Certification Report

Certified Reference Material BAM-U112a

Aqua Regia Extractable Trace Elements in Soil

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Summary

This report describes the certification of a reference material for the determination of aqua regia extractable mass fractions of As, Cd, Co, Cr, Cu, Hg, Ni, Pb, V and Zn in a sandy soil.
The certified reference material BAM-U112a is intended for the verification of measurement results obtained by standardised analytical protocols as well as for the validation of modified or new analytical procedures.

The following mass fractions were certified:

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.

CRM BAM-U112a is provided as a powder with particle sizes below 125 µm in a 100 mL screw-capped brown glass bottle containing (55 ± 1) g. 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).

The certified values are valid for a period of 36 months beginning with the dispatch of the reference material from BAM.

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1 Introduction

The determination of trace element contents of soils after extraction with aqua regia is a very common analytical task in the daily practice of testing laboratories involved in the assessment and monitoring of environmental pollution. In many countries it is prescribed by legislation which also demands that the reliability of measurement results must be assured by appropriate quality control.
A very useful tool for this purpose is the analysis of certified reference materials (CRM). However, in this context it is important to note that the used CRMs should match the matrix composition of the samples to be tested as close as possible. Given the wide variation of "real world" soils in terms of their mineral components, organic matter and anthropogenic contamination, there is a need for a wide range of different soil reference materials.

CRM BAM-U112a was prepared as a successor of the sold-out CRM BAM-U112 with an almost identical matrix composition. The intention was to certify aqua regia extractable mass fractions of the environmentally most relevant elements As, Cd, Co, Cr, Cu, Hg, Ni, Pb, V and Zn. Because these mass fractions are operationally defined, strict adherence to an agreed extraction procedure was requested as basic prerequisite for obtaining comparable analytical results.
For this reason, in the course of the certification project the two extraction protocols given in EN 16174 [1] as Method A and Method B were followed (see Annex E). Method A is based on thermal heating in an open apparatus under reflux conditions in analogy to the extraction procedure prescribed by ISO 11466 [2], whereas Method B is performed as a pressurised microwave-assisted extraction in a closed vessel at (175 ± 5) °C.

The certification of reference material BAM-U112a was carried out according to the "Guidelines for the production of BAM reference materials" [3] and in full compliance with the relevant ISO Guides [4 - 6].

2 Candidate material

The starting material for preparing CRM BAM-U112a was a sandy soil collected during a remediation campaign on the same industrially contaminated site in the Berlin region (Germany) as the raw material for its predecessor CRM BAM-U112.
The excavated field-moist soil 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 125 µm. Homogenisation and bottling of the ground material were performed using a spinning riffler according to the so-called "cross-riffling scheme" [7]. A total of 400 bottles each containing (55 ± 1) g was produced in August 2013 and stored at (20 ± 3) °C.

3 Homogeneity study

A total of 12 bottled units of the candidate material were selected using a systematic sample picking scheme following the sequence of bottling to give complete coverage of the production batch.

The selected units were analysed in triplicate each applying the extraction procedure according to EN 16174, Method B, and using sample intakes of 0.5 g.
Extraction solutions were analysed under repeatability conditions after randomisation in one run with one calibration, respectively. All measurement results and used analytical method are given in Annex C.

The estimates of analyte-specific inhomogeneity contributions u_{bb} to be included into the total uncertainty budgets were calculated according to ISO Guide 35 [6] using Eq. (1) and Eq. (2):

$$s_{bb} = \sqrt{\frac{MS_{among} - MS_{within}}{n}} \qquad (1)$$

$$u^{*}_{bb} = \sqrt{\frac{MS_{within}}{n}} \sqrt[4]{\frac{2}{N(n-1)}} \qquad (2)$$

where:

MS_{among}	mean of squared deviations between bottles (from 1-way ANOVA)
MS_{within}	mean of squared deviations within bottles (from 1-way ANOVA)
n	number of replicate sub-samples per bottle
N	number of bottles selected for homogeneity study

s_{bb} signifies the between-bottle standard deviation, whereas u^{*}_{bb} denotes the maximum heterogeneity that can potentially be hidden by an insufficient repeatability of the applied measurement method (which has to be considered as the minimum uncertainty contribution). In any case the larger of the two values was used as u_{bb}. Eq. (1) does not apply if MS_{within} is larger than MS_{among}.
The calculated relative values of s_{bb}, u^{*}_{bb} , and u_{bb} are given in the following Table 1.

