

Certificate of Analysis

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

Leil Beare

Neil Beare, PhD, Director 23 November 2020

Name: Leptosperin

CAS Number: 1431845-42-9

Structure: OH

Molecular Weight: $C_{22}H_{32}O_{15} = 536.48$

Lot Number: BDG 11870

Appearance: White, crystalline solid

Corrected Purity: 99.7 % (HPLC) - 0.3 % (methanol) - 3.3 % (water) = 96.2 %

Re-test Date: 23 November 2021

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.

BDG Synthesis

Gracefield Research Centre, Building F,

Gracefield Road, Lower Hutt, New Zealand.

• Contract research • BDG Synthesis is a division of B Dent Global Limited

Identity and Purity

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.

Residual Solvents: a small amount of methanol (0.3 % w/w) is observed.

Impurities: a trace of an unidentified impurity is 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 559.1638. $C_{22}H_{32}O_{15}Na$ [M+Na]⁺ requires m/z 559.1639. The deviation of 0.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (99.7 %). 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 47.59, H 6.04 %

C₂₂H₃₂O₁₅·1.0H₂O Requires: C 47.65, H 6.18 %, H₂O 3.25 %

 $C_{22}H_{32}O_{15}$ Requires: C 49.25, H 6.01 %

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. In the absence of a Karl-Fischer water analysis, we recommend that the "best-fit" water content be used when determining corrected purity.

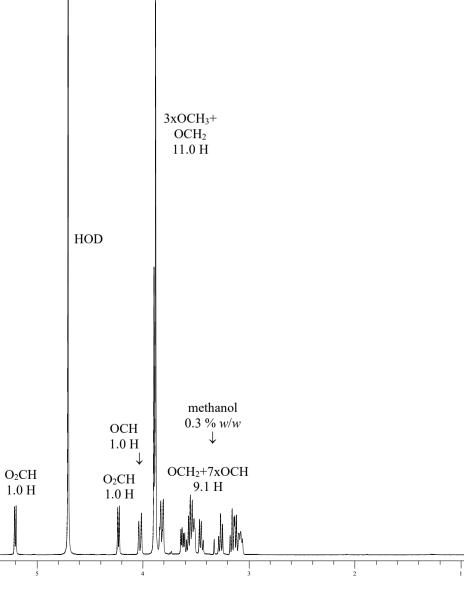
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

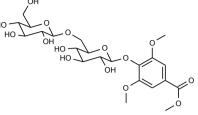
2xCH 2.0 H

BDG SYNTHESIS

Lot Number: BDG 11870

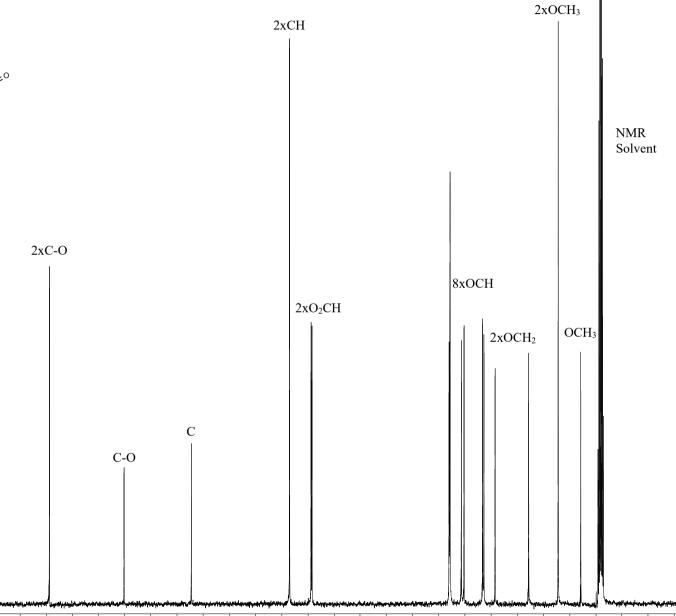






C=O

Lot Number: BDG 11870



Injection Date : 11/23/20 1:48:18 PM Seq. Line: 1 Sample Name : BDG 11870 Location: Vial 1 Acq. Operator : Bruce Hamilton Inj: 1 Inj Volume : 10 μl

: C:\HPCHEM\1\METHODS\LC30037A.M Acq. Method

Last changed : 11/23/20 1:47:10 PM by Bruce Hamilton

Analysis Method: C:\HPCHEM\1\METHODS\LC30037A.M

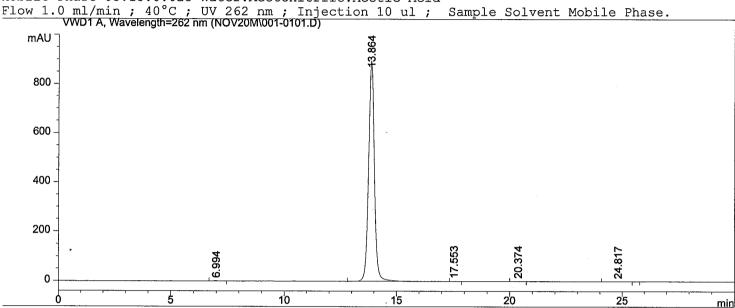
Last changed : 11/23/20 2:46:25 PM by Bruce Hamilton

(modified after loading)

BDG - Analysis of Leptosin

Phenomenex Luna C18(2) 250 x 4.6mm 5um + SecurityGuard C18 RP;

Mobile Phase 85:15:0.025 Water: Acetonitrile: Acetic Acid



Area Percent Report

Sorted By Signal Multiplier 1.0000 1.0000 Dilution

Signal 1: VWD1 A, Wavelength=262 nm

#	RetTime [min]		Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.994	VV	0.1630	17.23624	1.42618	0.1055
2	13.864	VBAS	0.2761	1.62903e4	896.23834	99.7276
3	17.553	VV X	0.2390	5.27559	2.64198e-1	0.0323
4	20.374	VV X	0.2977	13.34474	5.37354e-1	0.0817
5	24.817	T VV	0.5233	8.63997	2.10303e-1	0.0529

!otals : 1.63348e4 898.67637

Results obtained with enhanced integrator!

*** End of Report ***