



BDG SYNTHESIS

Certificate of Analysis

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

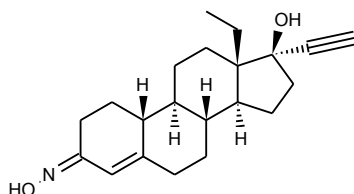
Barry Dent

Barry R. Dent, PhD, Director
19 March 2003

Name: Desacetylnorgestimate

CAS Number: 53016-31-2

Structure:



Molecular Weight: $C_{21}H_{29}NO_2 = 327.46$

Lot Number: BDG 2583.1

Appearance: White, crystalline solid

Corrected Purity: 99.7 % (HPLC) - 0.1 % (ethanol) = 99.6 %

Re-test Date: 19 March 2004

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. The material is known to be a mixture of E and Z oximes. As a consequence H4 appears as a pair of singlets in a 1.67 : 1 ratio.

Residual Solvents: a small amount of ethanol (0.1 % w/w) 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. Signals near the 3-oxime moiety appear as twinned sets.

High-resolution Mass Spectrum (FAB+)

Found m/z 328.2276. $C_{21}H_{30}NO_2$ $[M+H]^+$ requires m/z 328.2277. The deviation of 0.1 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

Two somewhat broadened, symmetrical peaks are observed (combined 99.7 %). 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 76.80, H 8.65, N 4.46 %
$C_{21}H_{29}NO_2$	Requires:	C 77.02, H 8.93, N 4.28 %

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).

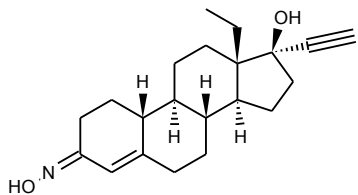
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

