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Certified Reference Material

BAM-U112a

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	10.3	0.5	10.4	0.7
Cd	4.12	0.15	4.09	0.17
Co	3.58	0.22	3.9	0.4
Cr	80.1	2.5	81.9	2.6
Cu	75.5	3.1	75	4
Hg	16.3	1.0	15.9	1.1
Ni	10.1	0.5	11.2	0.9
Pb	198	8	199	8
V	12.7	0.8	14.0	0.9
Zn	198	6	200	7

¹⁾ Unweighted mean value of the means of accepted data sets obtained by at least 14 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 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:

CERTIFICATE



Material Description

The certified reference material (CRM) BAM-U112a was prepared as a successor of the sold-out CRM BAM-U112 with an almost identical matrix composition. The starting material for both CRMs was a sandy soil collected from the same contaminated site near Berlin (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 125 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 analysis gave the following non-certified results:

Element	Si	Al	Ca	Fe	K
Mass fraction in %	38.2	2.5	2.1	1.0	1.2

Further informative analytical results characterizing the sample matrix:

Parameter	Mass fraction in %	Analytical method
Dry matter content at 105 °C	99.5	ISO 11465
Loss on ignition at 550 °C	2.4	EN 12879
Organic carbon (TOC)	1.4	ISO 10694
Inorganic carbon (TIC)	0.3	ISO 10694

CRM BAM-U112a is supplied in 100 mL brown glass bottles containing (55 ± 1) g.

Recommended Use

The intended purpose of CRM BAM-U112a is the verification of analytical results obtained for aqua regia extractable mass fractions of trace elements in soils applying the standardised 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-U112a 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 comparable measurement results for all types of soil samples. Depending on the origin of the material to be analysed, 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 As, Co, Cr, Ni, 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 (99.5 %) should be regarded as being indicative 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-U112a 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

To certify the candidate material BAM-U112a, an inter-laboratory comparison study was carried out involving the following laboratories:

Agrolab Labor GmbH, Bruckberg (Germany)

ALS Analytik Labor Schirmacher GmbH, Hamburg (Germany)

Analytik Institut Rietzler GmbH, Nürnberg (Germany)

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

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

Eurofins Analytico B.V., Barneveld (The Netherlands)

Eurofins Umwelt Ost GmbH, Niederlassung Freiberg, Halsbrücke (Germany)

EUROLAB s.r.l., Nichelino/Torino (Italy)

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

IHU – Geologie und Analytik, Gesellschaft für Ingenieur-, Hydro- und Umweltgeologie mbH, Stendal (Germany)

INRA – Laboratoire d'Analyses des Sols, Arras (France)

Institut Koldingen GmbH, Sarstedt (Germany)

Landesamt für Natur, Umwelt und Verbraucherschutz NRW, Düsseldorf (Germany)

Ramboll Finland Oy, Ramboll Analytics, Lahti (Finland)

SGS Institut Fresenius GmbH, Herten (Germany)

SPECTRO Analytical Instruments GmbH, Kleve (Germany)

VKTA – Verein für Kernverfahrenstechnik und Analytik e.V., Dresden (Germany)

Wageningen Universiteit, Chemisch Biologisch Laboratorium Bodem, Wageningen (The Netherlands)

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-U112a were performed on the basis of ISO Guide 35.

Metrological Traceability

The certified aqua regia extractable mass fractions of trace elements in CRM BAM-U112a 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-U112a“ (H. Scharf, BAM, May 2014)

(Download: BAM-homepage (www.bam.de) via links <Reference Materials> and <Certificates and reports>)

Guidelines for the production of BAM reference materials (BAM, updated version April 2010)

(http://www.bam.de/en/fachthemen/referenzmaterialien/referenzmaterialien_medien/bam_rm_guidelines.pdf)

- EN 16174 (2012): Sludge, treated biowaste and soil – Digestion of aqua regia soluble fractions of elements
- EN 12879 (2000): Characterization of sludges – Determination of the loss on ignition of dry mass
- 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

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