BDG SYNTHESIS

Certificate of Analysis

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

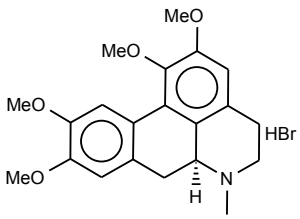
Barry Dent

Barry R. Dent, PhD, Director
13 November 2013

Name: **Glaucine HBr**

CAS Number: 5996-06-5

Structure:



Molecular Weight: $C_{21}H_{25}NO_4 \cdot HBr = 436.34$

Lot Number: BDG 14187.1

Appearance: Pale tan, crystalline solid

Corrected Purity: 99.2 % (HPLC) - 0.3 % (diethyl ether) - 0.3 % (water) = 98.6 %

Re-test Date: 13 November 2015

Storage and Handling:

Temperature:	refrigerate for prolonged storage; may be handled and shipped at ambient temperature.
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.

Mailing:
BDG Synthesis Limited
PO Box 38627, Wellington Mail Centre,
Wellington 5045, New Zealand.

Shipping:
BDG Synthesis Limited
Gracefield Research Centre, Building F,
69 Gracefield Rd, Lower Hutt 5010, New Zealand.

Phone: + 64 4 569 0520
Fax: + 64 4 569 0521

info@bdg.co.nz
www.bdg.co.nz

Identity and Purity

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 diethyl ether (0.3 % *w/w*) is observed.
Impurities: a trace of an unidentified impurity is seen in the baseline.

Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.

High-resolution Mass Spectrum (TOF MS ES+)

Found *m/z* 356.1857. $C_{21}H_{26}NO_4$ $[M+H]^+$ (free base) requires *m/z* 356.1862. The deviation of 1.4 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (99.2 %). 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.

Karl-Fischer Analysis

	Found:	H_2O 0.3 %
$C_{21}H_{25}NO_4 \cdot HBr$	Requires:	H_2O 0.0 %

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.

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.

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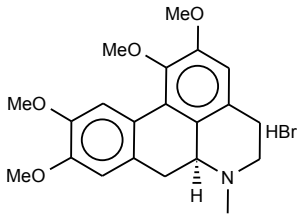
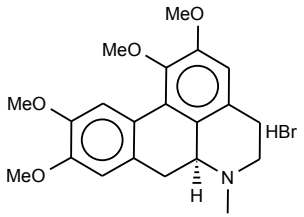
Proton NMR Spectrum of Glaucine HBr in $CDCl_3$



Lot Number: BDG 14187.1

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Glaucine HBr in $CDCl_3$



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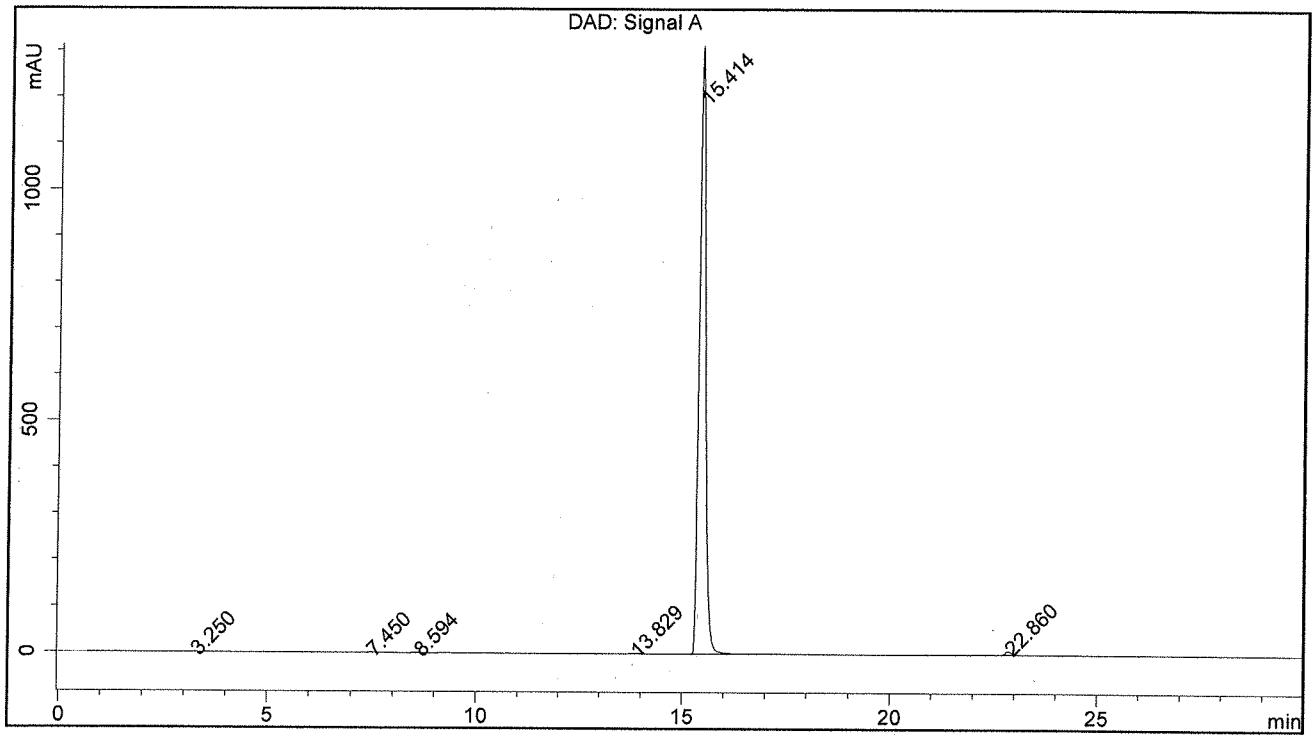
BDG - Analysis of Glaucine HBr

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase A : 70:30 20 mM diPotassium Hydrogen Phosphate pH=7.0 : Acetonitrile
Mobile Phase B : 30:70 20 mM diPotassium Hydrogen Phosphate pH=7.0 : Acetonitrile
Gradient (A:B) : T0=100:0, T20=0:100, T24=0:100, T26=100:0, T30=100:0
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 14187.1	**Instrument**	AnalyticalLC01
Acquisition	13/11/2013, 18:10:24	**Method (rev.)**	LC10548a (6)
Sequence	BDG_13Nov2013a	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.25 min	0.9973	5.6426	0.0860 min	0.039 %
2	7.45 min	1.0141	10.3304	0.1548 min	0.071 %
3	8.59 min	0.6603	5.8360	0.1191 min	0.040 %
4	13.83 min	0.7042	8.7426	0.1728 min	0.060 %
5	15.41 min	1309.7431	14346.0382	0.1678 min	99.184 %
6	22.86 min	8.2179	87.5015	0.1642 min	0.605 %