Table 1: Relative uncertainty contributions due to possible sample inhomogeneity

Element	$s_{bb,r}$ (%)	$u^{*}_{bb,r}$ (%)	$u_{bb,r}$ (%)
As	0.56	0.81	0.81
Cd	0.95	0.71	0.95
Co	0.97	0.84	0.97
Cr	0.33	0.70	0.70
Cu	$MS_{among} < MS_{within}$	1.03	1.03
Hg	$MS_{among} < MS_{within}$	1.63	1.63
Ni	$MS_{among} < MS_{within}$	1.12	1.12
Pb	1.02	0.64	1.02
V	$MS_{among} < MS_{within}$	0.72	0.72
Zn	0.29	0.70	0.70

NOTE: Although the minimum sample intake required for extraction according to Method A is much larger than for Method B (3 g vs. 0.5 g) and therefore less impact of possible sample inhomogeneity on measurement results is to be expected, the same relative uncertainty contributions $u_{bb,r}$ were taken into account when calculating the combined uncertainties of certified values referring to extraction according to Method A and Method B, respectively.

4 Stability study

Based on many years of experience gained at BAM with reference materials of similar matrix composition (in particular, with the predecessor CRM BAM-U112), it is very unlikely that aqua regia extractable mass fractions of elements will change if the samples are stored and handled properly. Nevertheless a stability check of the bottled material was performed.
Therefore, immediately after bottling selected units were stored at a temperature of -20 °C, +20 °C, +40 °C and +60 °C, respectively.

After a storage time of 2, 4 and 6 months, respectively, two bottles per temperature were analysed in duplicate for aqua regia extractable mass fractions applying extraction according to EN 16174, Method A. This procedure was chosen because of the larger sample intake compared to Method B in view to minimise the impact of possible sample inhomogeneity.
The extraction solutions were analysed under repeatability conditions in one run with one calibration. The measurement results (see Annex D) were evaluated for each analyte calculating the ratios R_t and their uncertainties u_t using Eq. (3) and Eq. (4), respectively:

$$R_t = X_t / X_{-20\,°C} \qquad (3)$$

$$u_t = (CV_t^2 + CV_{-20\,°C}^2)^{1/2} * R_t \qquad (4)$$

where X_t and $X_{-20\,°C}$ are the mean values of four analyses of samples stored at temperature t and of samples stored at the reference temperature -20 °C, respectively. CV_t and $CV_{-20\,°C}$ are the corresponding coefficients of variation.

Table 2: Results of the stability test after a storage time of 2, 4 and 6 months, respectively

