



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.

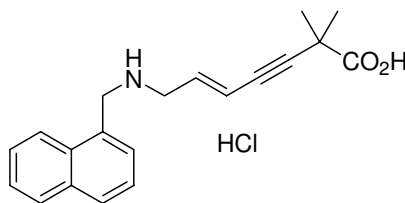
Barry Dent

Barry R. Dent, PhD, Director
2 October 2003

Name: *N*-Desmethylcarboxyterbinafine HCl

CAS Number: none (free base 99473-15-1)

Structure:



Molecular Weight: $C_{20}H_{21}NO_2 \cdot HCl = 343.85$

Lot Number: BDG 3658.7

Appearance: White, crystalline solid

Corrected Purity: 97.8 % (HPLC) – 1.5 % (acetone) – 1.3 % (water) = 95 %

Expiry Date: 2 October 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 small amount of acetone (1.5 % 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.

High-resolution mass spectrum (FAB+): found m/z 308.1666. $C_{20}H_{22}NO_2 [M+H]^+$ requires m/z 308.1651. The deviation of 5.0 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC: A sharp, symmetrical peak is observed (97.8 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 69.18, H 6.54, N 4.02 %.
$C_{20}H_{21}NO_2 \cdot HCl \cdot 0.2H_2O$	requires:	C 69.14, H 6.50, N 4.03 %.
$C_{20}H_{21}NO_2 \cdot HCl$	requires:	C 69.86, H 6.45, N 4.07 %.

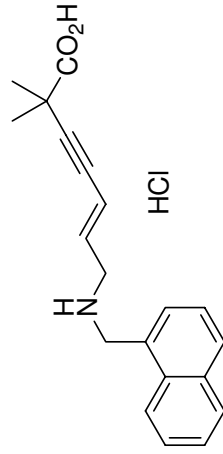
The elemental analyses fall slightly outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the "best-fit" hydrated molecular formula is given.

Karl Fischer Analysis:	Found H_2O	1.3 %.
$C_{20}H_{21}NO_2 \cdot HCl \cdot 0.2H_2O$	requires H_2O	1.0 %.

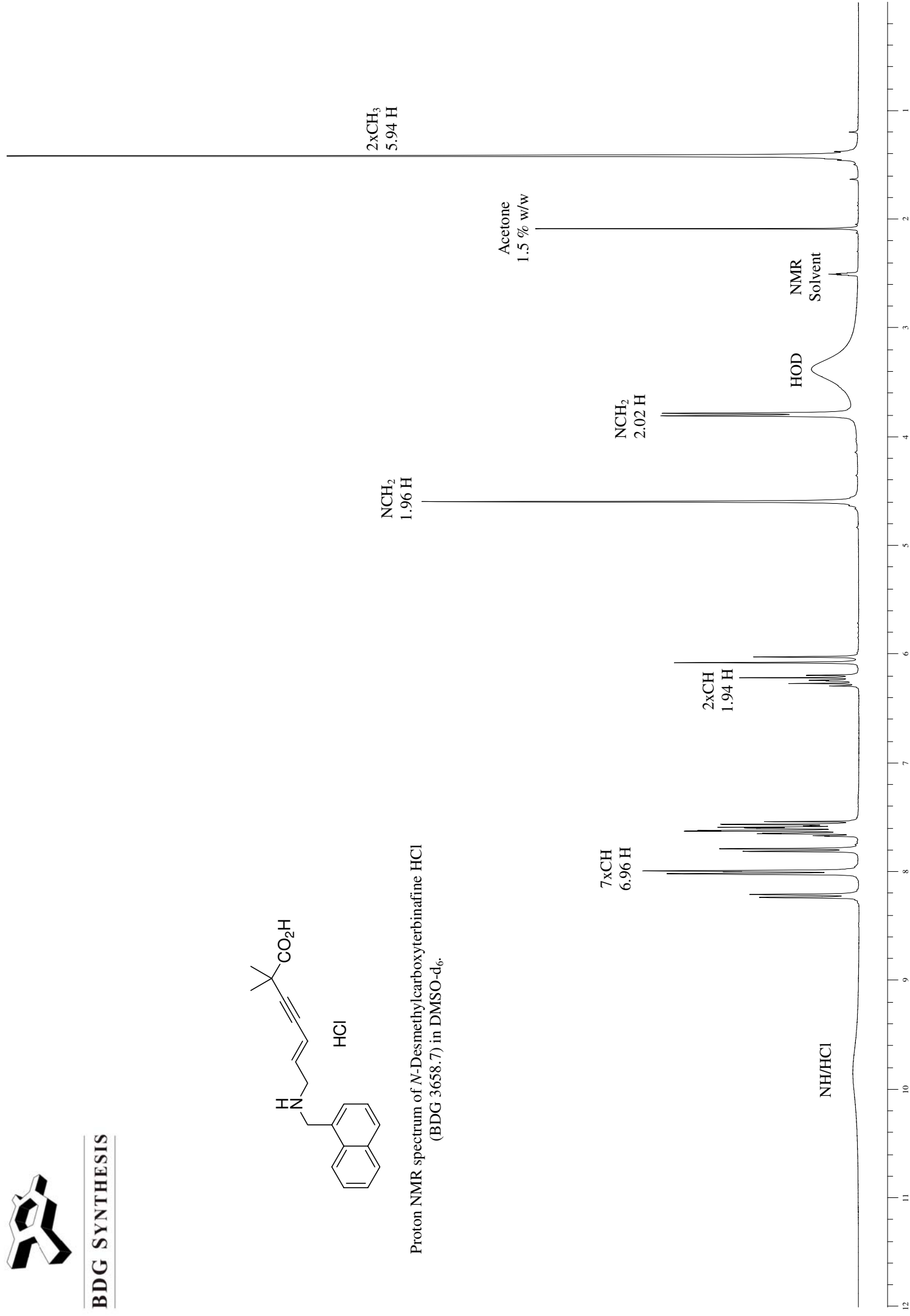
Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.



