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.

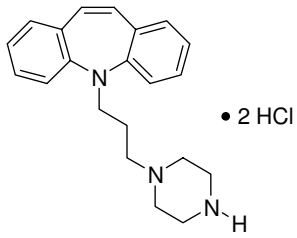
Barry Dent

Barry R. Dent, PhD, Director
1 December 2005

Name: Des-(2-hydroxyethyl)opipramol 2HCl

CAS Number: 4346-38-7

Structure:



Molecular Weight: $C_{21}H_{25}N_3 \bullet 2HCl = 392.36$

Lot Number: BDG 2995.1

Appearance: Pale yellow, crystalline solid

Corrected Purity: 99.9 % (HPLC) – 2.5 % (acetone) – 1.5 % (H_2O) = 95.9 %

Expiry Date: 1 December 2006
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.

• Custom synthesis of analytical reference standards, metabolites, stable isotope labelled compounds • Contract research • BDG Synthesis is a division of B Dent Global Limited

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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 (2.5 % *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* 320.2111. $C_{21}H_{26}N_3$ $[M+H]^+$ requires *m/z* 320.2121. The deviation of 3.1 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC: A broad, slightly tailing 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.

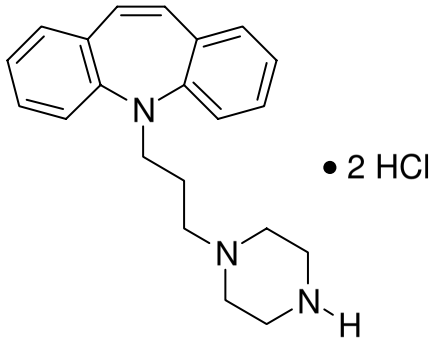
Elemental Analysis:	Found:	C 63.12, H 6.83, N 10.36, Cl 17.62 %
$C_{21}H_{25}N_3 \bullet 2HCl \bullet 0.33H_2O$	requires:	C 63.31, H 7.00, N 10.55, Cl 17.80 %
$C_{21}H_{25}N_3 \bullet 2HCl$	requires:	C 64.28, H 6.94, N 10.71, Cl 18.07 %

The elemental analyses fall somewhat 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	1.5 %.
$C_{21}H_{25}N_3 \bullet 2HCl \bullet 0.33H_2O$	requires H_2O	1.5 %.

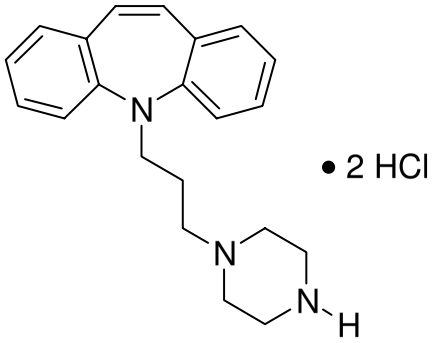
Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.

BDG SYNTHESIS



Proton NMR spectrum of Des-(2-hydroxyethyl)opipramol 2HCl (BDG 2995.1) in D_2O.

BDG SYNTHESIS



Carbon-13 NMR spectrum of Des-(2-hydroxyethyl)opipramol 2HCl (BDG 2995.1) in D_2O.

Data File D:\HPCHEM\1\DATA\2005\NOV05\BDG11B\008-0601.D Sample Name: BDG2995.1

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Injection Date  : 11/30/05 11:43:58 AM          Seq. Line :   6
Sample Name     : BDG2995.1                      Location : Vial 8
Acq. Operator   : YRLman                             Inj :   1
                                                Inj Volume : 10 µl
Acq. Method     : N:\LC1100\1\METHODS\LC10259A.M
Last changed    : 11/30/05 10:10:13 AM by YRLman
Analysis Method : N:\LC1100\1\METHODS\LC10259A.M
Last changed    : 11/30/05 2:17:28 PM by YRLman
                  (modified after loading)

BDG - isocratic analysis of opipramol on Luna C18, 5um, 250 x 4.6mm ID.  # LC10259
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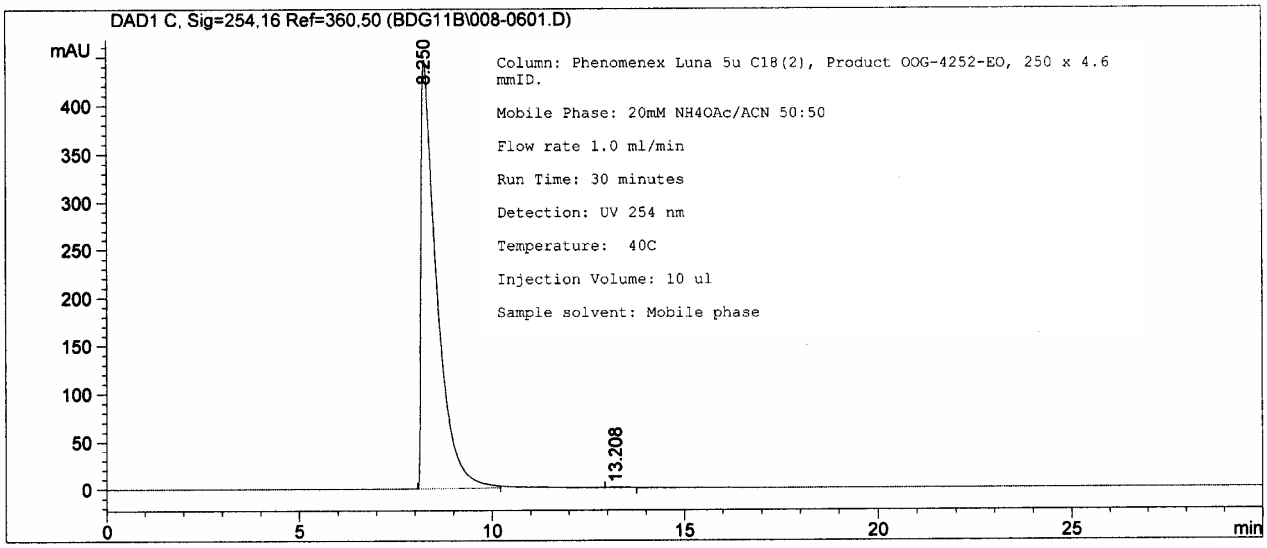


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==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   8.250 MM    0.4726 1.26642e4   446.65625  99.8520
   2  13.208 MM    0.5342   18.77267 5.85648e-1   0.1480

Totals :                  1.26830e4   447.24190

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
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