



## 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.

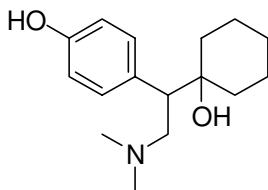
*Barry Dent*

Barry R. Dent, PhD, Director  
26 April 2006

**Name:** *O*-Desmethylvenlafaxine

**CAS Number:** 93413-62-8

**Structure:**



**Molecular Weight:**  $C_{16}H_{25}NO_2 = 263.38$

**Lot Number:** BDG 5432.1

**Appearance:** White, crystalline solid

**Corrected Purity:** 98.8 % (HPLC) – 0.5 % (methanol) = 98.3 %

**Expiry Date:** 26 April 2007

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: a small amount of methanol (0.5 % 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 (ESI+):** found  $m/z$  264.1962.  $C_{16}H_{26}NO_2$ ,  $[M+H]^+$ , requires  $m/z$  264.1958. The deviation of 1.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

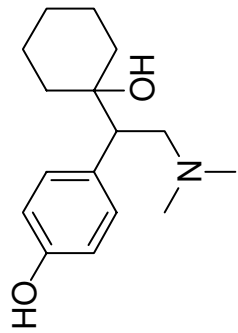
**HPLC:** A sharp, slightly tailing peak is observed (98.8 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

**Elemental Analysis:** Found: C 72.81, H 9.68, N 5.32 %  
 $C_{16}H_{25}NO_2$  requires: C 72.96, H 9.57, N 5.32 %

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 *O*-Desmethylvenlafaxine (BDG5432.1) in DMSO- $d_6$ .