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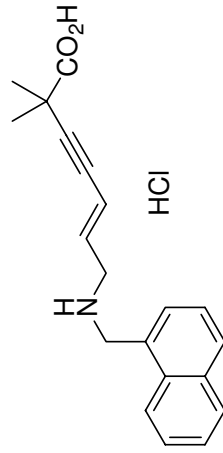


Proton NMR spectrum of *N*-Desmethylcarboxyterbinafine HCl
(BDG 3658.7) in DMSO- d_6 .

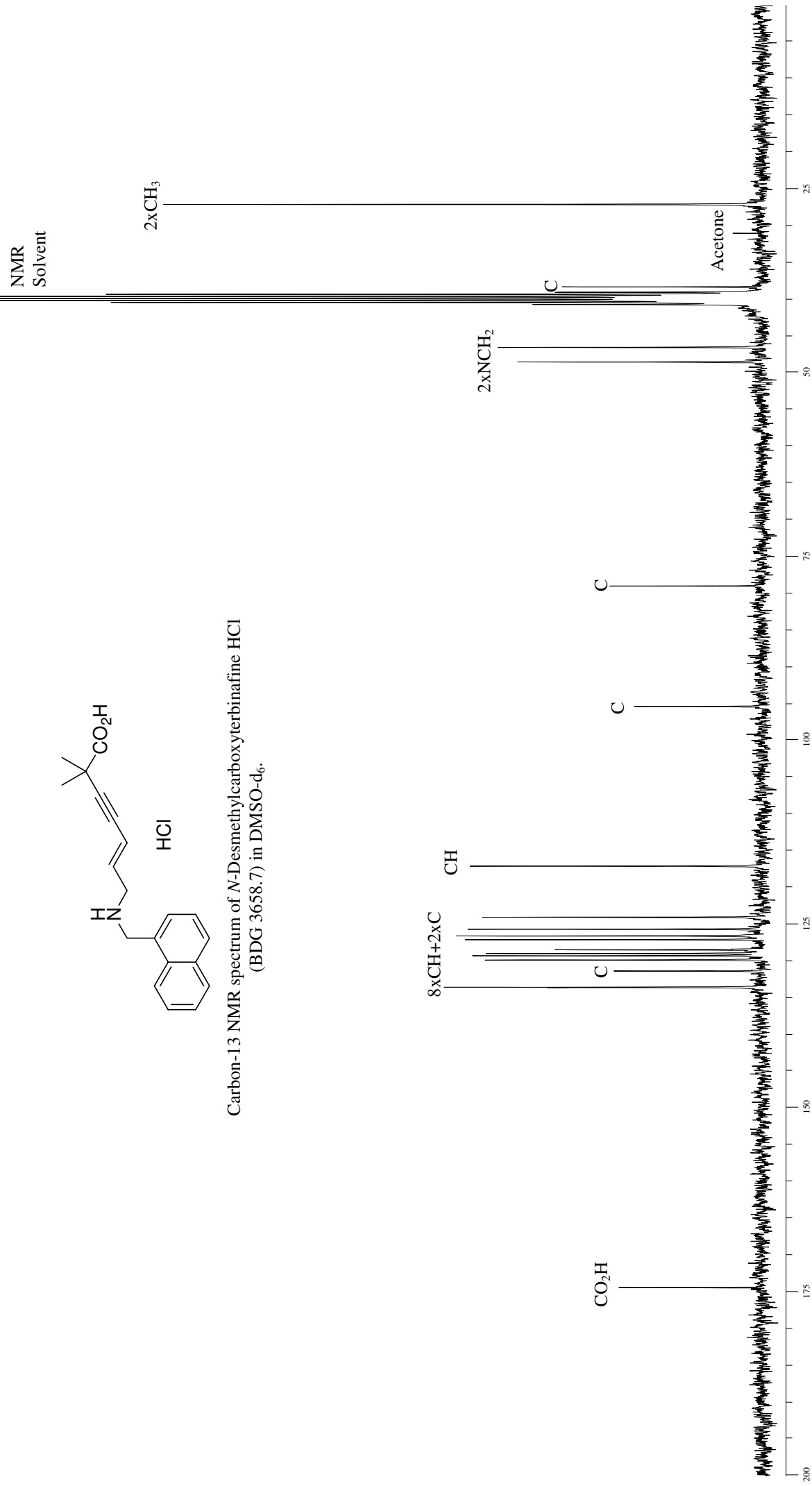




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Carbon-13 NMR spectrum of *N*-Desmethylcarboxyterbinafine HCl (BDG 3658.7) in DMSO- d_6 .



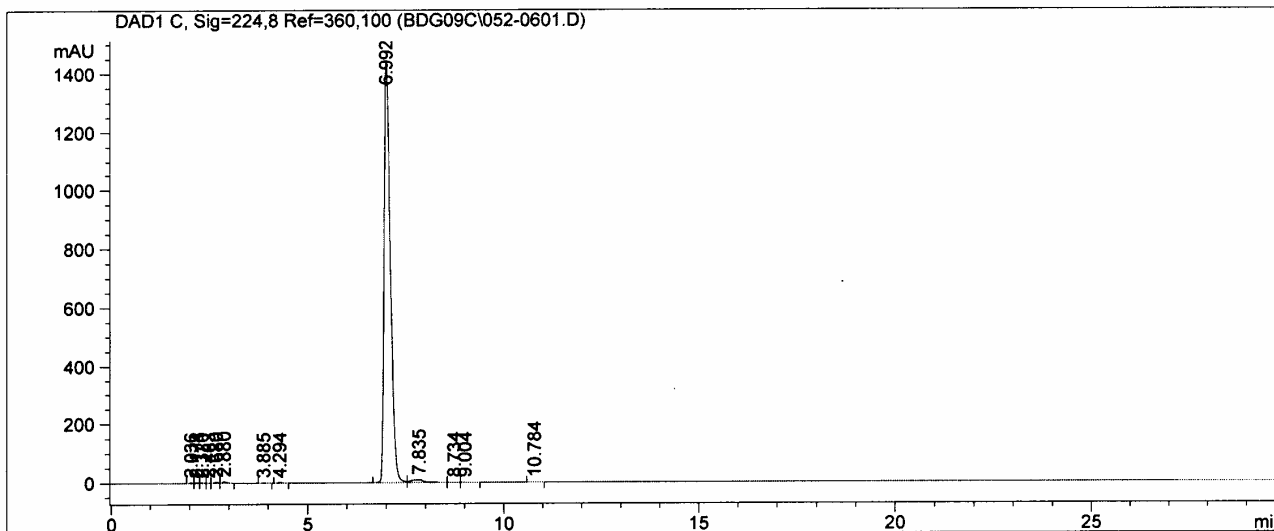
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Injection Date   : 9/10/03 3:45:42 PM           Seq. Line :    6
Sample Name     : BDG3658.7                   Location  : Vial 52
Acq. Operator   : admin                       Inj       :    1
                                           Inj Volume: 10 µl

Acq. Method     : N:\LC1100_2\1\METHODS\LC40080B.M
Last changed    : 9/10/03 3:44:32 PM by admin
                  (modified after loading)

Analysis Method : N:\LC1100_2\1\METHODS\LC40080B.M
Last changed    : 9/10/03 4:44:45 PM 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



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Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
    
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Signal 1: DAD1 C, Sig=224,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.036	BV	0.0854	8.59047	1.48641	0.0525
2	2.174	VV	0.0798	9.94875	1.87671	0.0608
3	2.320	VV	0.0842	9.61295	1.64445	0.0588
4	2.483	VV	0.0784	5.24265	9.79833e-1	0.0320
5	2.680	VV	0.1097	24.35128	3.03212	0.1488
6	2.880	VB	0.1064	40.08033	5.17019	0.2450
7	3.885	PP	0.1158	7.53102	1.00080	0.0460
8	4.294	BB	0.1216	14.24340	1.81481	0.0871
9	6.992	MF	0.1846	1.60083e4	1445.62622	97.8424
10	7.835	FM	0.3511	214.50153	10.18241	1.3110
11	8.734	FM	0.2677	8.02052	4.99403e-1	0.0490
12	9.004	FM	0.2426	6.27711	4.31317e-1	0.0384
13	10.784	MM	0.2489	4.61838	3.09270e-1	0.0282

Totals : 1.63613e4 1474.05396

Results obtained with enhanced integrator!

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*** End of Report ***