

CERTIFICATE OF ANALYSIS

ERM[®]-BD272

Acrylamide in Crispbread		
Compound	Certified value ¹⁾	Uncertainty ²⁾
	Mass fraction in mg/kg	
Acrylamide	0.98	± 0.09

¹⁾ Unweighted mean of accepted mean values, independently obtained by 15 laboratories using different analytical methods. 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 characterisation as well as from homogeneity and stability testing were taken into account.

The certified properties 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 2 g.

NOTE

European Reference Material ERM[®]-BD272 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-IRMM. Information on these guidelines is available on the Internet (<http://www.erm-crm.org>).

Accepted as an ERM[®], Berlin, 2007-03-08

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Signed

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DESCRIPTION OF THE SAMPLE

The intended purpose of reference material ERM[®]-BD272 is i) validation of analytical procedures for the determination of acrylamide in food, and ii) quality assurance in the analytical laboratory.

The material ERM[®]-BD272 is a crispbread sample from commercial sources intended for human consumption. After fractionation by sieving and homogenisation, the sieve fraction < 500 µm was subdivided into 500 units of approximately 68 g. Units were filled in brown glass bottles with screw caps containing PTFE-inlays, bottles are sealed with shrinking foil. The material is stored at BAM at –20°C until dispatch.

The between-bottle homogeneity was evaluated by analysis of variance (ANOVA) on 10 out of 500 bottles (4 replicate analyses per bottle). Extensive stability tests provided sound evidence for a minimum validity of the material 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

The certification study involved the following 15 laboratories using various analytical methods for the determination of acrylamide in food:

Laboratory	City/State
BAM, I.2	Berlin; Germany
Chemisches und Veterinäruntersuchungsamt Sigmaringen	Sigmaringen; Germany
Chemisches und Veterinäruntersuchungsamt Stuttgart	Fellbach; Germany
General Chemical State Laboratory, Food Division & Division of Environment	Athens; Greece
German Research Centre of Food Chemistry Lab (1)	Garching; Germany
German Research Centre of Food Chemistry Lab (2)	Garching; Germany
IRMM	Geel; Belgium
Kantonales Labor Zürich	Zürich; Switzerland
Lebensmittelchemisches Institut Köln	Köln; Germany
Lebensmittelversuchsanstalt	Vienna; Austria
National Food Administration Sweden	Uppsala; Sweden
Nestlé Research Centre	Lausanne; Switzerland
Public Analyst's Laboratory Dublin	Dublin; Ireland
VWA/KvW Keuringsdienst van Waren Zuid Limburg, Noord Brabant	Eindhoven; The Netherlands
W.E.J. GmbH	Hamburg; Germany

ANALYTICAL METHODS USED FOR CERTIFICATION

The following analytical methods were employed by the participating laboratories:

Derivatisation	Analytical method
Bromination	GC-MS
Without	GC-MS
using 2-mercapto-benzoic acid	HPLC-ESI MS/MS
Without	HPLC-ESI-MS/MS

The acrylamide mass fraction is, although not method-specific, clearly a parameter which is influenced by the method employed for its determination, namely the extraction and (if applicable) derivatisation procedure. The measurement step takes traceability from calibration using the pure substance (AA, 99+%; Merck Darmstadt), sample preparation steps from spiking using D₃-AA (99+%; Polymer Source), both having

independently confirmed purities. Overall recovery was estimated to 95 - 105 %. Remaining systematic between-method biases are sufficiently covered by the allowance made for the intercomparison contribution to the total uncertainty budget. The certified value is traceable to the SI.

SAFETY INFORMATION

Acrylamide is a (suspected) carcinogen and should be handled with due caution. Although acrylamide content in the samples is at trace levels, any use other than the intended of the content of the bottles is strictly prohibited. Personnel handling the material must adequately be trained and follow normal safety precautions.

INSTRUCTIONS FOR USE

Before withdrawing a sub-sample the bottle should be allowed to reach room temperature and is to be 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 $-20\text{ }^{\circ}\text{C}$ in its original bottle.

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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[®]-BD272 is available on request from BAM.

Supply of Reference Materials by Bundesanstalt für Materialforschung und –prüfung:

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