



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.

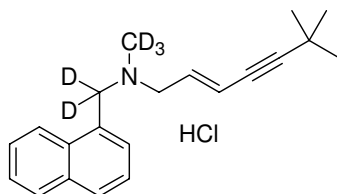
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

Barry Dent

Barry R. Dent, PhD, Director
17 November 2006

Name: Terbinafine-d₅ HCl
CAS Number: none (78628-80-5 unlabelled)

Structure:



Molecular Weight: C₂₁H₂₀D₅N•HCl = 332.94

Lot Number: BDG 6614.1

Appearance: White, crystalline solid

Purity by HPLC: 99.9 %

Isotopic Purity: Under 0.5 % d₀

Expiry Date: 17 November 2011

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

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

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. 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 (FAB+): found m/z 297.2394. $C_{21}H_{21}D_5N$ $[M+H]^+$ requires m/z 297.2379. The deviation of 4.9 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for d_0 material was seen (detection limit about 0.5 %).

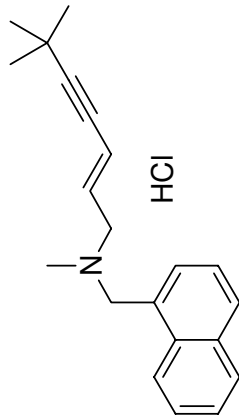
HPLC: A sharp, symmetrical peak is observed (99.9 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis: Found: C 75.68, H 6.29, D 3.00, N 4.21 %
 $C_{21}H_{20}D_5N \cdot HCl$ requires: C 75.76, H 6.36, D 3.03, N 4.21 %

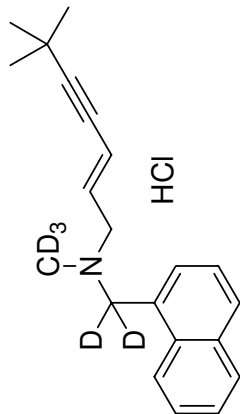
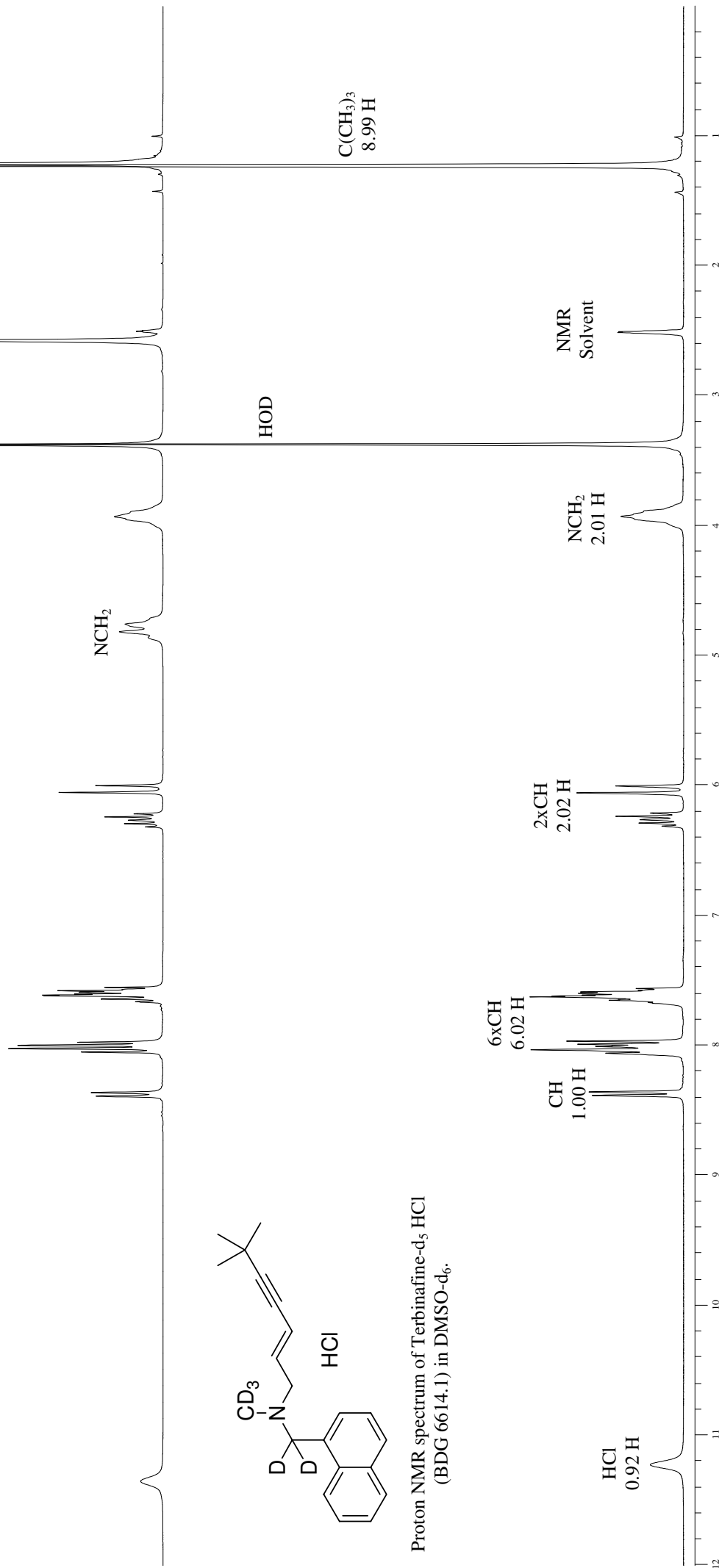
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).



