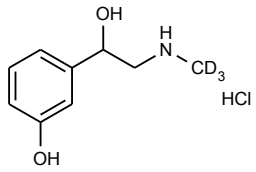
BDG SYNTHESIS

Certificate of Analysis

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Barry Dent

Barry R. Dent, PhD, Director
9 May 2008

Name:	**Phenylephrine-d_3 HCl**
CAS Number:	61-76-7 (unlabelled)
Structure:	
Molecular Weight:	$C_9H_{10}D_3NO_2 \cdot HCl = 206.68$
Lot Number:	BDG 8538.4
Appearance:	White, crystalline solid
Corrected Purity:	99.1 % (HPLC) - 0.4 % (ethanol) = 98.7 %
Isotopic Purity:	Under 0.5 % d_0
Re-test Date:	9 May 2013

Storage and Handling:

Temperature:	ambient laboratory temperature; may be refrigerated.
Humidity:	not believed to be hygroscopic; may be handled in normal laboratory atmosphere.
Light:	store in an amber vial and protect from bright light.
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

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Isotopic Labelling: the signal at the site of deuteration is absent compared with the spectrum of unlabelled material, indicating clean deuteration.
Residual Solvents: a small amount of ethanol (0.4 % *w/w*) is observed.
Impurities: traces of unidentified impurities are seen in the baseline.

Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Isotopic Labelling: the signal at the site of deuteration has collapsed to a small multiplet compared with the spectrum of unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 171.1211. $C_9H_{11}D_3NO_2$ $[M+H]^+$ (free base) requires *m/z* 171.1207. The deviation of 2.1 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 (99.1 %). 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 52.03, H 5.50, D 3.00, N 6.72 %
$C_9H_{10}D_3NO_2 \cdot HCl$	Requires:	C 52.30, H 5.36, D 2.92, N 6.78 %

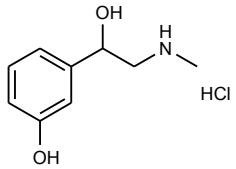
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

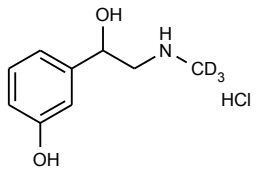
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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. This compound is intended for use as an analytical reference material and it is not for human administration. Structures are shown with relative stereochemistry unless otherwise specified.

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

BDG SYNTHESIS

Proton NMR Spectrum of Phenylephrine HCl (top) and Phenylephrine-d_3 HCl (bottom) in D_2O





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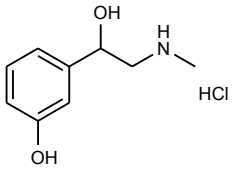

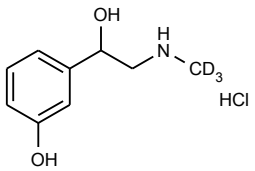

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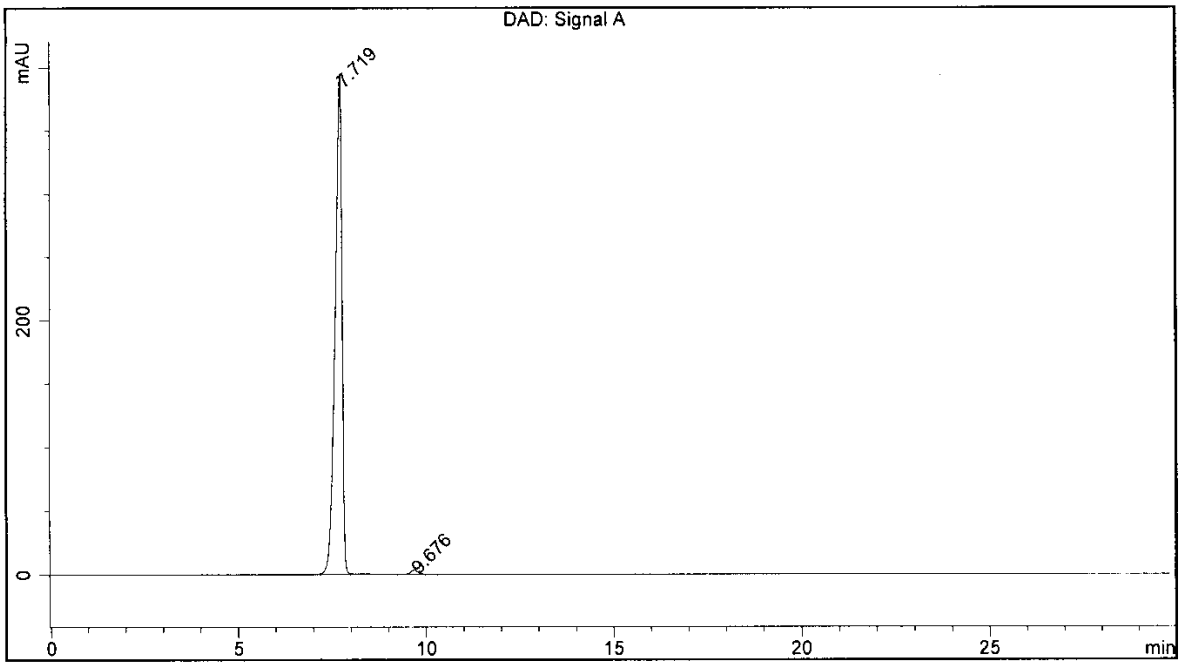
BDG - Analysis of Phenylephrine-d3 hydrochloride

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 55:45 Water : Methanol containing 5 mM Sodium Heptanesulphonate; adjusted to pH 3.0 with Phosphoric Acid
Flow Rate : 1.0 mL/min
Sample Solvent : Mobile Phase
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 280 nm

Sample Name	BDG 8538.4	Instrument	AnalyticalLC01
Acquisition	01/05/2008, 19:06:07	Method (rev.)	LC10254a (4)
Sequence	BDG_01May2008e	Vial Position	2
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	7.72 min	394.4310	4984.1243	0.1854 min	99.090 %
2	9.68 min	3.4272	45.7865	0.2059 min	0.910 %