Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

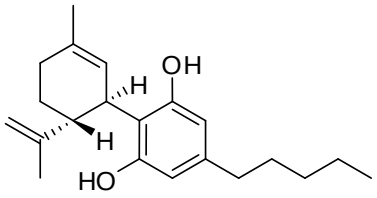
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D512c: Cannabidiol

Report ID: D512c.2024.01 (Bottled 211012)

Chemical Formula: $C_{21}H_{30}O_2$

Molecular Weight: 314.5 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
21-D-04	**13956-29-1**	**99.7 ± 0.4%**

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

IUPAC Name: 2-[(1R,6R)-6-Isopropenyl-3-methyl-2-cyclohexen-1-yl]-5-pentyl-1,3-benzenediol

Expiration of certification: The property values are valid till 25 January 2027, three years from the date of re-certification provided the **unopened** material is handled and stored in accordance with the recommendations below. The material as issued in the unopened container and stored as recommended below should be suitable for use beyond this date, subject to confirmation of batch stability from the issuing body. The expiry date/shelf life does not apply to sample bottles that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

Description: Off-white powder sourced from an external supplier, certified for identity and purity by NMIA. Packaged in amber glass bottles with a septum and crimped aluminium cap.

Intended use: This certified reference material is suitable for use as a primary calibrator.

Instructions for use: Equilibrate the bottled material to room temperature before opening.

Recommended storage: When not in use, this material should be stored at or below 4 ºC in a closed container in a dry, dark area.

Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance approach all impurities are quantified as a mass fraction and subtracted from 100%.

Stability: This material has demonstrated stability over a minimum period of three years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual and accelerated stability trials, the latter conducted at 40 ºC and 75% humidity for a 14 day period. The long-term stability of the compound in solution has not been examined.

Homogeneity assessment: The homogeneity of the material was assessed using purity assay by GC-FID on ten randomly selected 1-2 mg sub samples of the material. The material was judged to be sufficiently homogeneous at this level of sampling as the variation in analysis results between samples was not significantly different at a 95% confidence level from that observed on repeat analysis of the same sample.

Safety: Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust. Refer to the provided safety data sheet.

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
25 January 2024

This report supersedes any issued prior to 25 January 2024.

NATA **a**ccreditation No. 198 / Corporate Site No. 14214.

Legal notice: Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

Purity = (100 % - I_{ORG}) x (100 % - I_{VOL} – I_{NVR}) Equation 1

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

Warning: **This material is sensitive to the quality of the silanised glass liner when injected at elevated temperature (~ 250 ºC) into a GC instrument.**

GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	170 °C (1 min), 8 °C/min to 250 °C (6 min), 20 °C /min to 280 °C (10 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as *bis*-TMS derivative:	
	Initial analysis:	Mean = 99.7%, s = 0.05% (10 sub samples in duplicate, April 2021)
	Re-analysis:	Mean = 99.6%, s = 0.03% (5 sub samples in duplicate, April 2022)
	Re-analysis:	Mean = 99.7%, s = 0.01% (5 sub samples in duplicate, January 2024)
Thermogravimetric analysis:		Non-volatile residue < 0.2% mass fraction (April 2021). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material and/or degradation at elevated temperatures.
Karl Fischer analysis:		Moisture content < 0.4% mass fraction (April 2021, February 2022 and January 2024)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	DB-5MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	170 °C (1 min), 8 °C/min to 250 °C (6 min), 20 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	20/1
	The retention time of the parent compound is reported along with the major peaks in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (16.5 min):	314 (M^+, 7), 246 (13), 231 (100), 193 (6), 174 (8) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Toluene/hexane/diethylamine (25/10/1) Single spot observed, R_f = 0.4. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3520, 3409, 2963, 2923, 2855, 2829, 1622, 1581, 1442, 1214, 1019, 883 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 0.88 (3H, t, *J* = 6.9 Hz), 1.22 - 1.38 (4H, m), 1.56 (2H, quintet, *J* = 7.7 Hz), 1.66 (3H, s), 1.74 - 1.87 (2H, m), 1.79 (3H, bs), 2.04-2.16 (1H, m), 2.17 - 2.31 (1H, m), 2.39 (1H, dd, *J* = 3.7, 10.6 Hz), 2.44 (2H, t, *J* = 7.7 Hz), 3.85 (1H, m), 4.56 (1H, s), 4.66 (1H, m), 4.71 (1H, bs), 5.57 (1H, s), 5.98 (1H, bs), 6.17 (1H, bs), 6.28 (1H, bs) ppm
^{1}H NMR:	Instrument:	Bruker Avance III
	Field strength:	500 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 0.90 (3H, t, *J* = 7.1 Hz), 1.24 – 1.39 (4H, m), 1.55 (2H, quintet, *J* = 7.5 Hz), 1.64 (3H, s), 1.68 (3H, bs), 1.70-1.79 (2H, m), 2.00 (1H, bd, *J* = 16.8 Hz), 2.20 (1H, m), 2.38 (2H, t, *J* = 7.6 Hz), 2.90 (1H, m), 3.93 (1H, m), 4.43 (1H, m), 4.47 (1H, m), 5.29 (1H, bs), 6.08 (2H, s) ppm
^{13}C NMR:	Instrument:	Bruker Avance III
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 14.2, 20.7, 22.7, 23.8, 28.5, 30.5, 30.8, 31.6, 35.6, 37.4, 46.3, 108.1, 109.9, 111.0, 113.9, 124.3, 140.2, 143.2, 149.5, 154.1, 156.2 ppm
Melting point:		67-69 °C
Microanalysis:	Found:	C = 80.2%; H = 9.6% (May, 2021)
	Calculated:	C = 80.2%; H = 9.6% (Calculated for $C_{21}H_{30}O_2$)