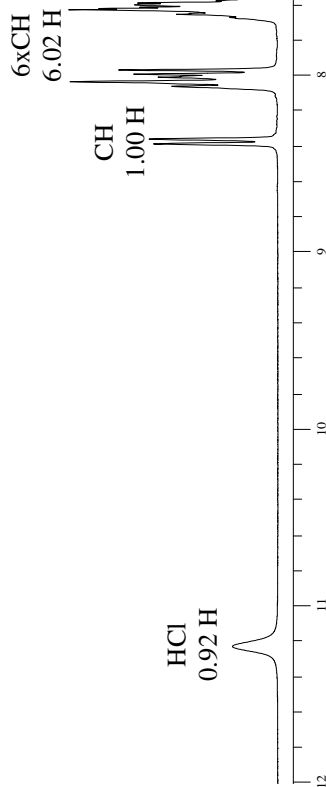
BDG SYNTHESIS



Proton NMR spectrum of Terbinafine HCl
in DMSO-d₆.

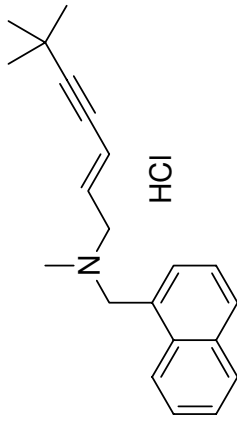


Proton NMR spectrum of Terbinafine-d₅ HCl
(BDG 6614.1) in DMSO-d₆.