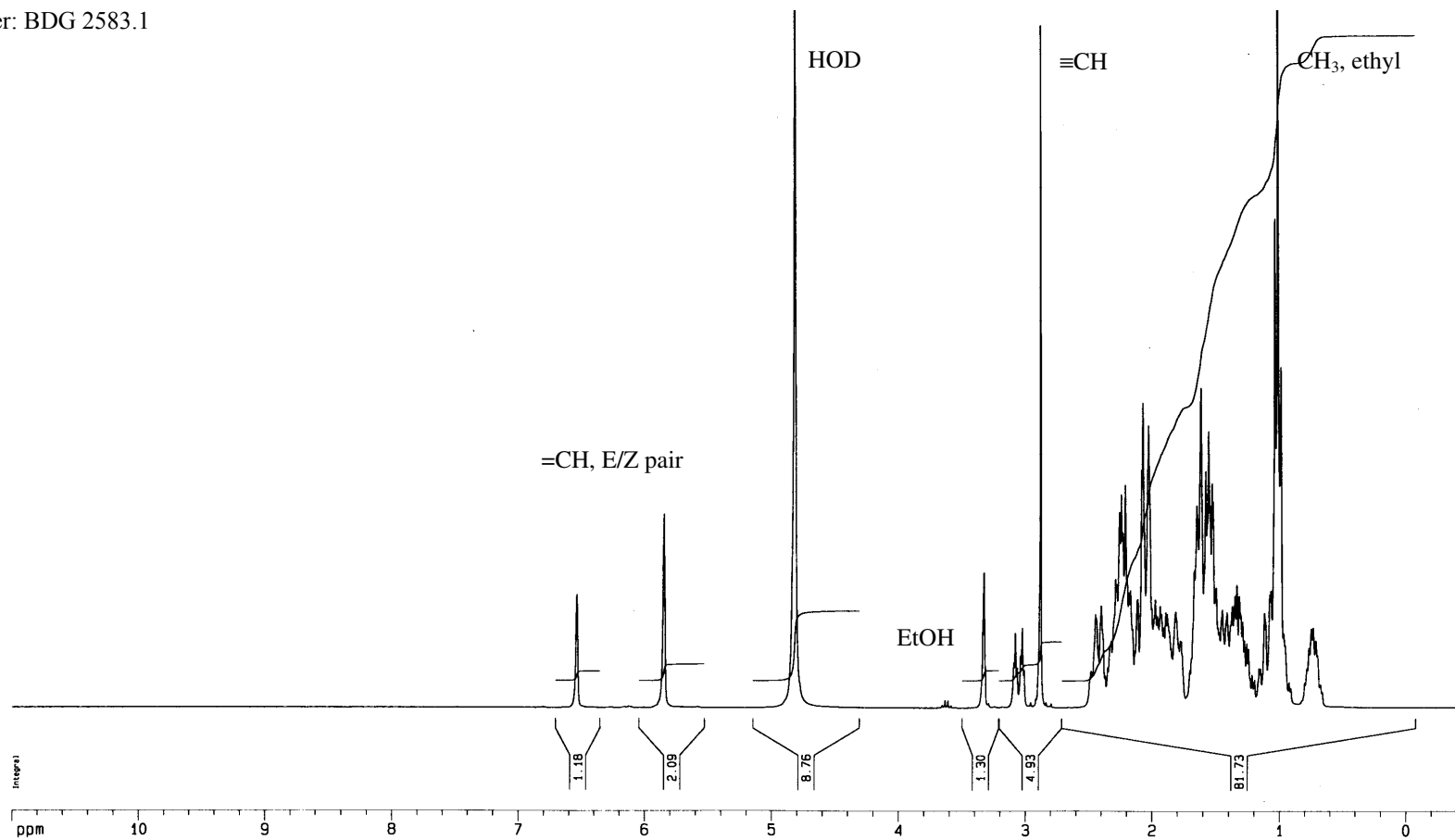


Proton NMR Spectrum of Desacetylnorgestimate in Methanol-d₄

BDG SYNTHESIS



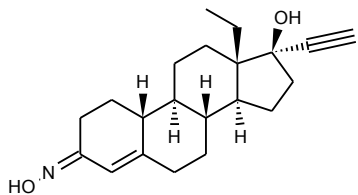
Lot Number: BDG 2583.1



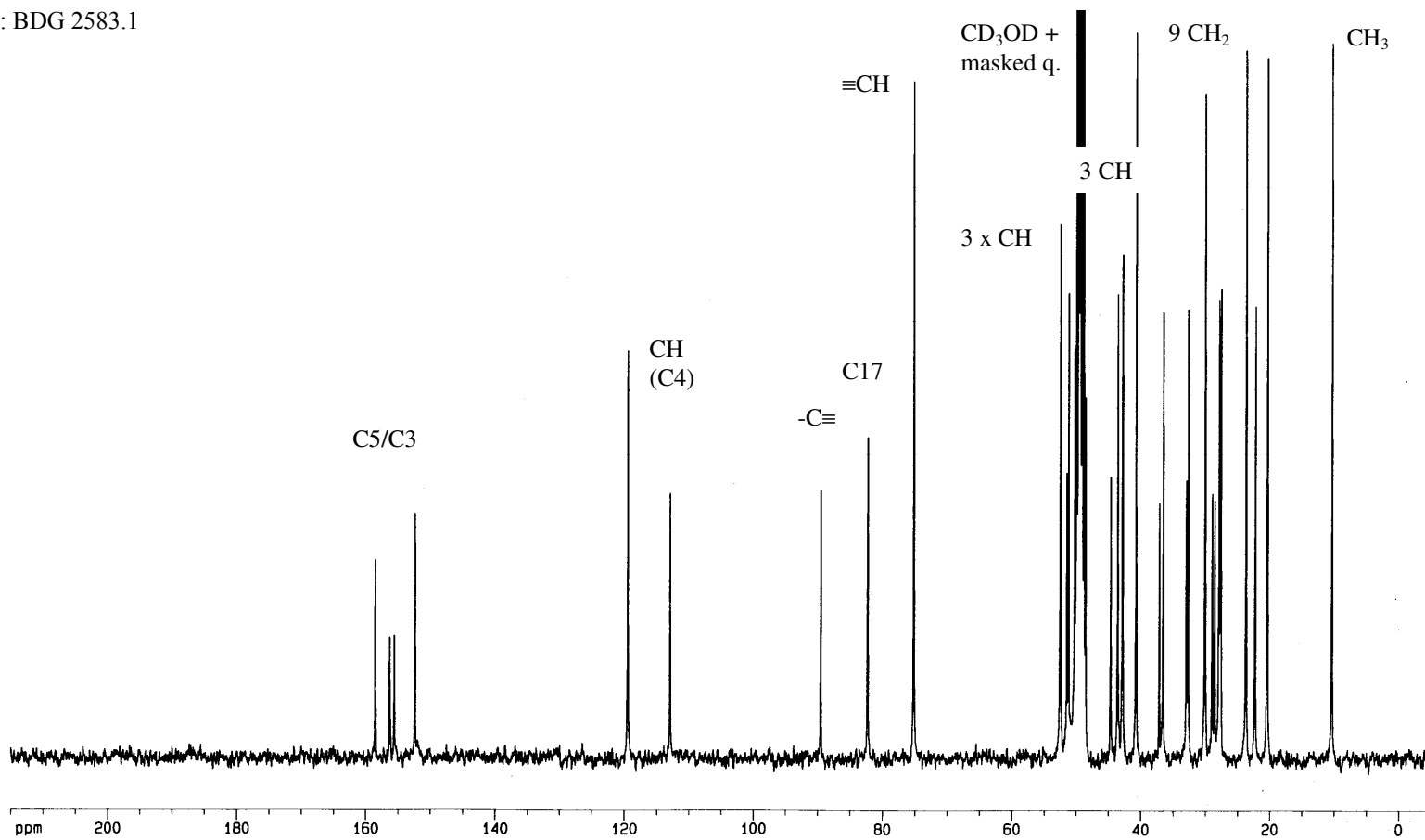


Carbon-13 NMR Spectrum of Desacetylnorgestimate in Methanol-d₄

BDG SYNTHESIS



Lot Number: BDG 2583.1



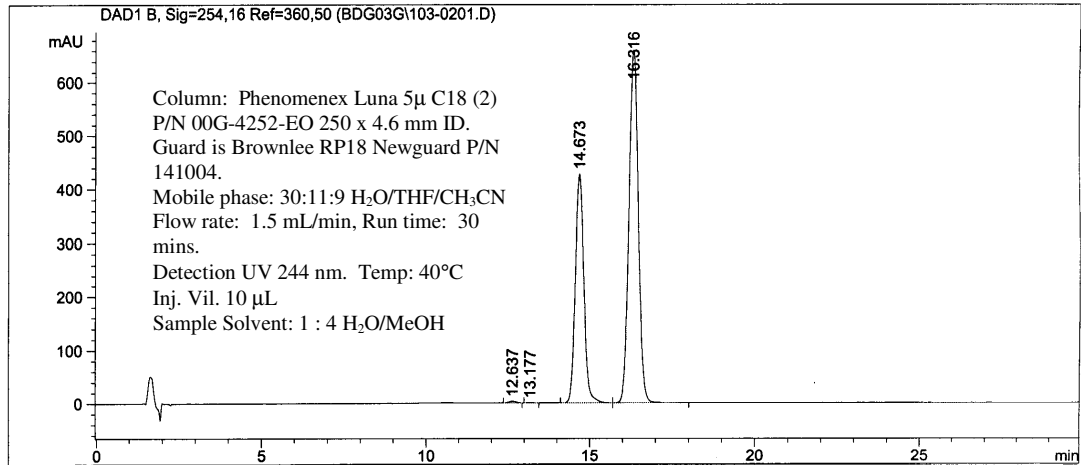
```

=====
Injection Date   : 3/11/03 7:32:29 AM           Seq. Line :    2
Sample Name     : BDG2583.1                   Location  : Vial 103
Acq. Operator   : admin                       Inj       :    1
                                           Inj Volume: 10 µl

Acq. Method     : N:\LC1100\1\METHODS\LC10133A.M
Last changed    : 3/11/03 7:53:04 AM by admin
                  (modified after loading)
Analysis Method : N:\LC1100\1\METHODS\LC10133A.M
Last changed    : 3/11/03 6:51:41 AM by admin

```

BDG - isocratic analysis of Norgestrel-3-oxime on Luna C18, 5µm, 250 x 4.6mm ID. # LC10133a



```

=====
                          Area Percent Report
=====

```

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000

```

Signal 1: DAD1 B, Sig=254,16 Ref=360,50

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.637	MM	0.2494	51.01440	3.40960	0.2439
2	13.177	MM	0.2788	9.63742	5.76128e-1	0.0461
3	14.673	MF	0.3029	7768.69580	427.42914	37.1484
4	16.316	FM	0.3304	1.30833e4	659.91016	62.5616

```
Totals :                2.09126e4  1091.32503
```

Results obtained with enhanced integrator!

```

=====
*** End of Report ***

```