



CERTIFICATE

CERTIFIED REFERENCE MATERIAL BAM-U110a

Contaminated Soil

Certified Value(s)

Total mass fractions:

Element	Mass fraction in mg/kg*	Uncertainty in mg/kg
As	15.8	1.7
Cd	7.3	0.8
Co	16.2	1.8
Cr	230	17
Cu	263	17
Hg	51	6
Mn	621	30
Ni	101	7
Pb	197	18
Zn	1000	55

*corrected for dry matter content of the soil sample at 105 °C (determination according to ISO 11465)

Aqua regia extractable mass fractions:

Element	Mass fraction in mg/kg*	Uncertainty in mg/kg
As	13.0	1.4
Cd	7.0	0.7
Co	14.5	1.2
Cr	190	18
Cu	262	18
Hg	49	5
Mn	580	34
Ni	96	8
Pb	185	15
Zn	990	55

*corrected for dry matter content of the soil sample at 105 °C (determination according to ISO 11465)

Calculation of uncertainty

U is the estimated expanded uncertainty with a coverage factor of $k = 2$, corresponding to a level of confidence of approx. 95 %, as defined in the Guide to the Expression of Uncertainty in Measurement, (GUM, ISO/IEC Guide 98-3:2008). The combined standard uncertainty was calculated as the positive square root of the combined variances and adding a bias term to capture the deviation from BAM-U110:

$$U = U_{new} = 2 \cdot u_{shift} + |bias| \quad \text{with} \quad u_{shift} = \sqrt{u_{cert}^2 + u_{bias}^2}$$

u_{cert} uncertainty contribution calculated from the results of the certification interlaboratory comparison and the homogeneity test

$bias$ absolute measured difference between BAM-U110 and BAM-U110a

u_{bias} uncertainty contribution for estimating the difference between BAM-U110 and BAM-U110a

End of Validity

This certificate is valid until there is a revocation from the producer of the material.

Material Description

BAM-U110a is based on the same batch of candidate material as BAM-U110. The mass fractions of the certified elements of BAM-U110a were checked against BAM-U110 and were found slightly different. Therefore, the uncertainties of the certified values were expanded by the bias of the measurements. All mass fractions were obtained from the certification inter-laboratory comparison of BAM-U110 from 2006.

Source and Preparation of Material

The starting material for preparing CRM BAM-U110 and BAM-U110a was an alluvial soil from an area periodically exposed to floods of river Saale. The site is located near the city of Halle (Saxony-Anhalt, Germany) and contaminated by pollutants of industrial and municipal origin.

The soil was collected from a depth between 0.2 and 0.8 m; according to sieve analysis the material was classified as silty clay loam.

The raw material was dried in a convection oven at 30 °C to constant mass and then passed over a vibrating 2 mm screen discarding the fraction > 2 mm. The material passing the screen was ground in a ball mill (with grinding bowls and balls made of agate) completely to particle sizes below 63 µm. Homogenisation and bottling of the ground material was performed using a tilted drum mixer equipped with a gravimetrically controlled filling device.

Matrix Composition

Determination of main matrix constituents of the bottled reference material performed at BAM by X-ray fluorescence analysis gave the following results:

Element	Si	Al	Ca	Fe	K	S	Mg
Mass Fraction (%)	25.7	5.1	4.1	2.8	1.9	1.1	1.0

The given values for these non-certified total mass fractions are in good agreement with results submitted by some of the participants in the interlaboratory comparison (see Annex).

Further analytical results characterising the sample matrix:

Parameter	Mass Fraction	Analytical Method
Dry Matter Content at 105 °C	97.3 %	ISO 11465
Loss on Ignition at 550 °C	13.3 %	DIN 38414-3
Organic Carbon (TOC)	6.7 %	ISO 10694
Inorganic Carbon (TIC)	0.8 %	ISO 10694
Carbon (total mass fraction)	7.5 %	Automatic CHN-Analyzer
Hydrogen	1.2 %	Automatic CHN-Analyzer
Nitrogen	0.4 %	Automatic CHN-Analyzer
pH in water	7.4	DIN ISO 10390
pH in CaCl ₂ solution	7.2	DIN ISO 10390

pH values in water and CaCl₂ solution (according to ISO 10390): 7.4 and 7.2, respectively.

Certification

The certified mass fractions of BAM-U110a are based on the results obtained for CRM BAM-U110 which was certified by means of an interlaboratory comparison organised by BAM. The total number of participating laboratories was 49, all of them with proven competence in soil analysis. All calculations were performed in accordance with ISO Guide 35. Individual data sets were rejected only in the case that they were identified as outlying laboratory means. Accepted laboratory means and used analytical methods are given in the annex to this certificate. Besides the results for certified parameters this annex also contains results for mass fractions of additional elements submitted by some of the participating laboratories. These results are given as indicative values if there were three or more data sets available, and as informative values in the case of less than three data sets. It is stressed that these values are not certified and that they are given for information purposes only.

Metrological Traceability

As the element contents of soils that can be extracted with aqua regia are process-defined parameters, the extractions were carried out in strict compliance with the convention procedure described in DIN ISO 11466.

Without exception, metals of known purity or commercially available standard solutions with certified element concentrations were used to prepare the calibration solutions.

