

CERTIFICATE OF ANALYSIS

Certified Reference Material

ERM[®] -BC715

Zearalenone in maize germ oil		
Measurand ¹⁾	Certified value ²⁾	Uncertainty ³⁾
	Mass fraction in $\mu\text{g kg}^{-1}$	
Zearalenone ⁴⁾	362	22
<p>¹⁾ Zearalenone determined using sample preparation, instrumental separation (HPLC) and mass spectrometric detection as specified in section 3 of the certification report.</p> <p>²⁾ The value given represents the unweighted mean value of 20 ampoule mean values analysed by BAM. The certified value is traceable to the International System of Units (SI) via an unbroken chain of calibrations to the pure analyte.</p> <p>³⁾ Estimated expanded uncertainty U with a coverage factor of $k = 2$, corresponding to a level of confidence of approximately 95%, as defined in the Guide to the expression of uncertainty in measurement (GUM), ISO/IEC Guide 98-3 (2008). Uncertainty contributions arising from characterisation as well as from homogeneity and stability testing were taken into account.</p> <p>⁴⁾ CAS number: 17924-92-4</p>		

The certified value will be valid for 24 months beginning with the dispatch of the material from BAM.

The minimum sample intake for one determination is 0.5 mL (corresponding to 0.45 g).

Note

The European Reference Material ERM[®]-BC715 was produced and certified under the responsibility of Bundesanstalt für Materialforschung und -prüfung (BAM) according to the principles laid down in the technical guidelines of the European Reference Materials[®] co-operation agreement between BAM-LGC-JRC.

Instructions for Use

Before initial opening, the ampoule should reach room temperature and must be shaken thoroughly. The stability of the reference material is not affected by short periods of handling at ambient temperature during transport and use. However, BAM cannot be held responsible for any alteration of the material occurring during handling and storage at the customer's premises, especially of opened samples. Exposure to sunlight may cause the isomerization of natural zearalenone (*trans*-isomer) to its *cis*-isomer. Therefore, the material is filled in an amber glass ampoule and should be protected from sunlight.

Sample No.:

Date of dispatch:

Material Description

This certified reference material (CRM) is available as refined maize germ oil with a natural content of the mycotoxin zearalenone (ZEN). ERM®-BC715 is intended to be used for performance control and validation of analytical methods for the determination of ZEN in refined maize oils. It may also be applicable for other similar matrices (vegetable edible oils). The candidate material was selected from a survey of commercial maize germ oils conducted in German retail markets. After combining and thorough mixing (homogenisation) of the selected samples, a total number of 569 units (à 9 mL oil) were filled in 10 mL amber glass ampoules and sealed immediately. Prior to filling the ampoules were pre-flushed with argon to exclude oxygen. The material is stored at -20 °C until the dispatch from BAM. The between-bottle homogeneity was evaluated by analysis of variance (ANOVA) on 20 out of 569 ampoules (5 replicate analyses per ampoule). Extensive stability tests provided sound evidence for a minimum validity of the certified value as indicated on page 1 of this certificate, provided the material is stored according to the instructions given on page 3.

Interlaboratory comparison study (ILC)

In addition to the in-house certification at BAM, an interlaboratory comparison study was conducted at the same time to support and confirm the certification. The 13 participants are displayed in alphabetical order:

Laboratory	City, Country
Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit	Oberschleißheim (DE)
Bioanalytik Weihenstephan, Zentralinstitut f. Ernährungs- u. Lebensmittelforschung	München (DE)
Chemisches Labor Dr. Wirts + Partner Sachverständigen GmbH	Hannover (DE)
Chemisches und Veterinäruntersuchungsamt Rheinland	Leverkusen (DE)
Chemisches und Veterinäruntersuchungsamt Westfalen	Bochum (DE)
Coop	Pratteln (CH)
Food GmbH	Jena (DE)
Institut Kirchhoff Berlin GmbH	Berlin (DE)
Landeslabor Berlin-Brandenburg	Berlin (DE)
LUFA-ITL GmbH	Kiel (DE)
Multilab Hamburg Bergedorf GC, SGS Germany GmbH	Hamburg (DE)
Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit	Braunschweig (DE)
Österreichische Agentur für Gesundheit und Ernährungssicherheit	Linz (AT)

Analytical Methods and Results of the Certification Study

The in-house certification study at BAM was conducted by using high performance liquid chromatography hyphenated to negative electrospray tandem mass spectrometry (HPLC-ESI-MS/MS) based on stable isotope dilution analysis using a certified ZEN calibrant and [¹³C₁₈]-ZEN as internal standard. The ILC participants applied methods of their own choice. These methods involved instrumental determination by HPLC coupled to mass spectrometry or fluorescence detection. The methods varied according to initial extraction, clean-up and measurement. After technical/statistical outlier treatment, a ZEN mass fraction of 356.083 µg kg⁻¹ was calculated from ILC (mean value of 9 laboratory means). Further details are given in the certification report.

Safety Information

The usual laboratory safety precautions have to be applied. No hazardous effects are to be expected when the material is used under conditions usually adopted for the analysis of foodstuff matrices low or moderately contaminated with zearalenone. Although the mycotoxin content in the sample is at trace levels, any use other than the intended one should be avoided. The personnel handling the material must be trained adequately and follow the regular safety precautions of the laboratory. It is strongly recommended that the reference material is handled and disposed of in accordance with the guidelines for hazardous materials legally in force at the site of end use and disposal.

Transport and Storage

Due to the proven stability ERM[®]-BC715 can be shipped at ambient temperature. On receiving, the ampoule must be stored at a temperature equal to or lower than +4 °C.

Metrological Traceability

Traceability of the certified value is directly established using HPLC-MS/MS stable isotope dilution analysis applying a certified ZEN standard as calibrant and [¹³C₁₈]-isotopically labelled ZEN as internal standard. The certified mass fraction is traceable via the certified calibrant used. The certified value of the calibrant is traceable to the International System of Units (SI), as stated on the respective certificate, due to the gravimetric preparation employed. Therefore, the mass fraction of ZEN in ERM[®]-BC715 is traceable to the SI.

Technical Report

A detailed technical report describing the preparation, the analytical procedures and the treatment of the analytical data used to certify ERM[®]-BC715 is available on request or can be downloaded from the BAM website (www.bam.de).

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Accepted as an ERM[®], Berlin, 18 May 2020

Bundesanstalt für Materialforschung und -prüfung (BAM)



Dr. S. Richter
Committee for Certification

Dr. M. Koch
Project Coordinator

BAM holds an accreditation as a reference material producer according to ISO 17034. This accreditation is valid only for the scope as specified in the certificate D-RM-11075-01-00.

DAKkS is a signatory of the multilateral agreement (MLA) between EA, ILAC and IAF for mutual acceptance.



This reference material is supplied by:

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