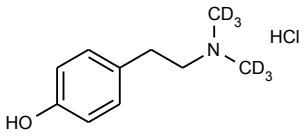
BDG SYNTHESIS

Certificate of Analysis

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

Barry Dent

Barry R. Dent, PhD, Director
22 January 2011

Name:	**Hordenine-d_6 HCl**
CAS Number:	6027-23-2 (unlabelled)
Structure:	
Molecular Weight:	$C_{10}H_9D_6NO \cdot HCl$ = 207.73
Lot Number:	BDG 10986.1
Appearance:	White, crystalline solid
Corrected Purity:	96.0 % (HPLC) - 0.2 % (2-propanol) = 95.8 %
Isotopic Purity:	Under 0.5 % d_0
Re-test Date:	22 January 2016
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.

Identity and Purity

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 what would be expected for unlabelled material, indicating clean deuteration.
Residual Solvents: a small amount of 2-propanol (0.2 % *w/w*) is observed.
Impurities: an unidentified impurity is observed.

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 what would be expected for unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 172.1616. $C_{10}H_{10}D_6NO$ $[M+H]^+$ requires *m/z* 172.1603. The deviation of 7.4 ppm is somewhat outside normally accepted limits for the establishment of identity by HRMS, and the mass spectral data should be considered in conjunction with other identity criteria. No signal for d_0 material was seen (detection limit about 0.5 %).

HPLC

A somewhat broadened, slightly tailing peak is observed (96.0 %). 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 58.08, H 4.96, D 5.96, N 6.69 %
$C_{10}H_9D_6NO{\cdot}HCl$	Requires:	C 57.82, H 4.85, D 5.82, N 6.74 %

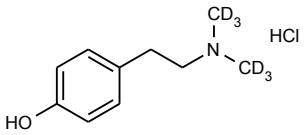
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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.

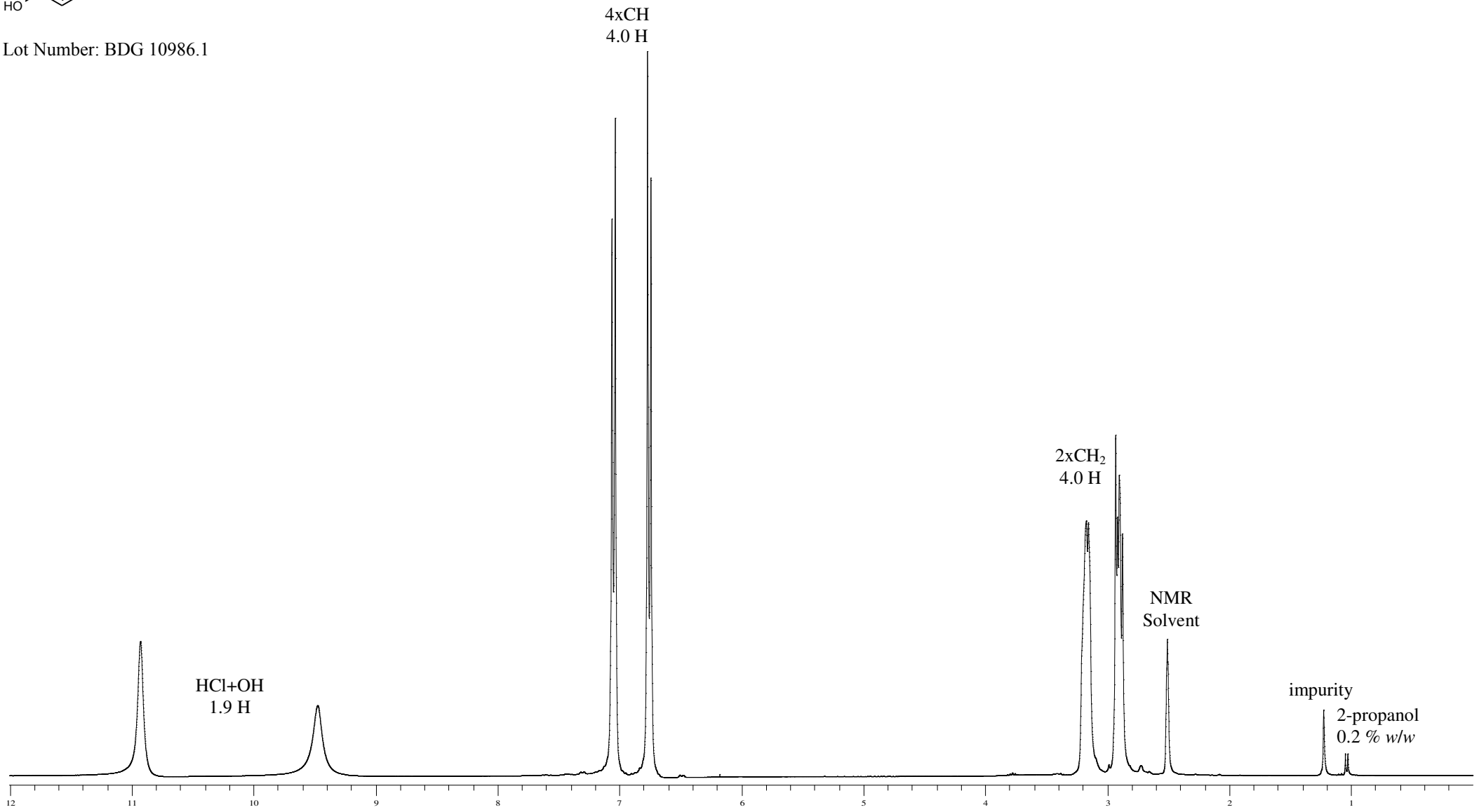
The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.

