



BDG SYNTHESIS

Certificate of Analysis

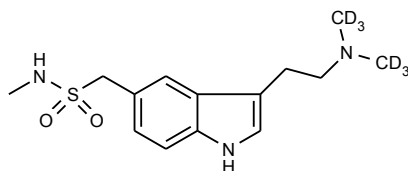
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Barry Dent

Barry R. Dent, PhD, Director
9 October 2013

Name: Sumatriptan-d₆
CAS Number: 103628-46-2 (unlabelled)

Structure:



Molecular Weight: C₁₄H₁₅D₆N₃O₂S = 301.44
Lot Number: BDG 12854.2
Appearance: White, crystalline solid
Corrected Purity: 99.4 % (HPLC) - 1.0 % (ethanol) = 98.4 %
Isotopic Purity: Under 0.5 % d₀
Re-test Date: 9 October 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.

Isotopic Labelling: signals at the site of deuteration are absent, compared with the spectrum of unlabelled material, indicating clean deuteration.

Residual Solvents: a small amount of ethanol (1.0 % w/w) is observed.

Impurities: no significant impurities are evident in the spectrum.

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 site 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 302.1805. $C_{14}H_{16}D_6N_3O_2S$ $[M+H]^+$ requires m/z 302.1809. The deviation of 1.3 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 broad, slightly tailing peak is observed (99.4 %). 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 55.87, H 5.08, D 4.06, N 13.75 %
$C_{14}H_{15}D_6N_3O_2S$	Requires:	C 55.78, H 5.02, D 4.01, N 13.94 %

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).

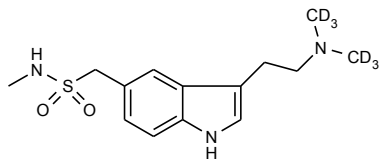
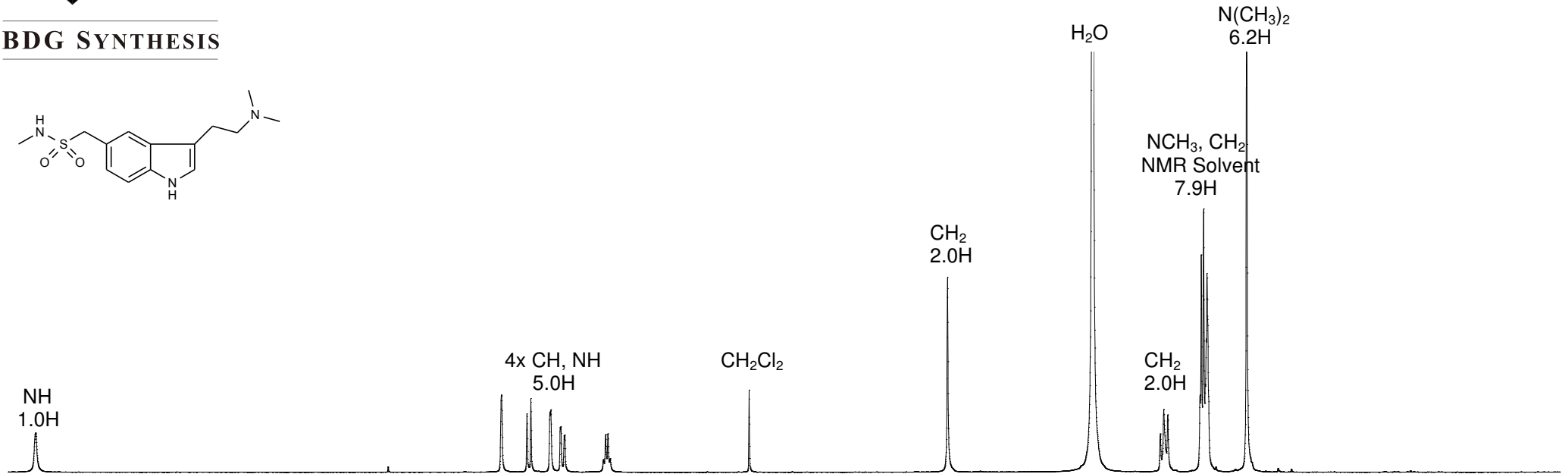
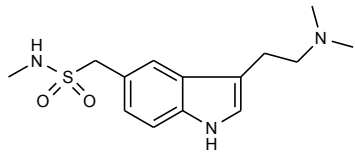
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

