



## 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.

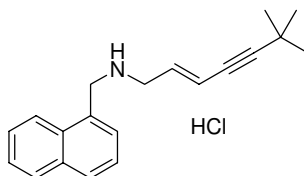
*Barry Dent*

Barry R. Dent, PhD, Director  
28 July 2003

**Name:** *N*-Desmethylterbinafine HCl

**CAS Number:** 152830-98-3

**Structure:**



**Molecular Weight:**  $C_{20}H_{23}N \cdot HCl = 313.87$

**Lot Number:** BDG 3617.5

**Appearance:** White, crystalline solid

**Purity by HPLC:** 99.1 %

**Expiry Date:** 28 July 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.

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: no residual solvents are observed.  
Impurities: no significant impurities are evident in the spectrum.

### Carbon-13 NMR Spectrum

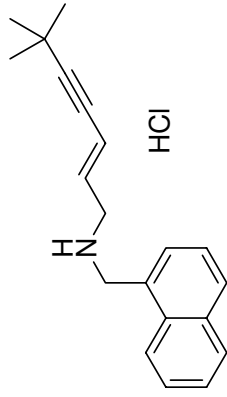
Identity: the signals are consistent with the proposed structure and in accord with literature where available.

**High-resolution mass spectrum (FAB+):** found  $m/z$  278.1920.  $C_{20}H_{24}N$   $[M+H]^+$  requires  $m/z$  278.1909. The deviation of 4.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

**HPLC:** A sharp, symmetrical peak is observed (99.1 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 76.73, H 7.90, N 4.68 %  
 $C_{20}H_{23}N \cdot HCl$  requires: C 76.53, H 7.71, N 4.46 %

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).



Proton NMR spectrum of *N*-Desmethylterbinafine HCl  
(BDG 3617.5) in DMSO- $d_6$ .

