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CERTIFICATE OF ANALYSIS

ERM®-BD476

Ochratoxin A (OTA) in red wine		
Compound	Certified value [1]	Uncertainty [2]
	Mass concentration in µg L^{-1}	
Ochratoxin A	0.52	± 0.11

[1] Unweighted mean of four independent results obtained by BAM using appropriate extraction, HPLC separation and MS/MS detection technique. The certified value is traceable to the SI.

[2] Estimated expanded uncertainty *U* with a coverage factor of *k* = 2, corresponding to a level of confidence of about 95 %, as defined in the Guide to the expression of uncertainty in measurement (GUM), ISO, 1995. Uncertainty contributions arising from the analytical method as well as from homogeneity and stability testing were taken into account.

The certified property value will be valid for 12 months beginning with the dispatch of the material from BAM; this validity may be extended as further evidence of stability becomes available.

The minimum sample intake is 10 mL.

NOTE

European Reference Material ERM®-BD476 was produced and certified under the responsibility of BAM – Federal Institute for Materials Research and Testing according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

Accepted as an ERM®, Berlin, 05 October 2010

Date of dispatch:

Signed

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DESCRIPTION OF THE SAMPLE

The intended purpose of reference material ERM®-BD476 is i) validation of analytical procedures for the determination of ochratoxin A in wine, and ii) quality assurance in the analytical laboratory.
The material ERM®-BD476 is a red wine sample from commercial sources intended for human consumption with a natural content of ochratoxin A (OTA) and ochratoxin B (OTB). After filtration and homogenisation a total of 205 units were bottled in amber glass flasks containing (51 ± 1) mL sealed with screw caps containing PTFE-inlays, and numbered in the order of leaving the bottling process. The material is stored at BAM at 4 °C until dispatch.
The between-bottle homogeneity was evaluated by analysis of variance (ANOVA) on 8 out of 205 bottles (4 replicate analyses per bottle). 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. Homogeneity and stability testing are described in detail in the certification report.

PARTICIPANTS

An interlaboratory comparison study (ILC) involving the following 15 laboratories was conducted in order to support the in-house certification study at BAM.

Laboratory	City / Country
Analytec Labor für Lebensmitteluntersuchung	Freilassing, Germany
Bundesinstitut für Risikobewertung	Berlin, Germany
Chemisches und Veterinäruntersuchungsamt Sigmaringen	Sigmaringen, Germany
Food GmbH Jena	Jena, Germany
General Chemical State Laboratory, Food Division & Division of Environment	Athens, Greece
Görtler Analytical Service GmbH	Vaterstetten, Germany
Institut für Lebensmittel, Arzneimittel und Tierseuchen (ILAT)	Berlin, Germany
Labor Dr. Scheller GmbH	Augsburg, Germany
Labor Eurofins Wiertz-Eggert-Jörissen	Hamburg, Germany
Landesuntersuchungsamt Rheinland-Pfalz	Trier, Germany
Lebensmittelversuchsanstalt	Vienna, Austria
Max Rubner-Institut	Detmold, Germany
Public Analyst´s Laboratory Dublin	Dublin, Ireland
SGS Germany GmbH	Hamburg, Germany
UIS Umweltinstitut synlab GmbH	Stuttgart, Germany

ANALYTICAL METHODS USED FOR CHARACTERISATION

For the in-house certification study at BAM an immunoaffinity clean-up step (IAC) after extraction was applied. High performance liquid chromatography (HPLC) technique was used for separation of the purified extract followed by MS/MS detection (MRM mode) with $^{13}C_{20}$ OTA as internal standard.
The ILC participant laboratories applied methods of their own choice. All laboratories used an IAC clean-up after extraction followed by HPLC analysis for separation of the purified extract. For detection and quantification either fluorescence or MS/MS detection combined with an external calibration were used.

SAFETY INFORMATION

Ochratoxin A is a (suspected) carcinogen and should be handled with due caution. Although the OTA content in the samples is at trace levels, the content of the bottles is not intended for nutritional use. Personnel handling the material must adequately be trained and follow regular laboratory safety precautions.

INSTRUCTIONS FOR USE

Before withdrawing a sub-sample the bottle should be allowed to reach room temperature and mixed thoroughly. Thereafter, the bottle is to be closed tightly. To the best of our knowledge, 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 alterations of the material occurring during transportation to, and handling and storage at, the customer's premises, especially of opened samples.

STORAGE

The material has to be stored at a temperature equal to or lower than 4 °C in its original bottle. Freezing has to be avoided.

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TECHNICAL REPORT

A detailed technical report (pdf file or paper copy; in English) describing the production, general characterisation as well as the analytical procedures applied and the treatment of the analytical data during certification of ERM®-BD476 is available on request from BAM.

Supply of Reference Materials by: BAM Federal Institute for Materials Research and Testing

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