Certified Reference Material

BAM-U115

Aqua Regia Extractable Trace Elements in Soil

Certified Values

Element	Extraction according to **EN 16174, Method A** *) (open vessel, reflux conditions)		Extraction according to **EN 16174, Method B** (microwave-assisted, 175 °C)	
	Mass fraction[1] in mg/kg	**Uncertainty** U[2] in mg/kg	**Mass fraction**[1] in mg/kg	**Uncertainty** U[2] in mg/kg
As	27.7	0.9	27.9	1.1
Cd	4.52	0.17	4.65	0.16
Co	7.35	0.28	7.3	0.4
Cr	96.9	2.7	99.6	2.9
Cu	167	5	171	6
Hg	4.00	0.17	4.07	0.14
Ni	29.3	1.1	29.9	1.0
Pb	164	6	169	5
V	22.4	1.1	23.5	1.1
Zn	342	9	349	12

[1] Unweighted mean value of the means of accepted datasets obtained by at least 11 different laboratories and/or different methods of measurement. The certified values are corrected to the dry mass content of the material determined according to ISO 11465. They are operationally defined by the analytical protocols given in EN 16174.

[2] 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).

*) Extraction procedure according to EN 16174, Method A, is identical to the analytical protocol given in ISO 11466.

The minimum amount of sample to be used for the determination of aqua regia extractable mass fractions of elements is 3 g when applying extraction according to Method A, and 0.5 g when applying extraction according to Method B (as prescribed by European Standard EN 16174).

This certificate is valid for a period of 36 months beginning with the dispatch of the reference material from BAM.

Date of dispatch:

Material Description

The certified reference material (CRM) BAM-U115 represents a mixture of sandy and loamy soils collected at different contaminated sites in the Berlin region (Germany).

The raw material was dried at ambient air to constant mass and then the fraction passing a 2 mm screen was ground in a ball mill (with grinding bowls and balls made of zirconia) completely to particle sizes below 63 µm. Homogenization and bottling of the ground material were performed using a spinning riffler.

Determination of main matrix constituents of the bottled reference material performed at BAM by semi-quantitative X-ray fluorescence (XRF) analysis gave the following non-certified results:

Element	Si	Al	Ca	Fe	K
Mass fraction in %	34.7	3.2	3.5	2.2	1.4

Mass fraction of any other element detectable with XRF was determined to be less than 0.2 %.

Further informative analytical results characterizing the sample matrix:

Parameter	Mass fraction in %	Analytical procedure
Dry matter content at 105 °C	98.6	ISO 11465
Loss on ignition at 550 °C	8.3	EN 15935
Organic carbon (TOC)	2.5	ISO 10694
Inorganic carbon (TIC)	1.1	ISO 10694

CRM BAM-U115 is supplied in 100 mL brown glass bottles containing (53 ± 1) g.

Recommended Use

The intended purpose of CRM BAM-U115 is the verification of analytical results obtained for aqua regia extractable mass fractions of trace elements in soils applying the standardized procedures prescribed by EN 16174 (Method A and Method B, respectively). In this context it is noted that the extraction procedure according to EN 16174, Method A, is identical to the analytical protocol given in ISO 11466.
As any reference material, CRM BAM-U115 can also be used for routine performance checks (quality control charts) or validation studies.
Please note that it cannot be expected that the two extraction methods specified in EN 16174 provide compatible measurement results for all types of soil samples. Depending on the origin of the material to be analyzed, extraction with aqua regia using the microwave-assisted closed vessel procedure might be prone to result in higher mass fractions for certain elements, in particular for Cr, Pb and V.

Handling

The material should be used as it is from the bottle. However, before taking a sub-sample a re-homogenisation by manual shaking of the closed bottle is strongly recommended.

It should be kept in mind that aqua regia extractable mass fractions of elements in soil are operationally defined. When performing analyses, the analytical protocols given in EN 16174 must strictly be followed.
All analytical results have to be corrected for dry mass content of the material which has to be determined according to ISO 11465 using a separate sub-sample. The value given in the table above (98.6 %) should be regarded as being informative only.

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 commonly adopted for the analysis of environmental samples.

Transport and Storage

CRM BAM-U115 can be shipped at ambient temperature. Upon receipt the material should be stored at a temperature below 25 °C in its original tightly closed bottle. When handling the sample, the bottle shall be left unclosed as briefly as possible. Care should be taken to avoid moisture pick-up once the bottle is opened.
BAM cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

Participating Laboratories (in alphabetic order)

To certify the candidate material BAM-U115, an inter-laboratory comparison study was carried out involving the following laboratories:

AGROLAB Agrar und Umwelt GmbH, Sarstedt (Germany)

ALcontrol Laboratories BV, Rotterdam (The Netherlands)

ALS Analytik Labor Schirmacher GmbH, Hamburg (Germany)

ALS Scandinavia AB, Luleå (Sweden)

AZBA – Analytisches Zentrum Berlin-Adlershof GmbH, Berlin (Germany)

Bundesanstalt für Materialforschung und –prüfung (BAM), Berlin (Germany)

Central Institute for Supervising and Testing in Agriculture (UKZUZ), Brno (Czech Republic)

Central Mining Institute, Laboratory of Waste Analyses, Katowice (Poland)

Eurofins Umwelt Ost GmbH, Niederlassung Freiberg, Bobritzsch-Hilbersdorf (Germany)

Eurofins Umwelt West GmbH, Wesseling (Germany)

Fraunhofer Institut für Molekularbiologie und Angewandte Oekologie (IME),
Umweltprobenbank und Elementanalytik, Schmallenberg (Germany)

ICA – Institut für Chemische Analytik GmbH, Leipzig (Germany)

UCL Umwelt Control Labor GmbH, Kiel (Germany)

UCL Umwelt Control Labor GmbH, Lünen (Germany)

SGS INSTITUT FRESENIUS GmbH, Herten (Germany)

SPECTRO Analytical Instruments GmbH, Kleve (Germany)

WESSLING GmbH, Labor Hannover, Hannover (Germany)

The statistical evaluation of measurement results obtained in the course of the certification project as well as all calculations to establish the certified values of CRM BAM-U115 were performed on the basis of ISO Guide 35.

Metrological Traceability

The certified aqua regia extractable mass fractions of trace elements in CRM BAM-U115 are operationally defined referring to the analytical protocols prescribed by EN 16174, Method A and Method B.

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References

Certification Report „CRM BAM-U115“ (August 2016)
(Download: https://www.bam.de via link *Specialized Portals*)

Guidelines for the Development and Production of BAM Reference Materials (February 2016)
(available on request)

EN 16174 (2012):	Sludge, treated biowaste and soil – Digestion of aqua regia soluble fractions of elements
EN 15935 (2012):	Sludge, treated biowaste, soil and waste – Determination of loss on ignition
ISO 10694 (1995):	Soil quality – Determination of organic and total carbon after dry combustion (elementary analysis)
ISO 11465 (1993):	Soil quality – Determination of dry matter and water content on a mass basis. Gravimetric method
ISO 11466 (1995):	Soil quality – Extraction of trace elements soluble in aqua regia
ISO Guide 35 (2006):	Reference materials – General and statistical principles for certification
ISO/IEC Guide 98-3 (2008):	Uncertainty of measurement – Part 3: Guide to the Expression of Uncertainty in Measurement (GUM)

Accepted as a BAM-CRM on September 07, 2016

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BAM holds an accreditation as a reference material producer according to ISO Guide 34 in combination with ISO/IEC 17025. 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.

DAkkS Deutsche Akkreditierungsstelle D-RM-11075-01-00