BDG SYNTHESIS

Certificate of Analysis

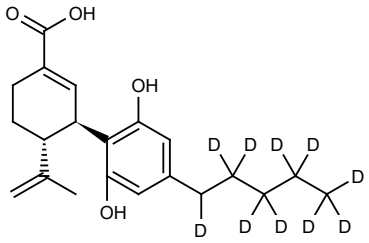
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Neil Beare, PhD, Director
25 October 2019

Name: **7-Nor-7-carboxycannabidiol-d_{10}**

CAS Number: none

Structure:



Molecular Weight: $C_{21}H_{18}D_{10}O_4$ = 354.51

Lot Number: BDG 11657

Appearance: White, crystalline solid

Corrected Purity: 100.0 % (HPLC) - 1.0 % (water) = 99.0 %

Isotopic Purity: Under 0.5 % d_0

Re-test Date: 25 October 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.

• Custom synthesis of analytical reference standards, metabolites, stable isotope labelled compounds • Contract research • BDG Synthesis is a division of B Dent Global Limited

Mailing:
BDG Synthesis
PO Box 38 627, Wellington Mail Centre,
Wellington, New Zealand.

Shipping:
BDG Synthesis
Gracefield Research Centre, Building F,
Gracefield Road, Lower Hutt, 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.
Isotopic Labelling: signals at the sites of deuteration are greatly diminished, compared with the spectrum of unlabelled material, indicating clean deuteration.
Residual Solvents: no residual solvents 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, indicating clean deuteration.

High-resolution Mass Spectrum (TOF MS ES+)

Found *m/z* 355.2688. $C_{21}H_{19}D_{10}O_4$ $[M+H]^+$ requires *m/z* 355.2694. The deviation of 1.7 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 sharp, symmetrical peak is observed (100.0 %). 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 70.31, H 5.08, D 5.72 %
$C_{21}H_{18}D_{10}O_4 \cdot 0.2H_2O$	Requires:	C 70.43, H 5.18, D 5.62 %, H_2O 1.01 %
$C_{21}H_{18}D_{10}O_4$	Requires:	C 71.15, H 5.12, D 5.68 %

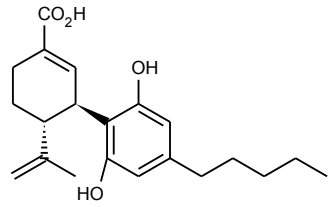
The elemental analyses fall slightly outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the "best-fit" hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we recommend that the "best-fit" water content be used when determining corrected purity.

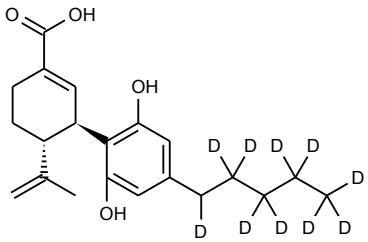
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

BDG SYNTHESIS

Proton NMR Spectrum of 7-Nor-7-carboxycannabidiol (top) and 7-Nor-7-carboxycannabidiol-d_{10} (bottom) in Methanol-d_4

