



## BDG SYNTHESIS

### Certificate of Analysis

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

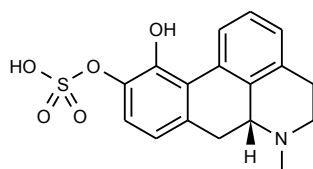
*Neil Beare*

Neil Beare, PhD, Director  
7 September 2017

**Name:** Apomorphine Sulfate

**CAS Number:** 1330267-27-0

**Structure:**



**Molecular Weight:**  $C_{17}H_{17}NO_5S = 347.39$

**Lot Number:** BDG 15752.3

**Appearance:** White, crystalline solid

**Corrected Purity:** 98.8 % (HPLC) - 1.1 % (methanol) - 1.3 % (dimethylformamide) - 0.2 % (water) = 96.2 %

**Re-test Date:** 7 September 2018

**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.  
Residual Solvents: small amounts of methanol (1.1 % w/w) and dimethylformamide (1.3 % w/w) are 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.

### High-resolution Mass Spectrum (TOF MS ES+)

Found  $m/z$  348.0903.  $C_{17}H_{18}NO_5S$   $[M+H]^+$  requires  $m/z$  348.0906. The deviation of 0.9 ppm is within normally accepted limits for the establishment of identity by HRMS.

### HPLC

A sharp, symmetrical peak is observed (98.8 %). 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 57.85, H 5.11, N 4.19 %
$C_{17}H_{17}NO_5S \cdot 0.2H_2O$	Requires:	C 58.17, H 5.00, N 3.99 %
$C_{17}H_{17}NO_5S$	Requires:	C 58.78, H 4.93, N 4.03 %

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 <sub>2</sub> O 0.2 %
$C_{17}H_{17}NO_5S \cdot 0.2H_2O$	Requires:	H <sub>2</sub> O 1.0 %

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.

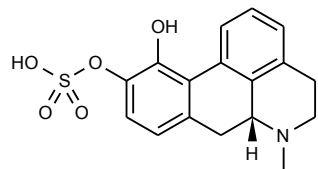
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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.