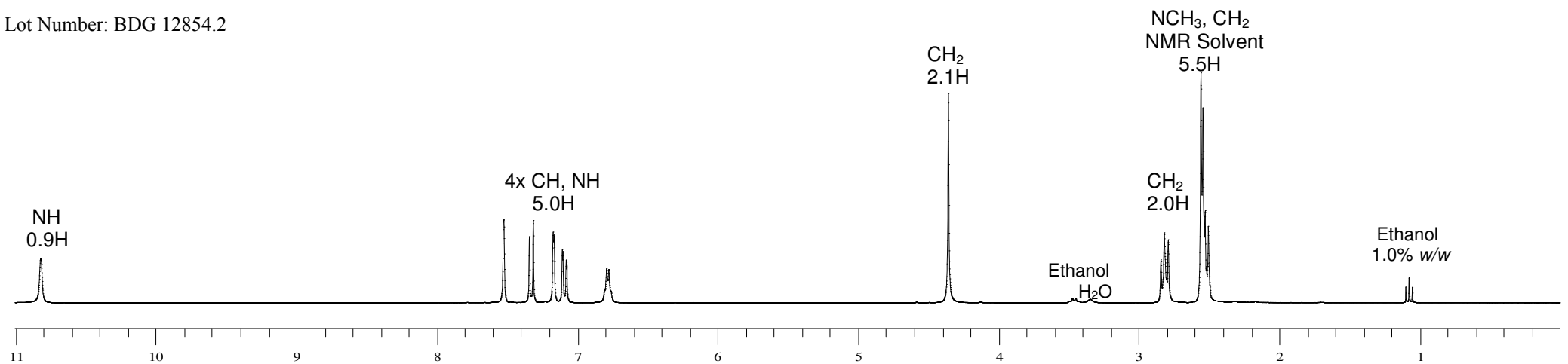


Proton NMR Spectrum of Sumatriptan (top) and Sumatriptan-d₆ (bottom) in DMSO-d₆

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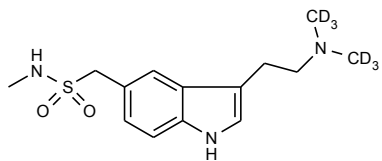
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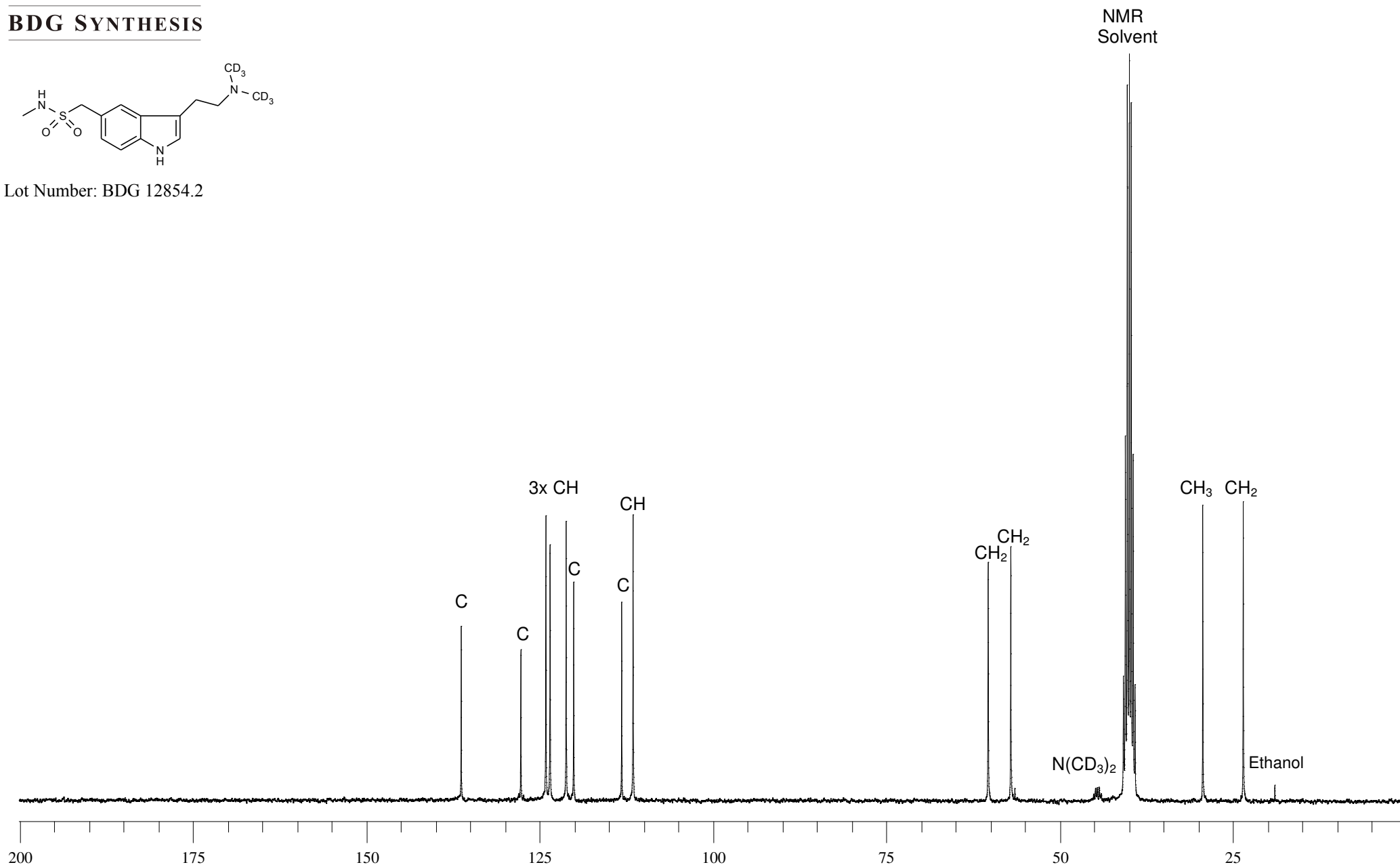


