CERTIFICATE OF ANALYSIS

ERM®-BD514

Cd in Cocoa		
	Certified value [1]	Uncertainty [2]
Element	Mass fraction in mg/kg	
Cd	0.541	0.024

[1] Unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory and/or with a different method of determination. The certified value is traceable to the SI (Système International d'Unités) by the use of pure substances of known stoichiometry for calibration.

[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 ISO/IEC Guide 98-3:2008 [Uncertainty of measurement -- Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)].

	Indicative Value [1]	Uncertainty [2]
Compound	Mass fraction in mg/kg	
Acrylamide	0.101	0.026

[1] Unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory and/or with a different method of determination. The informative value is traceable to the SI (Système International d'Unités) by the use of pure substances of known stoichiometry for calibration.

[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 ISO/IEC Guide 98-3:2008 [Uncertainty of measurement -- Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)].

The property values 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.

NOTE

European Reference Material ERM®-BD513 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.

Accepted as an ERM®, Berlin, 2019

Dr. S. Richter
Committee for Certification

Dr. S. Recknagel
Project Coordinator

Date of dispatch:

ADDITIONAL MATERIAL INFORMATION

Determination of moisture using Karl-Fischer-titration performed at BAM gave the following non-certified results (mean of 45 single measurements ± standard deviation):

3.70 % ± 0.19 %

The water content was seen to remain stable if the material is handled according to the instructions for use. The property values are valid for the material taken directly from the bag without drying prior to the analysis.

DESCRIPTION OF THE SAMPLE

The material ERM®-BD514 is a powdered cocoa sample from commercial sources intended for human consumption. After mixing in a drum hoop mixer for 24 h the cocoa powder was filled into sealed aluminised plastic bags using a sample divider. Each plastic bag contains ca. 8 g of certified reference material.

INTENDED USE

The intended purpose of reference material ERM®-BD514 is i) validation of analytical procedures for the determination of Cd and acrylamide in food, and ii) quality assurance in the analytical laboratory. The minimum sample size for chemical analysis is 0.5 g for Cd and 0.05 g for acrylamide.

INSTRUCTIONS FOR USE

Before taking a sub-sample the material should be allowed to reach room temperature and is to be mixed thoroughly. Thereafter, the bag is to be closed tightly and stored at a temperature equal to or lower than -20 °C. 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 °C in its original plastic bag.

PARTICIPANTS

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National Institute of Public Health - National Institute of Hygiene, Warsaw, Poland
National Laboratory for Health, Environment and Food, Maribor, Slovenia
Sanitary Veterinary and Food Safety Directorate, Bucharest, Rumania

MEANS OF ACCEPTED DATA SETS

Means of accepted data sets, mass fraction in mg/kg

Line no.	Cd
1	0.456
2	0.465
3	0.471
4	0.483
5	0.500
6	0.507
7	0.546
8	0.554
9	0.555
10	0.555
11	0.559
12	0.567
13	0.569
14	0.572
15	0.573
16	0.576
17	0.577
18	0.597
19	0.602
M	0.541
s_M	0.046
$\sqrt{\overline{s_l^2}}$	0.006

Means of accepted data sets, mass fraction in mg/kg

Line no.	Acrylamide
1	0.075
2	0.079
3	0.080
4	0.080
5	0.089
6	0.093
7	0.100
8	0.116
9	0.192
10	---
M	0.101
s_M	0.037
$\sqrt{\overline{s_l^2}}$	0.017

The laboratory results have been examined statistically. Where a " --- " appears in the table it indicates that outlying results have been omitted.

M : mean of laboratory means s_M : standard deviation of laboratory means

$\sqrt{\overline{s_l^2}}$: averaged repeatability standard deviation (square root of the mean of laboratory variances)

ANALYTICAL METHOD USED FOR CERTIFICATION

Element	Line no.	Method
Cd	1, 6, 9, 12, 14, 18, 19	ICP-MS, dissolution in HNO_3/H_2O_2, microwave
	2, 4, 11	ETAAS, dissolution in HNO_3/H_2O_2, microwave
	3, 16	ICP-MS, dissolution in HCl/HNO_3, microwave
	5	ETAAS, dissolution in $HNO_3/HCl/H_2O_2$, microwave
	7, 10, 13	ICP-MS, dissolution in HNO_3
	8	HR-ICP-MS, dissolution in HNO_3/H_2O_2, microwave
	15	ID-ICP-MS, dissolution in HNO_3/H_2O_2, HPA
	17	ETAAS, dissolution in HNO_3, microwave

Abbreviations:

ETAAS: Electrothermal atomic absorption spectrometry
ICP-MS: Inductively coupled plasma mass spectrometry
HPA: High pressure asher
HR-ICP-MS: High-resolution inductively coupled plasma mass spectrometry

Compound	Dataset no.	Method
Acrylamide	1	GC-MS acc. to DIN 10785, D_3-Acrylamide as ISTD
	2, 3, 4, 8	LC-MS/MS acc. to EN 16618, D_3-Acrylamide as ISTD
	5, 9	LC-MS/MS, D_5-Acrylamide as ISTD
	6	LC-MS/MS, D_3-Acrylamide as ISTD
	7	LC-MS/MS, C_{13}-Acrylamide as ISTD

Abbreviations:

GC: Gas chromatography
ISTD: Internal standard
LC: Liquid chromatography
MS: Mass spectrometry

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TECHNICAL REPORT

A detailed technical report describing the production, general characterisation as well as the analysis procedures applied, and the treatment of the analytical data used to certify CRMs ERM®-BD513, ERM®-BD514 and ERM®-BD515 is available on request or can be downloaded from BAM website (www.bam.de).

Supply of this Reference Material by:

Bundesanstalt für Materialforschung und -prüfung (BAM)

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