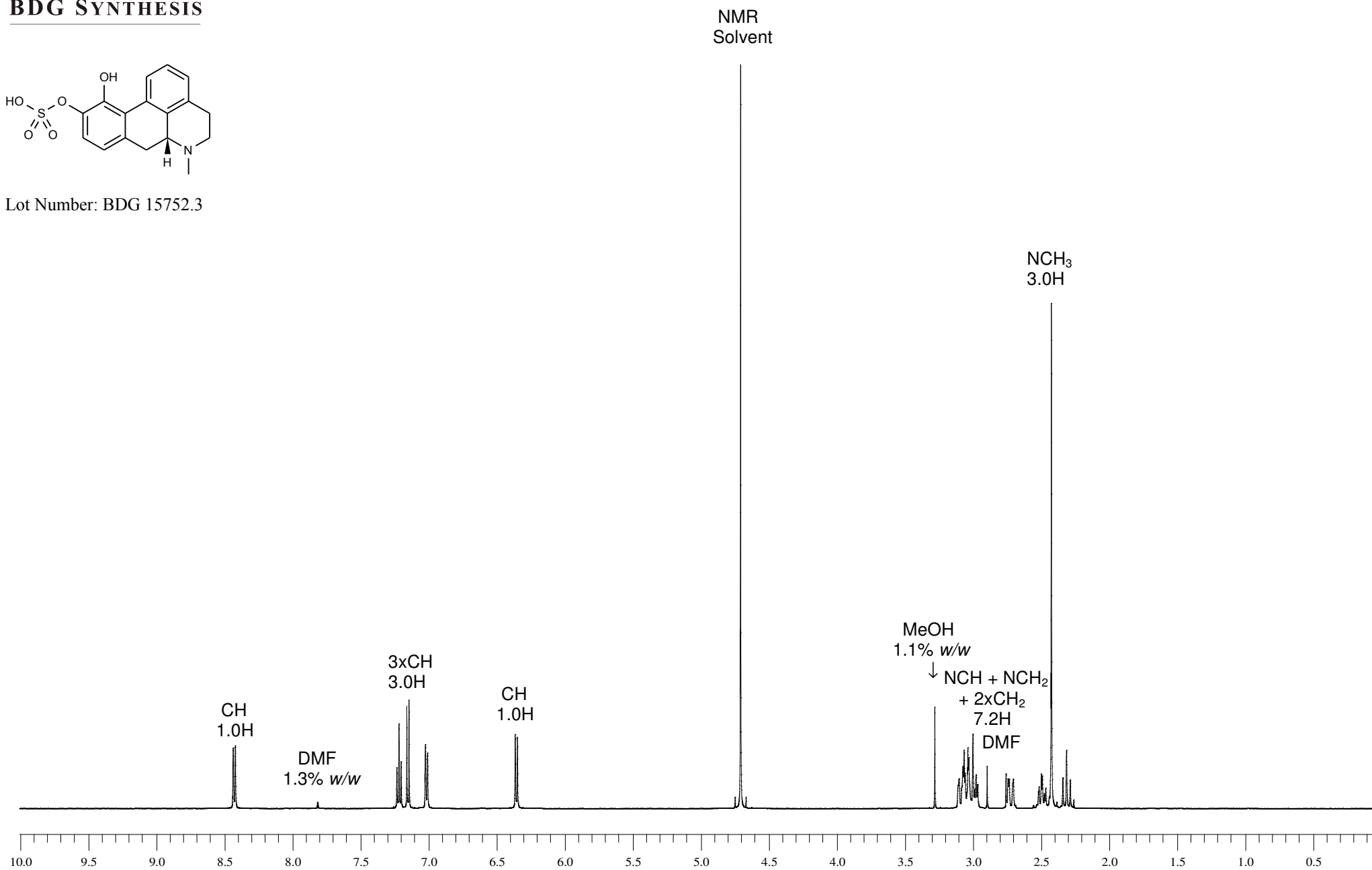


# Proton NMR Spectrum of Apomorphine Sulfate in D<sub>2</sub>O + NaOD

**BDG SYNTHESIS**



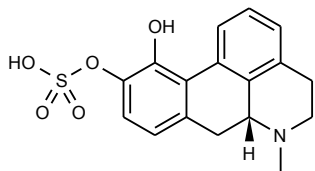
Lot Number: BDG 15752.3



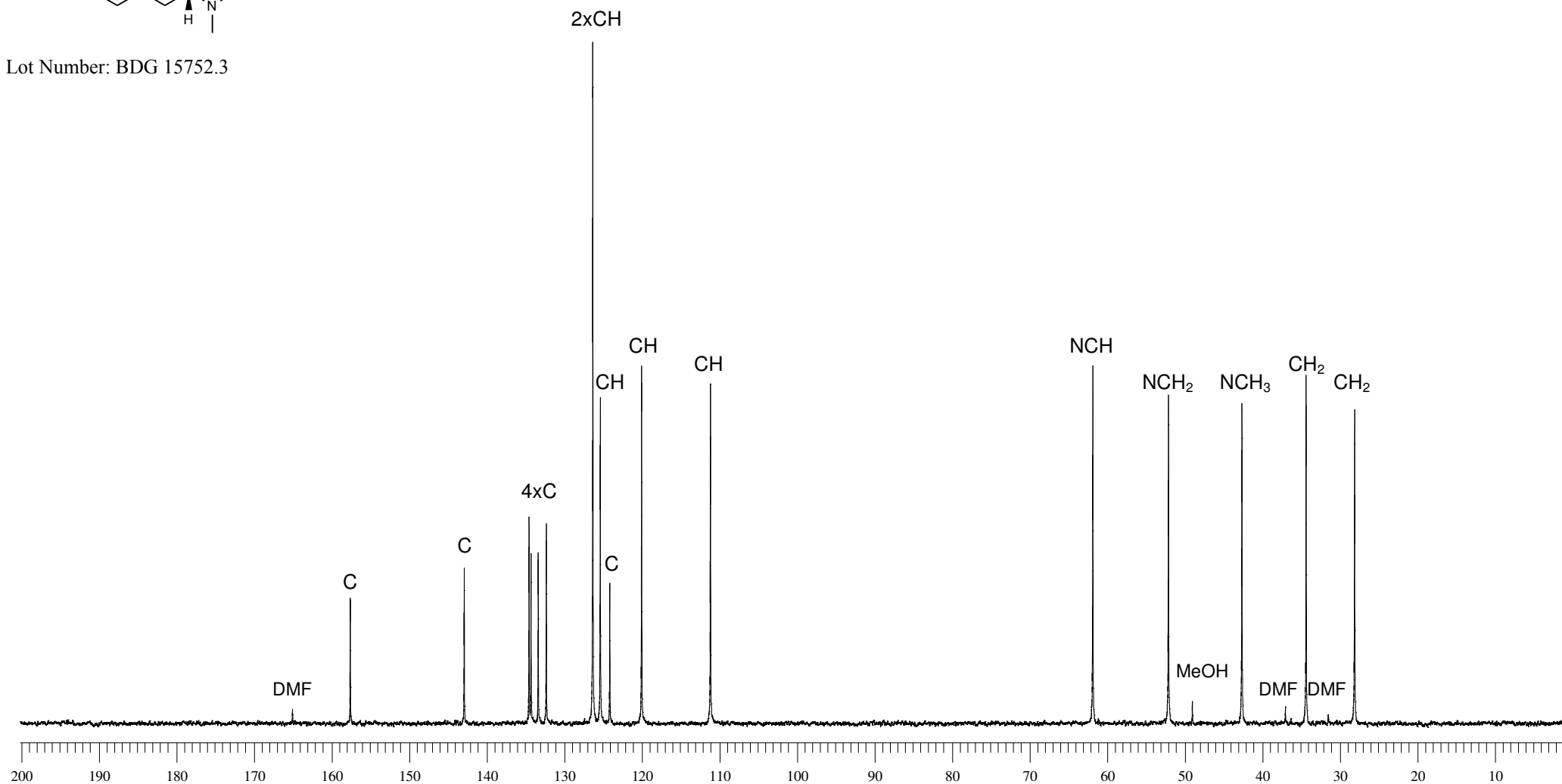


# Carbon-13 NMR Spectrum of Apomorphine Sulfate in D<sub>2</sub>O + NaOD

**BDG SYNTHESIS**



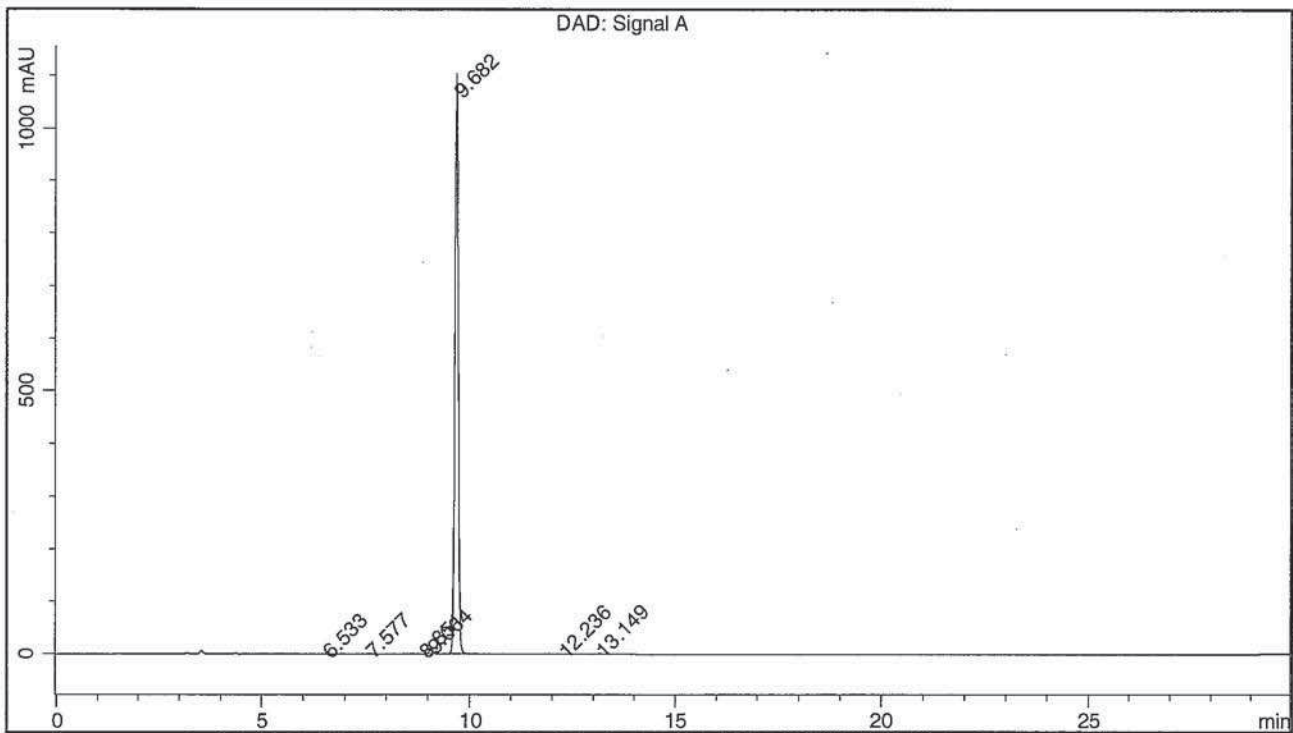
Lot Number: BDG 15752.3



BDG - Analysis of Apomorphine Sulfate

Column : Phenomenex Luna C18 (2) 5um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm  