$C(CH_3)_3$   
9.04 H

$CH_2$   
2.00 H

$CH_2$   
2.00 H

HOD

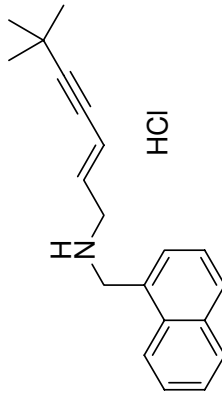
NMR  
Solvent

2xCH  
1.98 H

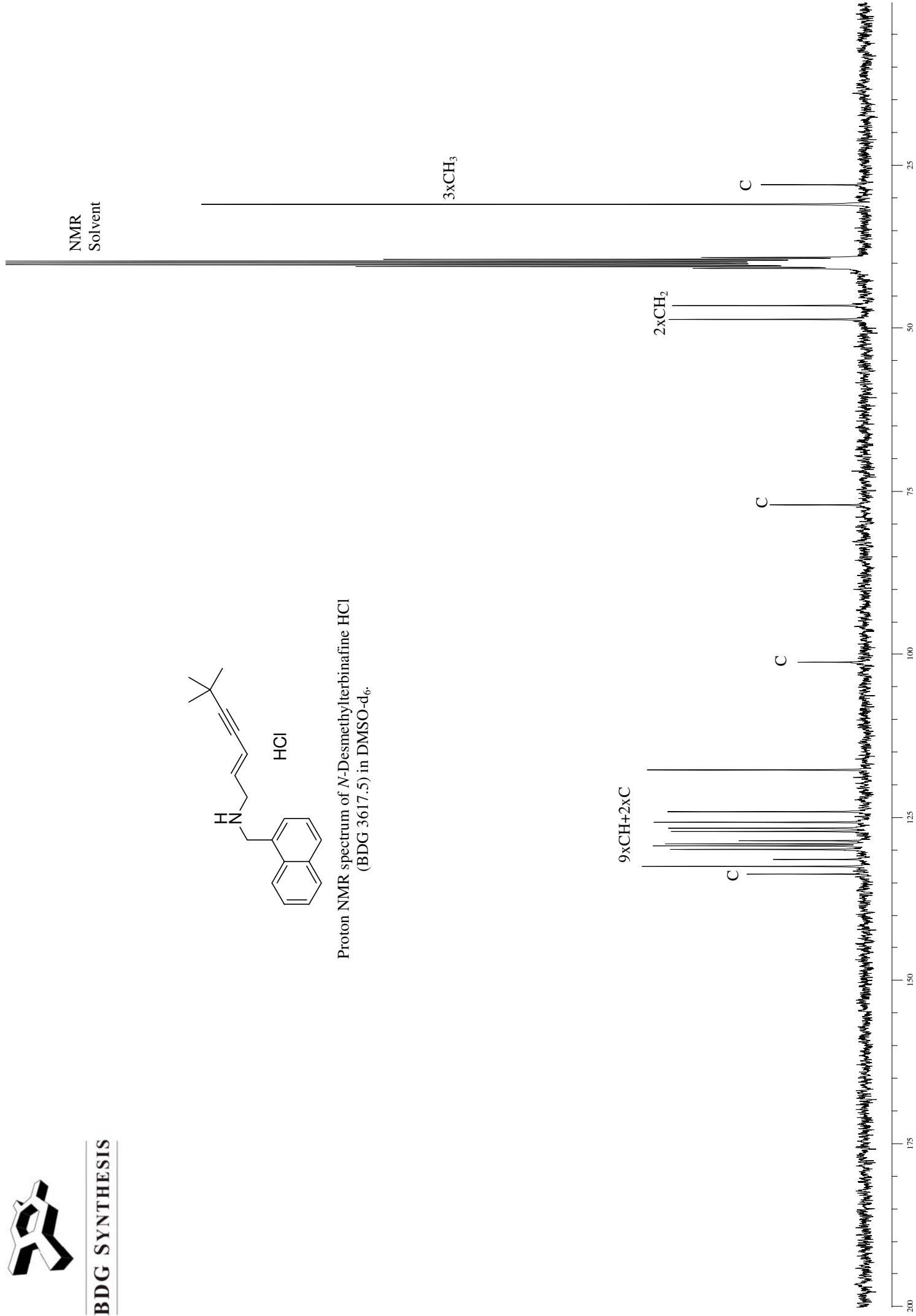
7xCH  
7.01 H

NH+HCl  
1.97 H





Proton NMR spectrum of *N*-Desmethylterbinafine HCl  
(BDG 3617.5) in DMSO- $d_6$ .



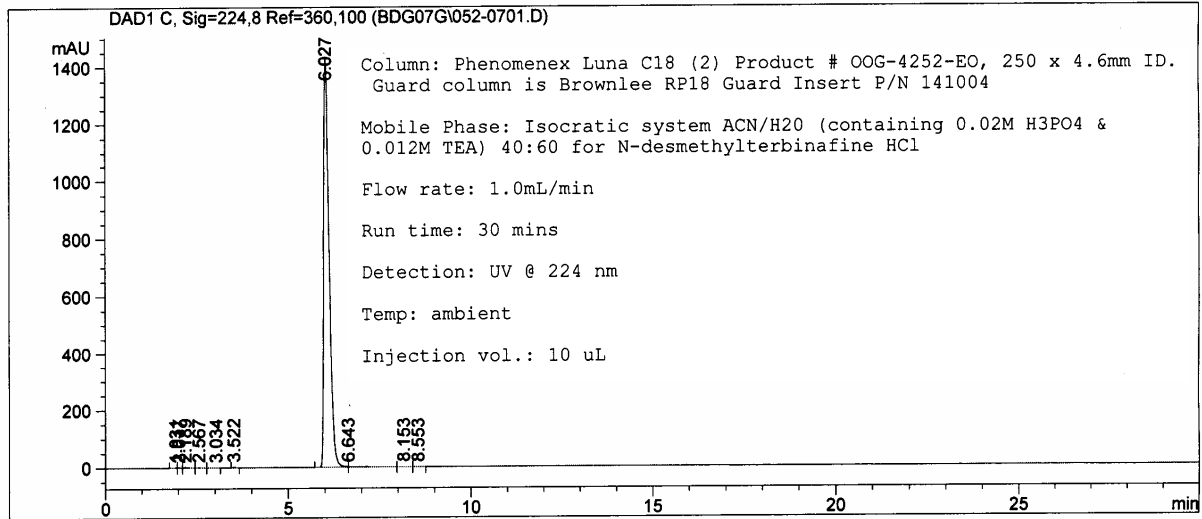
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Injection Date : 7/23/03 5:56:01 PM      Seq. Line : 7
Sample Name    : BDG 3617.5              Location  : Vial 52
Acq. Operator  : admin                   Inj      : 1
                                           Inj Volume: 10 µl

Acq. Method    : N:\LC1100_2\1\METHODS\LC40080B.M
Last changed   : 7/23/03 5:54:57 PM by admin
                (modified after loading)

Analysis Method : N:\LC1100_2\1\METHODS\LC40080B.M
Last changed   : 7/24/03 9:08:15 AM by admin
                (modified after loading)
    
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Isocratic analysis of (N-desmethyl)terbinafine-d5 on Luna C18(2) column with ACN/H2O (0.02M H3PO4 acid & 0.012M TEA) # LC40080



Area Percent Report

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Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000
    
```

Signal 1: DAD1 C, Sig=224,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.931	MF	0.0891	5.08873	9.51947e-1	0.0344
2	2.017	FM	0.0965	7.02313	1.21285	0.0474
3	2.189	FM	0.1239	21.30118	2.86542	0.1438
4	2.567	FM	0.1000	9.58176	1.59627	0.0647
5	3.034	FM	0.2641	5.89211	3.71873e-1	0.0398
6	3.522	BB	0.0763	10.65554	2.13385	0.0719
7	6.027	MF	0.1704	1.46844e4	1436.09509	99.1236
8	6.643	FM	0.6142	60.76591	1.64893	0.4102
9	8.153	FM	0.2969	6.35969	3.57042e-1	0.0429
10	8.553	FM	0.2468	3.15682	2.13224e-1	0.0213

Totals : 1.48142e4 1447.44649

Results obtained with enhanced integrator!

\*\*\* End of Report \*\*\*