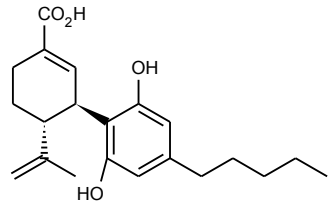


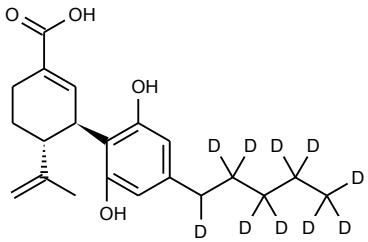


Lot Number: BDG 11657

BDG SYNTHESIS

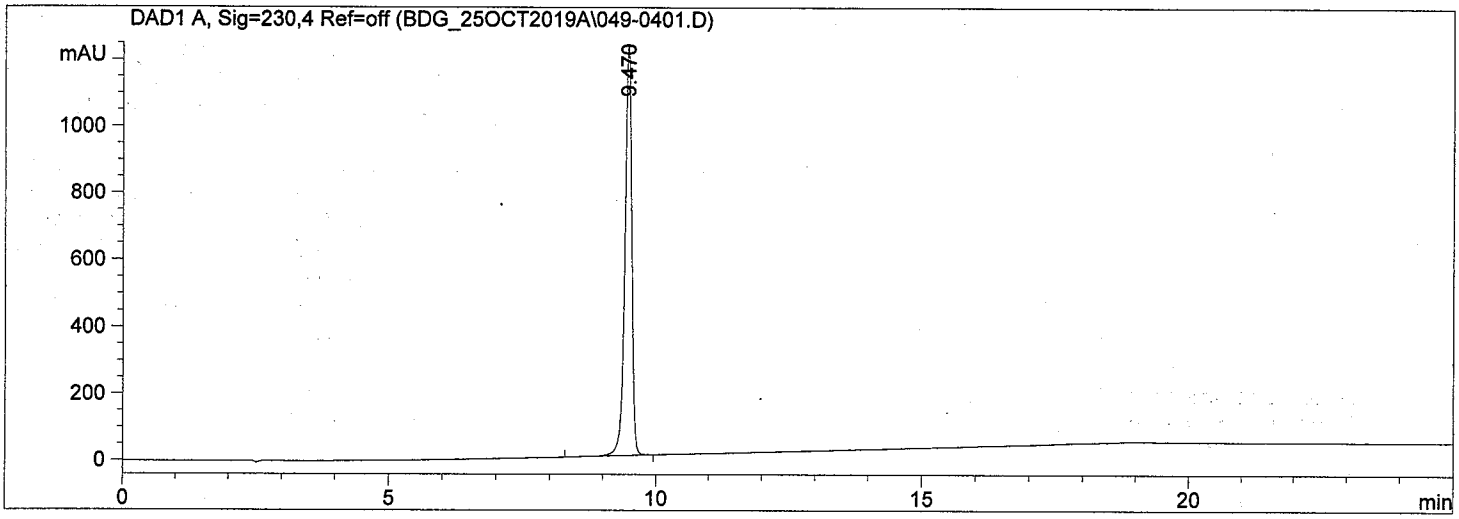
Carbon-13 NMR Spectrum of 7-Nor-7-carboxycannabidiol (top) and 7-Nor-7-carboxycannabidiol-d_{10} (bottom) in Methanol-d_4





Lot Number: BDG 11657

Acq. Operator : Bruce Hamilton
Acq. Instrument : Instrument 1
Injection Date : 10/25/2019 2:21:40 PM
Seq. Line : 4
Location : Vial 49
Inj : 1
Inj Volume : 10 µl
Acq. Method : C:\CHEM32\1\METHODS\2018\LC20101M.M
Last changed : 10/25/2019 12:50:43 PM by Bruce Hamilton
Analysis Method : C:\CHEM32\1\METHODS\2018\LC20101M.M
Last changed : 10/25/2019 3:54:20 PM by Bruce Hamilton
(modified after loading)
Method Info : BDG - Analysis of 7-Nor-7-Carboxycannabidiol-d10
Column : Phenomenex Luna C18(2) 5 um 250 x 4.6 mm
Guard : Phenomenex SecurityGuard C18 4 x 3 mm
Mobile Phase A : 30:70:0.05 Water : Methanol : Trifluoroacetic Acid
Mobile Phase C : 10:90:0.05 Water : Methanol : Trifluoroacetic Acid
Gradient (A:C) : T0=100:0, T15=0:100, T22=0:100, T23=100:0, T27=100:0
Sample Solvent : 30:70 Water : Methanol, Detection : UV 230 nm,
Flow : 1 ml/min., Column Temperature : 35 C, Injection : 10 ul.



Area Percent Report

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

Signal 1: DAD1 A, Sig=230,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.470	BB	0.1362	1.10003e4	1231.91064	100.0000
Totals :				1.10003e4	1231.91064	

*** End of Report ***