Participating Laboratories

ANTEUM GmbH, Berlin
AUA Agrar- und Umweltanalytik GmbH, Jena
AZBA Analytisches Zentrum Berlin-Adlershof GmbH, Berlin
Berliner Wasserbetriebe, Geschäftsbereich Labor, Berlin
Bodenuntersuchungs-Institut Koldingen GmbH, Burgwedel
Bundesanstalt für Geowissenschaften und Rohstoffe (BGR), Referat B 4.25, Hannover
Bundesanstalt für Materialforschung und -prüfung (BAM), Fachgruppe I.1 (AG I.12), Berlin
Bundesanstalt für Materialforschung und -prüfung (BAM), Fachgruppe I.1 (AG I.13), Berlin
Bundesanstalt für Materialforschung und -prüfung (BAM), Fachgruppe I.1 (AG I.14), Berlin
Bundesanstalt für Materialforschung und -prüfung (BAM), Fachgruppe I.4 (AG I.43), Berlin
Chemisches Labor Dr. Vogt, Abfall-Wasser-Abwasseranalysen GmbH, Karlsruhe
Chemisches Laboratorium Dr. E. Weßling GmbH, Hannover
Chemisches Untersuchungslabor Dr. Lörcher, Ludwigsburg
Deutsche Steinkohle AG, Zentrallabor Saar, Saarbrücken

Dr. Fechter GmbH, Umweltlabor und Ingenieurbüro, Berlin
EMPA, Abt. Anorganische Chemie/Feststoffcharakterisierung, Dübendorf (Switzerland)
Forschungsinstitut für Bergbaufolgelandschaften e. V., Analytisches Labor, Finsterwalde
Fraunhofer-Institut für Molekularbiologie und Angewandte Oekologie IME, Schmallebenberg
G.E.O.S. Freiberg Ingenieurgesellschaft mbH, Umweltlabor, Tuttendorf
Geologischer Dienst Nordrhein-Westfalen, Krefeld
GKSS-Forschungszentrum Geesthacht GmbH, Institut für Küstenforschung, Geesthacht
görtler analytical services gmbh, Flöha
HDLGN - LUFA, Hessisches Dienstleistungszentrum für Landwirtschaft, Gartenbau u.
Naturschutz, Kassel
I.U.T. Institut für Umwelttechnologien GmbH, Berlin
ICA Institut für Chemische Analytik GmbH, Leipzig
IfE-Analytik GmbH, Leipzig
IHU - Geologie und Analytik, Gesellschaft für Ingenieur-, Hydro- u. Umweltgeologie mbH,
Stendal
Industrie- und Umweltlaboratorium Vorpommern GmbH, Greifswald
INNOLAB GmbH, Harburg
INSTITUT FRESENIUS Chemische und Biologische Laboratorien AG, Taunusstein
Institut Fresenius WPW GmbH, Saarbrücken
Institut für Spektrochemie und Angewandte Spektroskopie (ISAS), Dortmund
IUS Institut für Umweltanalytik und Schadstoffchemie GmbH, Stuttgart
Justus-Liebig-Universität Gießen, Institut für Landschaftsökologie und
Ressourcenmanagement, Gießen
Laborgesellschaft für Umweltschutz mbH, Neustadt
Landesamt für Geowissenschaften und Rohstoffe Brandenburg, Kleinmachnow
Landeslabor Brandenburg, Fachbereich U3, Potsdam
LUA Labor für Umweltanalytik GmbH, Schwerin
METALEUROP WESER GmbH, Labor für Arbeits- und Umweltschutz, Nordenham
PWU Potsdamer Wasser- und Umweltlabor GmbH & Co. KG, Potsdam
Ruhranalytik, Laboratorium für Kohle und Umwelt GmbH, Herne
SEWA Gesellschaft für Sediment- und Wasseranalytik mbH & Co. KG, Essen
TU Bergakademie Freiberg, Institut für Mineralogie, Freiberg
UCL - Umwelt Control Labor GmbH, Lünen
UFZ Umweltforschungszentrum Leipzig-Halle GmbH, Sektion Analytik, Leipzig
Umweltbundesamt (UBA), Labor für Bodenanalytik, Berlin
Verein für Kernverfahrenstechnik und Analytik Rossendorf e. V., Fachbereich KAA, Dresden
wave GmbH, Umweltlabor, Stuttgart
Wehrwissenschaftliches Institut für Schutztechnologien - ABC-Schutz, Dezernat 510, Munster

Recommendations for Correct Use and Storage

The CRM BAM-U110a is available as a powder with particle sizes below 63 µm and is supplied in 100 mL brown glass bottles containing (60 ± 1) g.

The material is intended for the verification of analytical results obtained by standardised procedures as well as for the validation of modified or new analytical procedures. Furthermore, it can be used for quality control or calibration purposes if X-ray fluorescence spectrometry or other methods of direct solid-state analysis are applied.

The certified values are valid for a minimum sample intake per analysis of at least 500 mg.

The material is stable provided the sample will be stored at a temperature below 30 °C in a dust-free and dry environment avoiding any kind of contamination.

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

When extracting the sample with aqua regia the analytical protocol prescribed by ISO 11466 or DIN EN 16174 must strictly be followed. All analytical results have to be corrected for dry matter content of the material which should be determined according to ISO 11465 using a separate sub-sample.

Technical Report

A detailed technical report describing the analysis procedures and the treatment of the analytical data used to certify BAM-U110a is available on request or can be downloaded from BAM website (www.bam.de).

Accepted as a BAM-CRM on March 14, 2024

Bundesanstalt für Materialforschung und -prüfung (BAM)

Dr. S. Richter
Committee for Certification

Dr. S. Recknagel
Project Coordinator

BAM holds an accreditation as a reference material producer according to ISO 17034. 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.



This Certified Reference Material is offered by:

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