 Mobile Phase A : 85:15 5 mM Sodium Heptane Sulphonate pH=2.2 (H3PO4) : Acetonitrile  
 Mobile Phase A : 50:50 5 mM Sodium Heptane Sulphonate pH=2.2 (H3PO4) : Acetonitrile  
 Gradient (A:B) : T0=100:0, T20=0:100, T25=0:100, T26=100:0, T30=100:0  
 Flow Rate : 1 mL/min  
 Column Temperature : 35 C  
 Sample Solvent : 1% Acetic Acid  
 Injection Volume : 10 uL  
 Detection : UV at 280 nm

<b>Sample Name</b>	BDG 15752.3	<b>Instrument</b>	AnalyticalLC01
<b>Acquisition</b>	07/09/2017, 14:47:24	<b>Method (rev.)</b>	LC10717a ( 6 )
<b>Sequence</b>	BDG_07Sep2017b - Reprocessed	<b>Vial Position</b>	1
<b>Operator</b>	solvation010\cerityadmin	<b>Injection</b>	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	6.53 min	0.3615	4.1745	0.1507 min	0.067 %
2	7.58 min	1.5745	5.4878	0.0527 min	0.088 %
3	8.85 min	0.3809	5.5755	0.1908 min	0.090 %
4	9.06 min	8.1819	48.2890	0.0889 min	0.776 %
5	9.68 min	1107.1776	6151.2103	0.0868 min	98.820 %
6	12.24 min	0.3837	3.3487	0.1238 min	0.054 %
7	13.15 min	1.3947	6.5487	0.0748 min	0.105 %