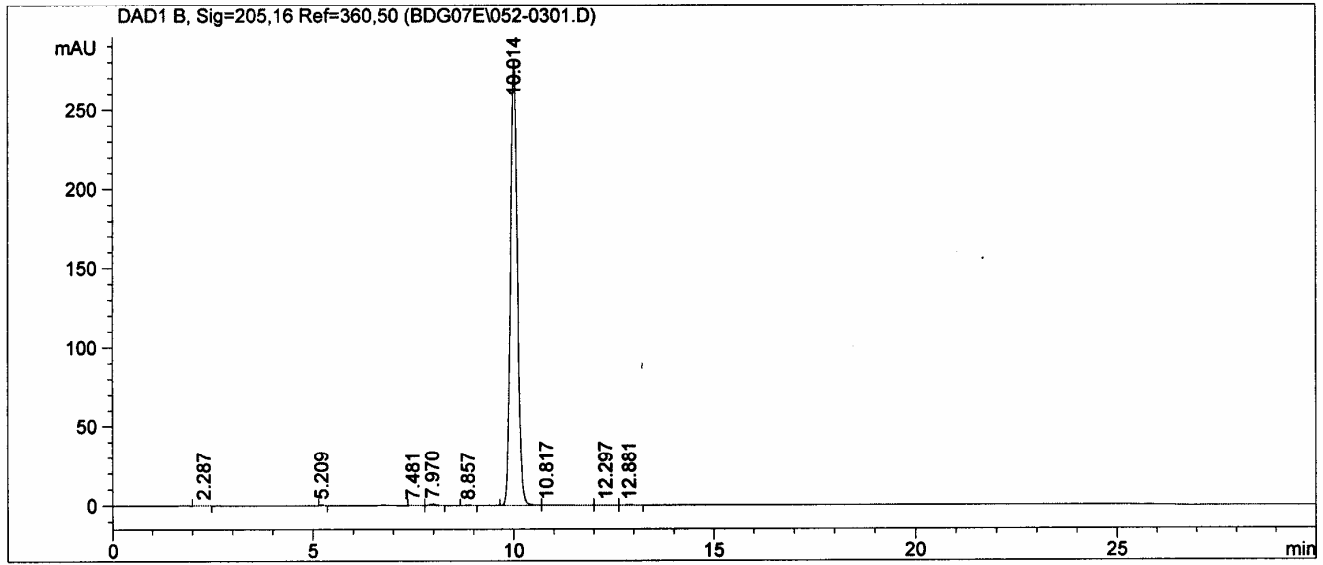
25

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Injection Date   : 7/15/03 11:53:13 AM          Seq. Line :    3
Sample Name     : BDG 4388.5                  Location  : Vial 52
Acq. Operator   : admin                       Inj       :    1
                                           Inj Volume: 10 µl

Acq. Method    : N:\LC1100_2\1\METHODS\LC40142A.M
Last changed   : 6/27/03 11:15:49 AM by admin
Analysis Method: N:\LC1100_2\1\METHODS\LC40142A.M
Last changed   : 7/15/03 1:54:55 PM by admin
                (modified after loading)
    
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BDG - isocratic analysis of androsterone derivative on Luna C18, 5µm, 250 x 4.6mm ID. # LC40142



Area Percent Report

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Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000
    
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Signal 1: DAD1 B, Sig=205,16 Ref=360,50

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.287	MM	0.2270	3.24810	2.38433e-1	0.0955
2	5.209	MM	0.1050	3.60856	5.72893e-1	0.1061
3	7.481	FM	0.2495	3.54371	2.36697e-1	0.1042
4	7.970	FM	0.1686	9.50186	9.39567e-1	0.2794
5	8.857	MM	0.1927	1.97927	1.71164e-1	0.0582
6	10.014	MF	0.1988	3363.05493	281.95935	98.9051
7	10.817	FM	0.7912	10.48696	2.20906e-1	0.3084
8	12.297	FM	0.3136	2.26369	1.20318e-1	0.0666
9	12.881	FM	0.2434	2.59724	1.77855e-1	0.0764

Totals : 3400.28432 284.63718

Results obtained with enhanced integrator!

*** End of Report ***