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CERTIFICATE OF ANALYSIS

ERM®-CC018

Trace elements in contaminated sandy soil		
Analyte	Aqua regia extractable mass fraction in mg/kg [1] (extraction according to ISO 11466)	
	Certified value [2]	Uncertainty [3]
Arsenic	22.9	± 1.3
Cadmium	5.4	± 0.5
Chromium	129	± 6
Cobalt	5.9	± 0.4
Copper	80	± 4
Lead	289	±10
Mercury	1.38	± 0.06
Nickel	25.8	± 1.8
Vanadium	19.4	± 1.0
Zinc	313	±13

[1] All results are corrected to the dry mass content of the material determined after drying to constant mass at (105 ± 2) °C.

[2] Unweighted mean value of the means of accepted sets of data obtained in different BAM working groups using different methods of determination. The certified values are traceable to the SI (Système International d'Unites) via calibration using substances with certified purity.

[3] Estimated expanded uncertainty *U* with a coverage factor $k = 2.5$, 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). The approach for calculating the coverage factor is described in the Certification Report.

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

Date of dispatch:

The minimum amount of sample to be used for the determination of aqua regia extractable mass fractions of elements is 3 g (as prescribed by ISO 11466).

NOTE

European Reference Material ERM®-CC018 was produced and certified under the responsibility of BAM Bundesanstalt für Materialforschung und -prüfung according to the principles laid down in ISO Guide 35:2006 and in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-IRMM-LGC. Information on these guidelines is available via the internet (http://www.erm-crm.org).

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ADDITIONAL MATERIAL INFORMATION

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 %	38.8	2.1	2.6	1.4	0.9

Further informative analytical results characterising the sample matrix:

Parameter	Mass fraction in %	Analytical method
Dry mass content at 105 °C	99.1	ISO 11465
Loss on ignition at 550 °C	4.6	DIN 38414-3
Total organic carbon (TOC)	2.4	ISO 10694
Total inorganic carbon (TIC)	0.4	ISO 10694

pH values in water and $CaCl_2$ solution (according to ISO 10390): 7.6 and 7.5, respectively.

DESCRIPTION OF THE SAMPLE

The material is provided as a powder with particle sizes below 63 µm in a 100 mL screw-capped brown glass bottle containing (55 ± 1) g.

The starting material was a sandy soil collected from a stockpile excavated during a remediation campaign on an industrial wasteland in the Berlin region (Germany).
The raw material was dried at ambient air to constant mass and afterwards 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. Homogenisation and bottling of the ground material was performed using a spinning riffler.

ANALYTICAL METHODS USED FOR CERTIFICATION

The ERM®-CC018 was certified on the basis of analytical results obtained in three BAM working groups with 12 independent operator/equipment combinations in total. The following analytical methods were used:

- Cold-vapour atomic absorption spectrometry
- Cold-vapour atomic fluorescence spectrometry
- Electrothermal atomic absorption spectrometry
- Flame atomic absorption spectrometry
- Hydride generation atomic absorption spectrometry
- Inductively coupled plasma optical emission spectrometry

- Inductively coupled plasma mass spectrometry
- Solid sampling advanced mercury analyzer

As aqua regia extractable mass fractions of elements are operationally-defined parameters, extraction of the soil sample was performed strictly following the analytical protocol prescribed by ISO standard 11466.

SAFETY INFORMATION

The usual laboratory safety precautions apply.

INSTRUCTIONS FOR USE AND INTENDED USE

Before withdrawing a sub-sample, the bottle shall be shaken for at least two minutes to ensure homogenisation of the contents.
Analytical results have to be corrected to the dry mass content of the material which shall be determined according to ISO 11465 using a separate sub-sample. The dry mass content of the material given in the table above (99.1 %) should be regarded as being indicative only.

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 methods.

Please note that the certified values are not valid if there are deviations from the extraction procedure prescribed by ISO standard 11466. In particular, extraction with aqua regia using microwave assisted closed vessel procedures is prone to result in higher mass fractions.

STORAGE

Samples should be stored at (20 ± 3) °C in the dark. The material picks up moisture when in prolonged contact with humid air. Spoilage by moulds may occur at water contents exceeding 8 % by mass and damage the whole sample. Care should be taken to avoid moisture pick up once the bottles are opened.

However, BAM cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

LEGAL NOTICE

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REFERENCE

ISO 11466:1995-03: Soil quality – Extraction of trace elements soluble in aqua regia

TECHNICAL REPORT

A detailed technical report describing the analytical procedures and the statistical treatment of the analytical data used to certify the reference material ERM®-CC018 is available on request or can be downloaded from BAM website (www.bam.de/en/fachthemen/referenzmaterialien/index.htm).

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