4xCH  
4.01 H

N(CH<sub>3</sub>)<sub>2</sub>  
6.00 H

HOD

CH+CH<sub>2</sub>  
3.06 H

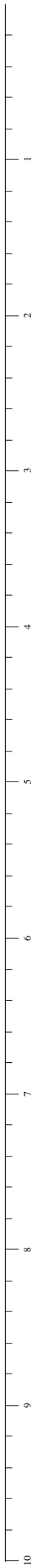
5xCH<sub>2</sub>  
10.27 H

OH  
0.84 H

MeOH →  
0.48 % w/w

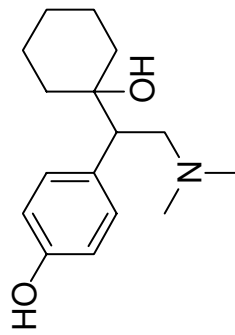
OH  
0.86 H

NMR Solvent

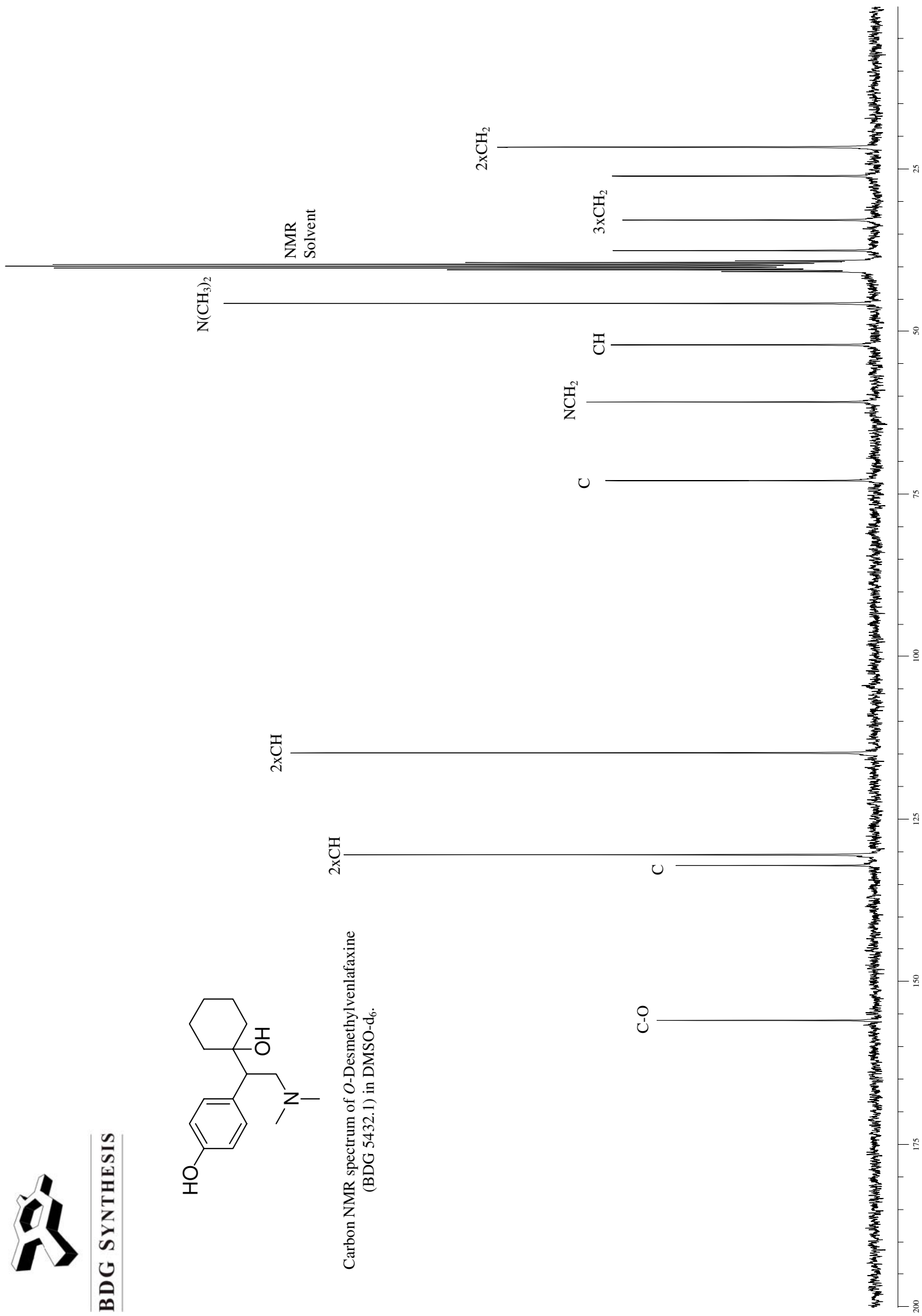




## BDG SYNTHESIS



Carbon NMR spectrum of *O*-Desmethylvenlafaxine (BDG 5432.1) in DMSO- $d_6$ .

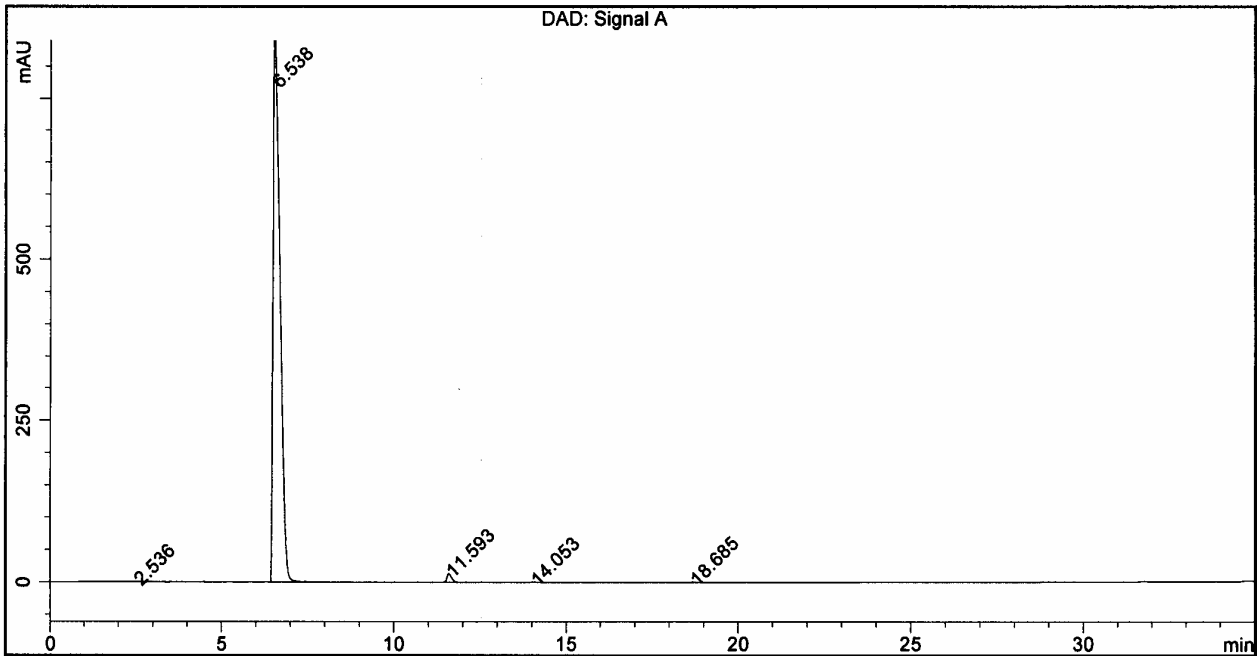


# Solvation Analytical Report

**BDG - Analysis of venlafaxine and similar compounds**

Column : Phenomenex Luna C8, 5um 250 mm x 4.6 mm.  
 Guard : Phenomenex Security Guard C8 4 mm x 3 mm  
 Mobile Phase A: 10 mM KH<sub>2</sub>PO<sub>4</sub> ( pH=4.5 )  
 Mobile Phase B: CH<sub>3</sub>CN  
 Gradient ( A:B ) : T0=80:20, T30=40:60, T35=40:60, T40=80:20  
 Post Time : 5 mins.  
 Flow Rate : 1 mL/min  
 Sample : 1 mg/mL Initial Mobile Phase  
 Column Temp : 20C  
 Injection Volume : 10 ul  
 Detection : UV at 225 nm

<b>Sample Name</b>	BDG 5432.1	<b>Instrument</b>	Analytical LC 01
<b>Acquisition</b>	17-Apr-06, 01:29:41	<b>Method (rev.)</b>	LC10033 ( 7 )
<b>Sequence</b>	BDG_16Apr2006b - Reprocessed	<b>Vial Position</b>	4
<b>Operator</b>	solvation010\cerityadmin	<b>Injection</b>	2 of 2



**Area Percent Report**

Peak #	RT	Height	Area	Width	Area %
1	2.54	0.6245	2.7459	0.0653	0.023
2	6.54	868.1474	11959.9982	0.2027	98.806
3	11.59	13.3442	127.4471	0.1414	1.053
4	14.05	0.8696	6.8872	0.1224	0.057
5	18.68	0.8311	7.4439	0.1365	0.061