BDG SYNTHESIS

Proton NMR Spectrum of Hordenine-d_6 HCl in DMSO-d_6



Lot Number: BDG 10986.1



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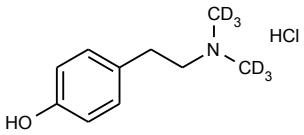
Carbon-13 NMR Spectrum of Hordenine-d_6 HCl in DMSO-d_6

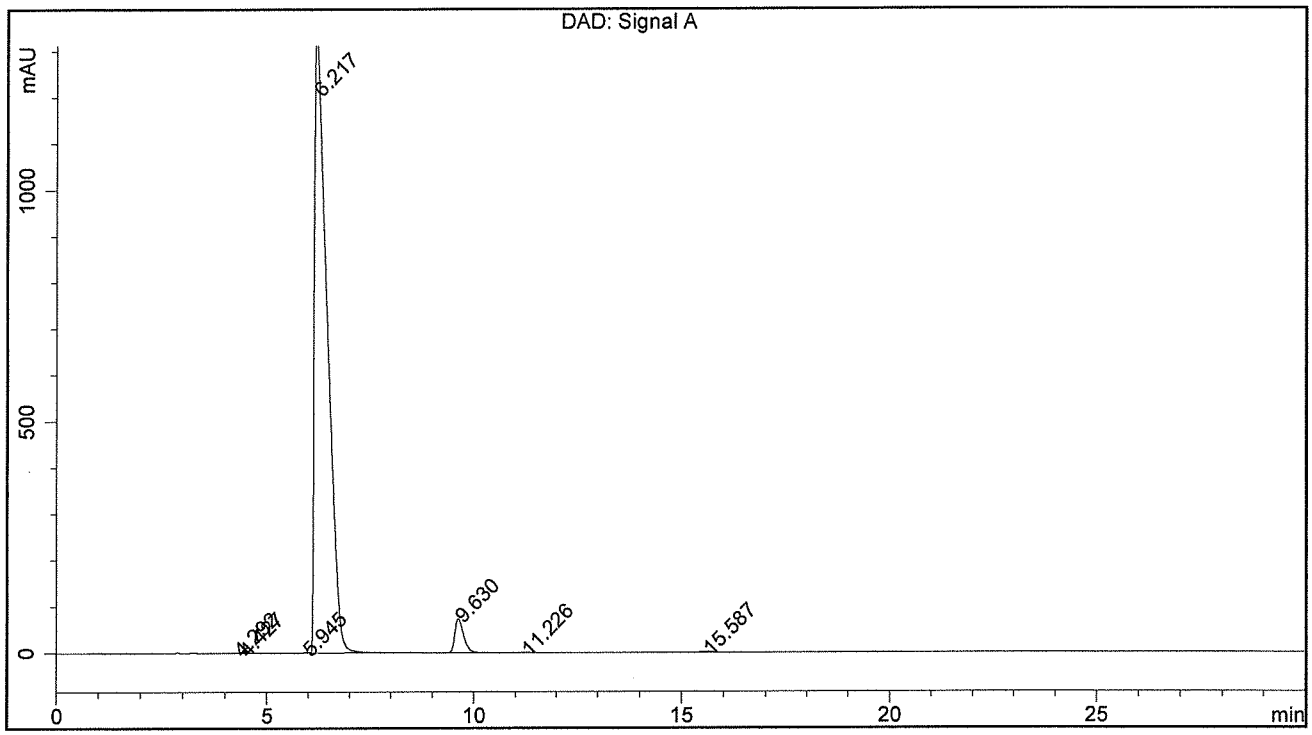


Lot Number: BDG 10986.1

BDG - Analysis of Hordenine-d6 HCl

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 95:5 25mM Potassium diHydrogen Phosphate pH=3.0 : Acetonitrile
Flow Rate : 1.0 mL/min
Sample Solvent : Initial Mobile Phase
Injection Volume : 10 uL
Column Temperature : 20C
Detection : UV at 222 nm

Sample Name	BDG 10986.1	**Instrument**	AnalyticalLC01
Acquisition	22/01/2011, 12:34:25	**Method (rev.)**	LC10420b (9)
Sequence	BDG_22Jan2011e - Reprocessed	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.29 min	0.9134	6.0346	0.0971 min	0.020 %
2	4.43 min	1.0258	15.4837	0.2021 min	0.052 %
3	5.94 min	1.7193	15.0836	0.1264 min	0.051 %
4	6.22 min	1340.7527	28633.3752	0.3192 min	96.006 %
5	9.63 min	72.7900	1091.6401	0.2330 min	3.660 %
6	11.23 min	0.6122	10.2724	0.2337 min	0.034 %
7	15.59 min	2.2984	52.6079	0.3370 min	0.176 %