Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

NATA ACCREDITED FOR TECHNICAL COMPETENCE

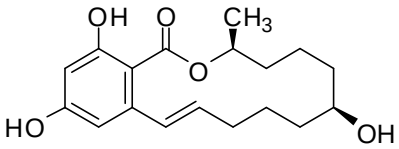
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1796: β-Zearalenol

Report ID: P1796.2019.01

Chemical Formula: $C_{18}H_{24}O_5$

Molecular Weight: 320.4 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
03-AV-05	**71030-11-0**	**99.3 ± 0.3%**

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

IUPAC name: (3R,7R,11E)-7,14,16-Trihydroxy-3-methyl-3,4,5,6,7,8,9,10-octahydro-1H-2-benzoxacyclotetradecin-1-one.

Expiration of certification: The property values are valid till 17 June 2024, i.e. five 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: White crystalline powder prepared by sourced from an external supplier, and certified for identity and purity by NMIA. Packaged in amber glass bottles with a septum and crimped aluminium cap or screw top 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 five years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials. 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 HPLC with UV detection on seven 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 a 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.
21 June 2019

This report supersedes any issued prior to 21 June 2019.

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 HPLC with UV detection, 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 elemental microanalysis.

HPLC:	Instrument	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Alltima C-18, 5 μm (4.6 mm × 150 mm)
	Column oven:	40 °C
	Mobile Phase:	MeOH:MilliQ H_2O (60:40)
	Flow Rate:	1.0 mL/min
	Detector:	Shimadzu PDA SPD-M20A operating at Max plot
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.01% (7 sub samples in duplicate, August 2003)
	Re-analysis:	Mean = 99.9%, s = 0.001% (5 sub samples in duplicate, August 2009)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, July 2014)
	Re-analysis:	Mean = 99.9%, s = 0.004% (5 sub samples in duplicate, June 2019)
Karl Fischer analysis:		Moisture content 0.4 % mass fraction (August 2009) Moisture content 0.7 % mass fraction (June 2014)
Thermogravimetric analysis:		Volatiles content and non-volatile residue < 0.3% mass fraction (August 2003 & October 2006)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quatro Micro
	Operation:	Positive ion mode, direct infusion at 5 □L/min
	Solvent:	MeOH/H_2O with 0.1% CH_3CO_2H
	Ionisation:	ESI spray voltage at 2.8 kV positive ion
	Desolvation temp:	200 °C
	EM voltage:	600 V
	Scan Range:	50-500 *m/z*
	Peak:	343.1 (MNa^+, 50), 321.2 (MH, 100) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate (1:1) Single spot observed, R_f = 0.21 Visualisation with UV light (254 nm).
IR:	Instrument:	BioRad FTS 3000MX FT-IR.
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3492, 3176, 2945, 2857, 1688, 1607, 1358, 1315, 1263, 1172, 1019, 967, 761 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX600
	Field strength:	600 MHz
	Solvent:	Acetone-d_6
	Spectral data:	δ 1.34 (3H, d), 3.42 (1H, bs, OH), 6.28 (1H, d), 6.53 (1H, d), 6.85 (1H, d), 9.15 (1H, bs, OH), 11.04 (1H, bs, OH) ppm
^{13}C NMR:	Instrument:	Bruker DMX300
	Field strength:	75 MHz
	Solvent:	Acetone-d_6
	Spectral data:	δ 19.2, 19.3, 22.7, 30.4, 32.2, 34.5, 36.7, 67.1, 70.8, 101.7, 103.5, 112.1, 128.5, 132.5, 137.5, 157.1, 159.6, 168.6 ppm
Melting point:		175 - 177 °C
Microanalysis:	Found:	C = 67.5%, H = 7.6% (May 2003)
	Calculated:	C = 67.5%, H = 7.6% (Calculated for $C_{18}H_{24}O_5$)