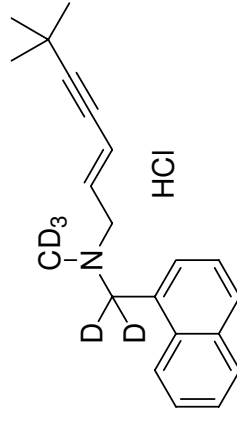
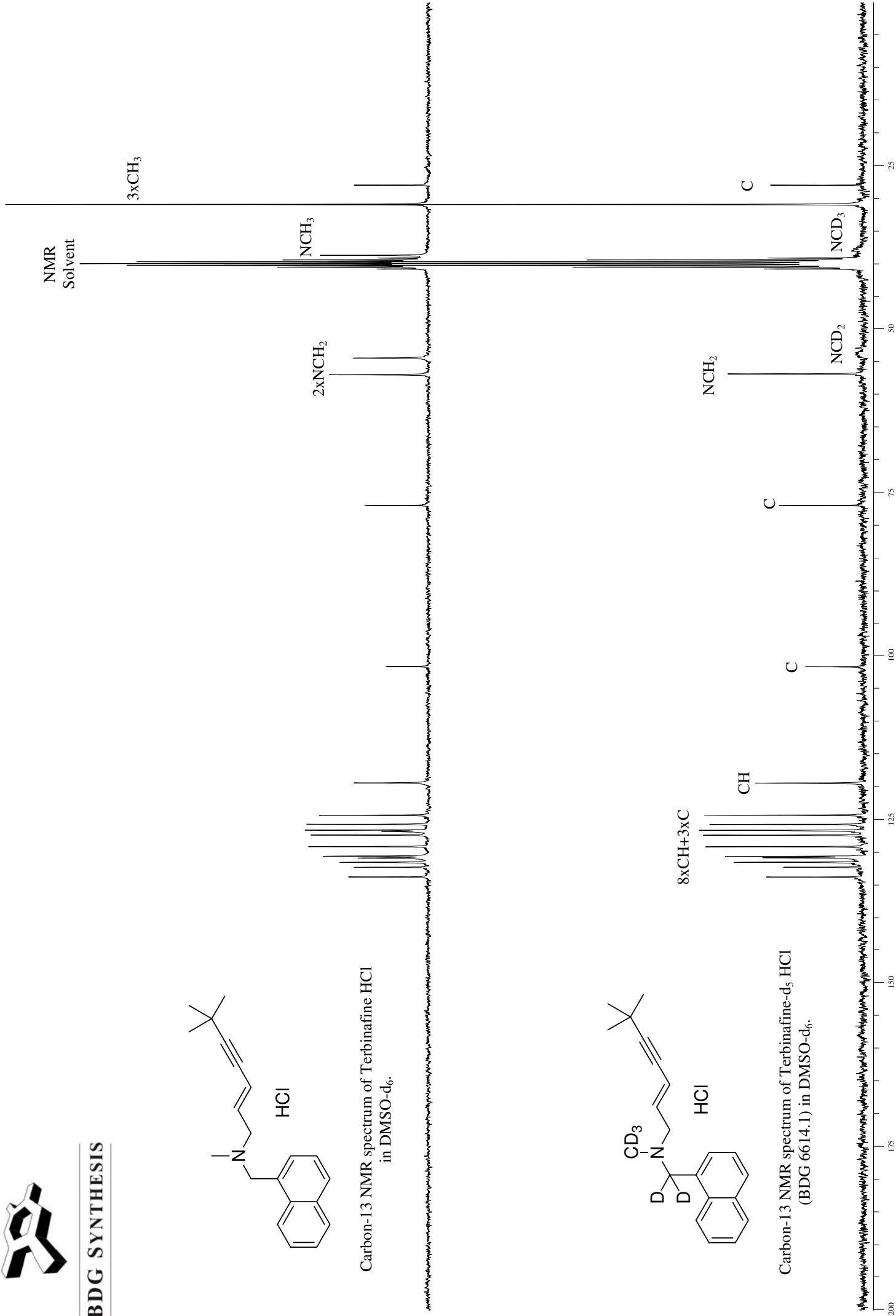




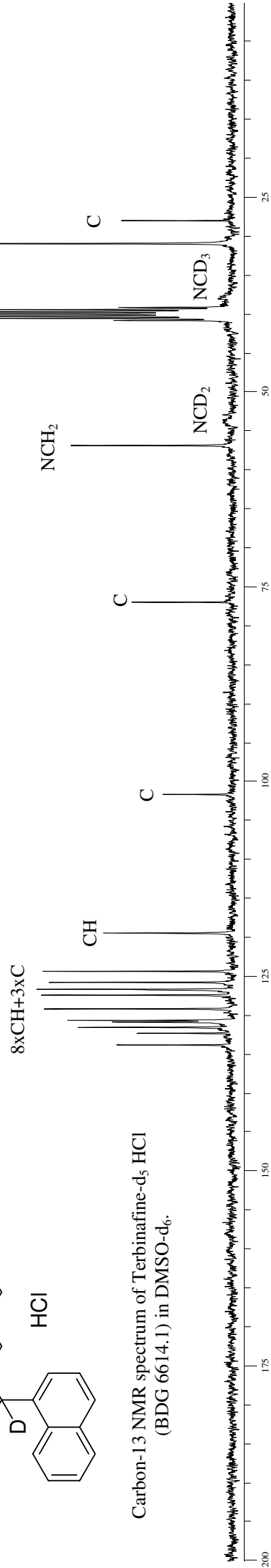
BDG SYNTHESIS



Carbon-13 NMR spectrum of Terbinafine HCl
in DMSO-d₆.