Carbon-13 NMR Spectrum of Sumatriptan-d₆ in DMSO-d₆

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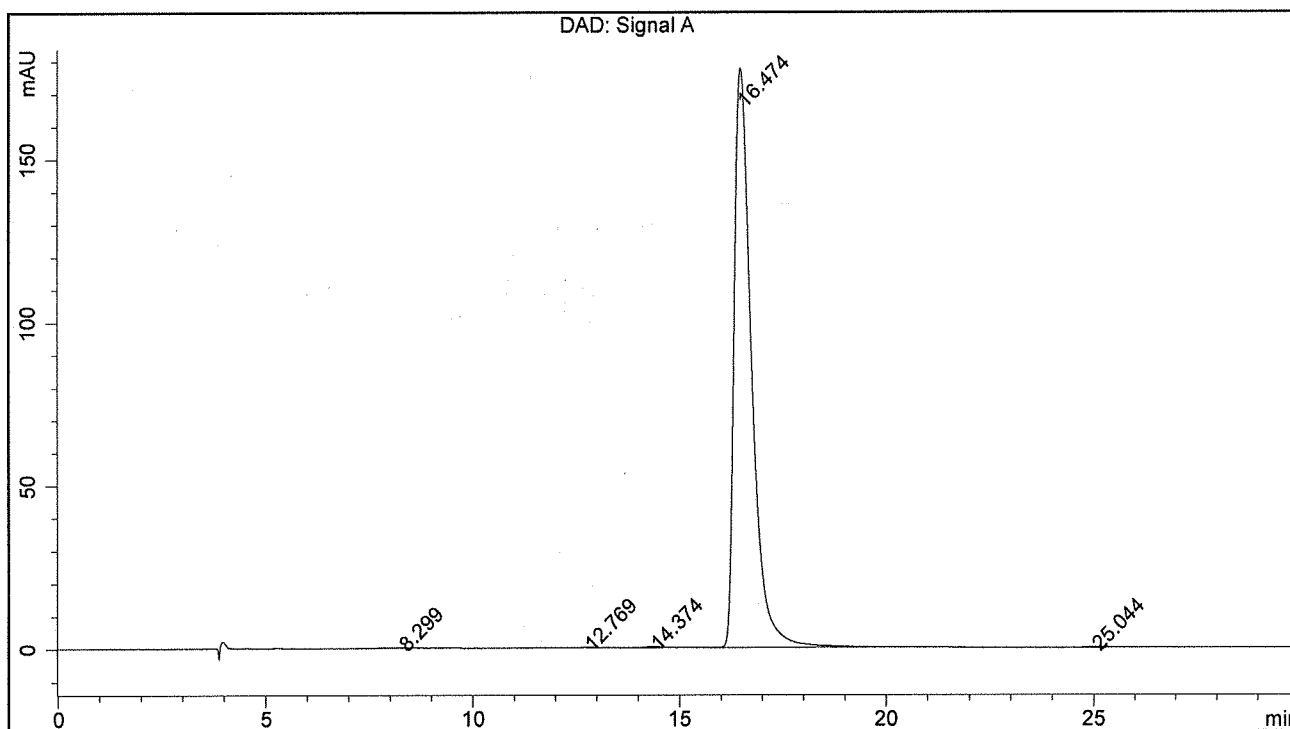
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BDG - Analysis of Sumatriptan-d6

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 92:8 Dilute EP Buffer pH=6.5 : Acetonitrile
 Buffer = 2.5 mM Dibutylamine + 6.25 mM NaH₂PO₄ + 2.1 mM Phosphoric Acid to pH=6.5 (NaOH)
 Injection Volume : 10 uL
 Flow Rate : 1.0 mL/min
 Column Temperature : 25C
 Sample Solvent : 70:30 Water: Acetonitrile
 Detection : UV at 282 nm

Sample Name	BDG 12854.2	Instrument	AnalyticalLC01
Acquisition	09/10/2013, 12:04:27	Method (rev.)	LC10456a (9)
Sequence	BDG_09Oct2013a - Reprocessed	Vial Position	31
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	8.30 min	0.1381	6.5583	0.5740 min	0.126 %
2	12.77 min	0.1612	3.1548	0.2604 min	0.060 %
3	14.37 min	0.3939	10.5159	0.3475 min	0.201 %
4	16.47 min	177.2766	5191.1443	0.4456 min	99.405 %
5	25.04 min	0.2587	10.8424	0.5090 min	0.208 %