BDG SYNTHESIS

Certificate of Analysis

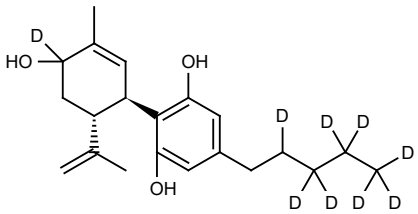
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Neil Beare, PhD, Director
9 September 2019

Name: **6-Hydroxycannabidiol-d_9**

CAS Number: none

Structure:



Molecular Weight: $C_{21}H_{21}D_9O_3$ = 339.52

Lot Number: BDG 14408.2

Appearance: Off-white, crystalline solid

Corrected Purity: 99.8 % (HPLC) - 0.5 % (ethyl acetate) - 0.6 % (hexanes) = 98.7 %

Isotopic Purity: Under 0.5 % d_0

Re-test Date: 9 September 2024

Storage and Handling:

Temperature:	freeze (-20 °C) 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.

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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: signals at the sites of deuteration are greatly diminished, compared with the spectrum of unlabelled material.
Residual Solvents: small amounts of ethyl acetate (0.5 % *w/w*) and hexanes (0.6 % *w/w*) are 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 sites of deuteration have collapsed to small multiplets, compared with the spectrum of unlabelled material.

High-resolution Mass Spectrum (TOF MS ES+)

Found *m/z* 340.2845. $C_{21}H_{22}D_9O_3$ $[M+H]^+$ requires *m/z* 340.2838. 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 %) although small signals were observed for d_1 and d_2 material.

HPLC

A sharp, symmetrical peak is observed (99.8 %). 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 74.35, H 6.50, D 5.57 %
$C_{21}H_{21}D_9O_3$	Requires:	C 74.29, H 6.23, D 5.34 %

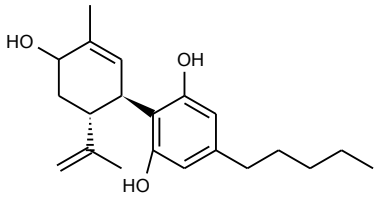
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

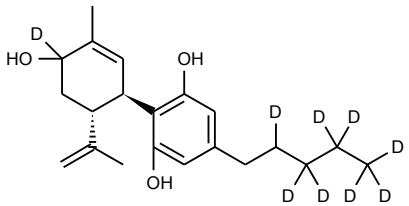
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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 6-Hydroxycannabidiol (top) and 6-Hydroxycannabidiol-d_9 (bottom) in $CDCl_3$

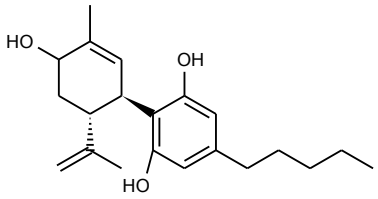


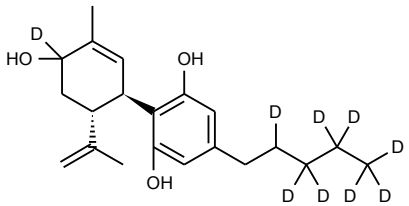


Lot Number: BDG 14408.2

BDG SYNTHESIS

Carbon-13 NMR Spectrum of 6-Hydroxycannabidiol (top) and 6-Hydroxycannabidiol-d_9 (bottom) in $CDCl_3$





Lot Number: BDG 14408.2

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Acq. Operator   : Bruce Hamilton                  Seq. Line :   4
Acq. Instrument : Instrument 1                     Location : Vial 1
Injection Date  : 9/9/2019 4:03:13 PM                  Inj :   1
                                                 Inj Volume : 10 µl
Acq. Method     : C:\CHEM32\1\METHODS\2018\LC20101F.M
Last changed    : 9/9/2019 2:25:21 PM by Bruce Hamilton
Analysis Method : C:\CHEM32\1\METHODS\2018\LC20101F.M
Last changed    : 9/10/2019 9:49:46 AM by Bruce Hamilton
                  (modified after loading)
Method Info     : BDG - Analysis of 6-Hydroxycannabidiol-d9
                  Column  : Phenomenex Luna C18(2) 5 um  250 x 4.6 mm
                  Guard : Phenomenex SecurityGuard C18 4 x 3 mm
                  Mobile Phase A : 30:70 Water : Methanol
                  Mobile Phase C : Methanol
                  Gradient (A:B) : T0=100:0, T15=0:100, T25=0:100, T26=100:0, T30=100:0
                  Sample Solvent : 30:70 Water : Methanol,   Detection : UV 212 nm,
                  Flow : 1 ml/min.,   Column Temperature : 35 C,    Injection : 10 ul.
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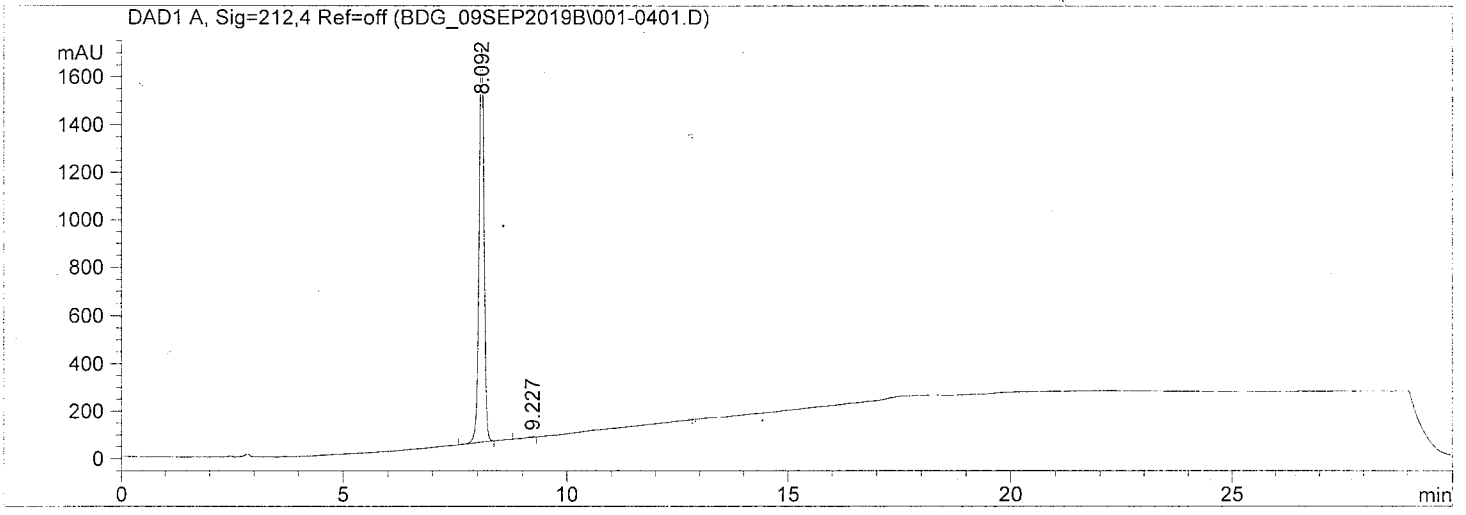


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                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=212,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.092 BB    0.1234 1.29436e4  1652.64014  99.8204
   2   9.227 BB    0.1233   23.29258    2.85277   0.1796

Totals :                  1.29669e4  1655.49291


                          *** End of Report ***
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