Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

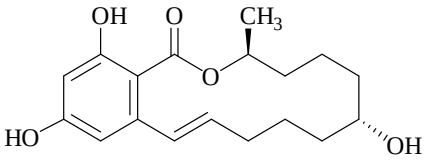
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1795: α-Zearalenol

Report ID: P1795.2023.01

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-04	36455-72-8	98.8 ± 0.9%

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

IUPAC name: (3*S*,7*R*,11*E*)-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 27 July 2033, ten 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 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 all impurities are quantified as a mass fraction and subtracted from 100%.

Stability: This material has demonstrated stability over a minimum period of ten 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 six 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.
28 July 2023

This report supersedes any issued prior to 28 July 2023.

NATA Accreditation No. 198 / Corporate Site No. 14214.

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.

$$\text{Purity} = (100\ \% - I_{ORG}) \times (100\ \% - I_{VOL} - I_{NVR}) \qquad \text{Equation 1}$$

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:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Alltech C-18, 5 μm (4.6 mm x 150 mm)
	Mobile Phase:	Methanol/MilliQ water (70:30 v/v)
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 PDA operating at 235 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.4%, s = 0.02% (6 sub samples in duplicate, August 2003)
	Re-analysis:	Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, August 2009)
	Re-analysis:	Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, July 2014)
	Re-analysis:	Mean = 99.6%, s = 0.01% (6 sub samples in duplicate, July 2019)
	Re-analysis:	Mean = 99.5%, s = 0.05% (5 sub samples in duplicate, July 2023)
Karl Fischer analysis:		Moisture content 0.2% mass fraction (August 2009) Moisture content 0.5% mass fraction (June 2014) Moisture content 0.7% mass fraction (May 2019) Moisture content 0.8% mass fraction (July 2023)
Thermogravimetric analysis:		Volatiles content < 0.1% and non-volatile residue < 0.2% mass fraction (April 2003)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP 5890/5971A
	Column:	BPX-5, 30 m × 0.22 mm I.D. × 0.25 μm
	Program:	175 °C, 5 °C/min to 250°C, 20 °C/min to 280 °C (10 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	320 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention time of the parent compound as the *tris*-TMS derivative is reported along with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (22.6 min):	536 (M^+, 1), 431 (1), 305 (10), 197 (13), 183 (10), 129 (5), 73 (100) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate (1:1) Single spot observed, R_f = 0.5.
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm^{-1}, KBr powder
	Peaks:	3310 (OH), 2941, 2857, 1642 (C=O), 1607 (C-O), 1581, 1354, 1316, 1259, 1172, 1022, 842 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX300
	Field strength:	300 MHz
	Solvent:	Acetone-d_6 (2.05 ppm)
	Spectral data:	δ 1.15 (1H, m), 1.41 (3H, d, *J* = 6.0 Hz), 1.40-1.80 (7H, m), 1.94 (2H, m), 2.32 (2H, m), 3.32 (1H, bs, OH), 3.78 (1H, m), 4.98 (1H, m), 5.75 (1H, m), 6.31 (1H, d, *J* = 3.0 Hz), 6.48 (1H, d, *J* = 3.0 Hz), 7.19 (1H, d, *J* = 15.5 Hz), 9.10 (1H, bs, OH), 12.18 (1H, bs, OH) ppm
^{13}C NMR:	Instrument:	Bruker DMX300
	Field strength:	75 MHz
	Solvent:	Acetone-d_6
	Spectral data:	δ 21.7, 22.8, 23.9, 31.6, 33.1, 36.1, 38.1, 66.6, 75.1, 103.2, 103.9, 110.1, 134.1, 134.8, 145.6, 163.8, 167.2, 173.1 ppm
Melting point:		165-168 °C
Microanalysis:	Found:	C = 67.4%; H = 7.5% (August 2003)
	Calculated:	C = 67.5%; H = 7.5% (Calculated for $C_{18}H_{24}O_5$)