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.

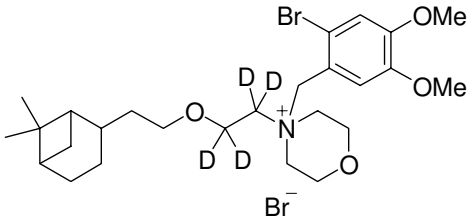
Barry Dent

Barry R. Dent, PhD, Director
26 August 2005

Name: Pinaverium-d_4 bromide

CAS Number: none (53251-94-8 unlabelled)

Structure:



Molecular Weight: $C_{26}H_{37}D_4BrNO_4^+Br^-$ = 595.45

Lot Number: BDG 4866.3

Appearance: White, crystalline solid

Corrected Purity: 99.3 % (HPLC) – 0.9 % (2-butanone) = 98.4 %

Isotopic Purity: Under 0.5 % d_0

Expiry Date: 26 August 2010
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.
Isotopic labelling: signals at the sites of deuteration are greatly diminished, compared with the spectrum of unlabelled commercial material, indicating clean deuteration. The slight differences between the spectra are associated with concentration effects.
Residual solvents: a small amount of 2-butanone (0.9 % *w/w*) is observed.
Impurities: an unidentified isomer of the product is seen in both spectra.

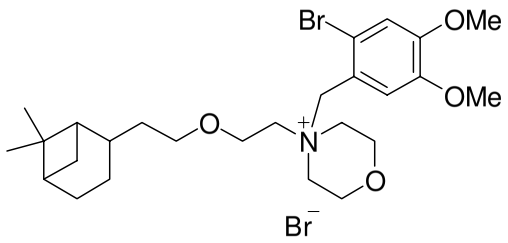
Carbon-13 NMR Spectrum
Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Isotopic labelling: signals at the sites of deuteration have collapsed to small multiplets compared with the spectrum of unlabelled material, indicating clean deuteration.

High-resolution mass spectrum (ESI+): found *m/z* 514.2482. $C_{26}H_{37}D_4BrNO_4$ $[M]^+$ requires *m/z* 514.2470. The deviation of 2.3 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 %).

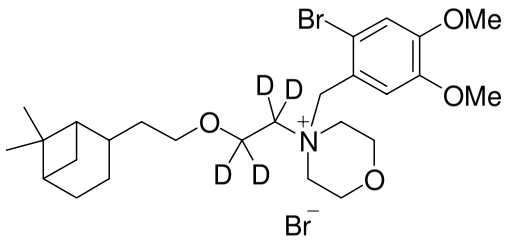
HPLC: A sharp, slightly tailing peak is observed (99.3 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis: Found: C 52.35, H 6.43, D 1.41, N 2.50 %
$C_{26}H_{37}D_4BrNO_4^+Br^-$ requires: C 52.45, H 6.26, D 1.35, N 2.35 %
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

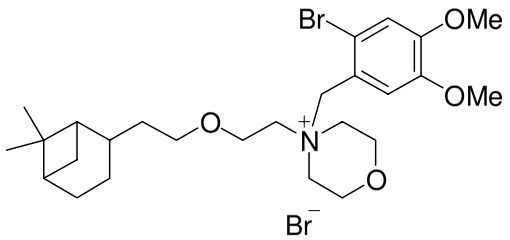


Proton NMR spectrum of Pinaverium bromide
(BDG 4849) in $CDCl_3$.

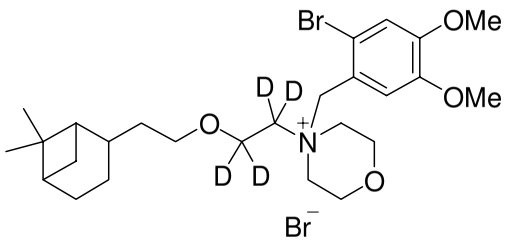


Proton NMR spectrum of Pinaverium-d_4 bromide
(BDG 4866.3) in $CDCl_3$.

BDG SYNTHESIS



Carbon-13 NMR spectrum of Pinaverium bromide (BDG 4849) in $CDCl_3$.



Carbon-13 NMR spectrum of Pinaverium-d_4 bromide (BDG 4866.3) in $CDCl_3$.

Data File D:\HPCHEM\1\DATA\2005\AUG05\BDG08F\032-0301.D Sample Name: BDG 4866.3

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==========================================================================
Injection Date  : 8/19/05 11:32:51 AM          Seq. Line :   3
Sample Name     : BDG 4866.3                    Location : Vial 32
Acq. Operator   : YRLman                           Inj :   1
                                               Inj Volume : 10 µl
Method          : N:\LC1100_3\1\METHODS\LC50240A.M
Last changed    : 8/19/05 10:59:22 AM by YRLman

BDG - isocratic analysis of pinaverium bromide on Luna C18, 5um, 250 x 4.6mm ID.  # LC50240
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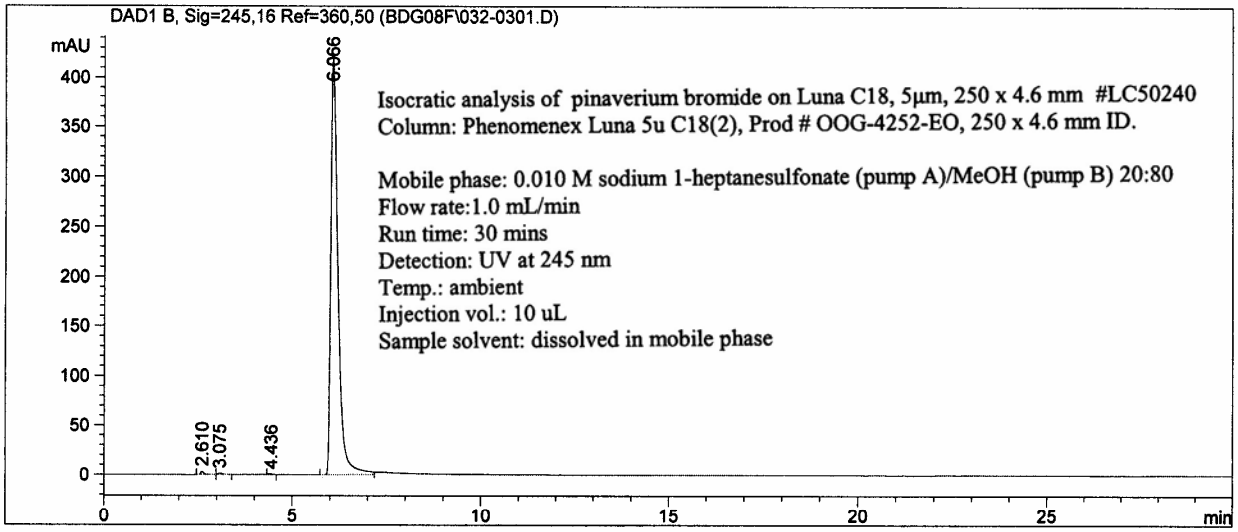


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==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: DAD1 B, Sig=245,16 Ref=360,50

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   2.610 MF    0.0933   19.55766    3.49234   0.3484
   2   3.075 FM    0.1512   12.30349    1.35592   0.2192
   3   4.436 FM    0.1603    6.88602 7.15817e-1   0.1227
   4   6.066 MM    0.2209 5575.36328  420.62192  99.3098

Totals :                   5614.11045  426.18599

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