Carbon-13 NMR spectrum of Terbinafine-d₅ HCl
(BDG 6614.1) in DMSO-d₆.



BDG - Analysis of Terbinafine-d5 hydrochloride

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm

Guard : Phenomenex Security Guard C18 RP 4 x 3 mm

Mobile Phase : 50:50 Water + 0.02M Phosphoric Acid + 0.012M Triethylamine : Acetonitrile

Flow Rate : 1.0 mL/min

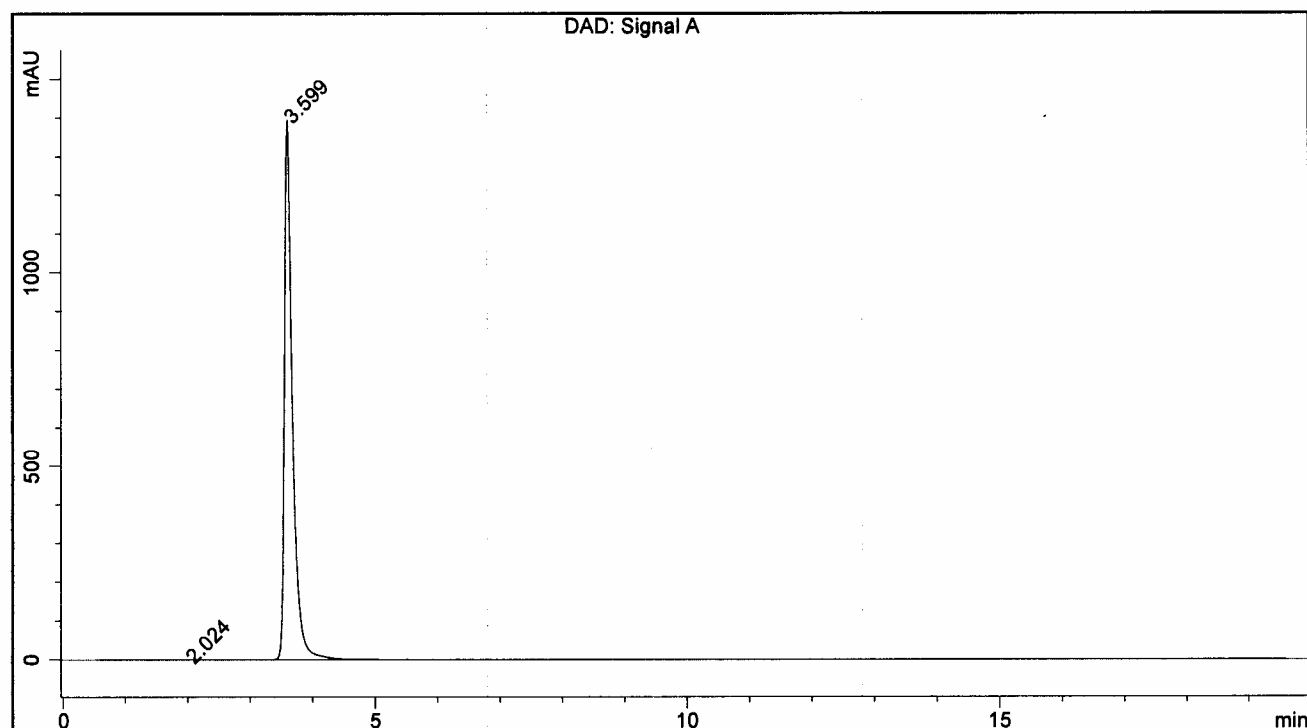
Sample Solvent : Mobile Phase

Column Temperature : 20C

Injection Volume : 10 uL

Detection : UV at 224 nm

Sample Name	BDG 6614.1	Instrument	AnalyticalLC01
Acquisition	09/11/2006, 21:02:25	Method (rev.)	LC10105a (4)
Sequence	BDG_09Nov2006c - Reprocessed	Vial Position	2
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	2.02 min	0.2936	3.1394	0.1408 min	0.025 %
2	3.60 min	1394.8157	12425.4455	0.1299 min	99.975 %