Element	Storage time	$R_t \pm u_t$		
		Storage at 20 °C	Storage at 40 °C	Storage at 60 °C
As	2 months	0.9961 ± 0.0281	0.9990 ± 0.0305	1.0029 ± 0.0261
	4 months	1.0029 ± 0.0264	0.9962 ± 0.0261	1.0200 ± 0.0287
	6 months	1.0067 ± 0.0258	1.0135 ± 0.0278	1.0019 ± 0.0286
Cd	2 months	0.9947 ± 0.0363	1.0005 ± 0.0392	1.0033 ± 0.0320
	4 months	0.9956 ± 0.0278	1.0144 ± 0.0389	1.0108 ± 0.0265
	6 months	0.9955 ± 0.0273	0.9786 ± 0.0216	0.9844 ± 0.0252
Co	2 months	0.9958 ± 0.0166	0.9982 ± 0.0158	0.9984 ± 0.0248
	4 months	0.9970 ± 0.0242	1.0015 ± 0.0218	0.9861 ± 0.0253
	6 months	1.0057 ± 0.0523	1.0148 ± 0.0604	0.9987 ± 0.0499
Cr	2 months	0.9996 ± 0.0265	1.0136 ± 0.0326	1.0124 ± 0.0304
	4 months	0.9967 ± 0.0159	1.0088 ± 0.0176	1.0106 ± 0.0150
	6 months	0.9924 ± 0.0121	0.9991 ± 0.0116	0.9998 ± 0.0145
Cu	2 months	1.0090 ± 0.0364	1.0225 ± 0.0322	1.0063 ± 0.0364
	4 months	0.9958 ± 0.0305	1.0041 ± 0.0299	1.0115 ± 0.0314
	6 months	1.0015 ± 0.0176	0.9923 ± 0.0148	0.9927 ± 0.0203
Hg	2 months	0.9918 ± 0.0385	1.0245 ± 0.0472	0.9975 ± 0.0313
	4 months	1.0237 ± 0.0568	1.0205 ± 0.0607	1.0340 ± 0.0656
	6 months	1.0026 ± 0.0410	1.0083 ± 0.0423	0.9795 ± 0.0406
Ni	2 months	0.9806 ± 0.0416	0.9845 ± 0.0381	0.9961 ± 0.0383
	4 months	1.0099 ± 0.0294	1.0197 ± 0.0272	1.0178 ± 0.0263
	6 months	0.9942 ± 0.0216	0.9961 ± 0.0233	0.9981 ± 0.0241
Pb	2 months	0.9966 ± 0.0374	1.0039 ± 0.0380	1.0025 ± 0.0390
	4 months	1.0110 ± 0.0200	1.0040 ± 0.0344	1.0075 ± 0.0204
	6 months	0.9965 ± 0.0217	0.9930 ± 0.0233	0.9970 ± 0.0207
V	2 months	1.0062 ± 0.0185	0.9977 ± 0.0237	1.0078 ± 0.0204
	4 months	1.0039 ± 0.0108	1.0157 ± 0.0191	0.9984 ± 0.0130
	6 months	1.0116 ± 0.0140	0.9915 ± 0.0108	1.0023 ± 0.0113
Zn	2 months	0.9990 ± 0.0271	1.0086 ± 0.0304	1.0050 ± 0.0271
	4 months	0.9970 ± 0.0084	1.0061 ± 0.0244	1.0010 ± 0.0088
	6 months	0.9930 ± 0.0168	0.9836 ± 0.0270	0.9935 ± 0.0160

If one postulates that aqua regia extractable mass fractions of samples stored at -20 °C do not change over time, in case of ideal sample stability at a higher storage temperature *t* the ratio R_t should be 1. In reality, however, unavoidable random variations of measurement results as well as sample inhomogeneity have to be taken into account. Thus, a material can be considered stable at storage temperature *t* if the value 1 is comprised between $R_t - u_t$ and $R_t + u_t$. This precondition is fulfilled for all analytes and storage temperatures under test.

Measurement results obtained in the course of the stability study were evaluated using software SoftCRM, version 1.2.2 [8]. A significant impact of storage conditions on the stability of the certified properties could not be detected, neither of storage time nor of temperature (up to +60 °C). In all cases the slope of the linear regression did not differ significantly from zero. For this reason, taking into account an expansion of the total uncertainty of the certified values by an 'instability component' was not considered necessary.

Stability testing will be continued by further measurements of units stored at -20 °C, +20 °C and +40 °C over the period of availability of the material. Thus, the validity of the expiration date of three years after dispatch given in the certificate is maintained by post-certification measurements performed at BAM.

5 Inter-laboratory comparison (ILC)

5.1 Design of the certification study

The certification study was carried out as an inter-laboratory comparison (ILC). Laboratories invited to participate were selected on the basis of their expertise demonstrated in the course of preceding proficiency tests or validation projects organised by BAM in the field of inorganic soil analysis. All but one declared to operate an internal quality management system accredited to ISO/IEC 17025 [9] and covering the determination of aqua regia extractable trace elements in soils.
The strict observance of the requirements of EN 16174 concerning the extraction procedures to be applied as well as additional instructions given by BAM had been assured by all laboratories in advance. For analysis of the extraction solutions, participants were free to use the method of their choice.

Each participating laboratory received one unit of the bottled candidate material and had to analyse three independent sub-samples. In addition, the dry mass content of the material had to be determined on separate sub-samples by drying to constant mass at (105 ± 2) °C according to ISO 11465 [10]. All reported aqua regia extractable mass fractions were corrected to the dry mass content of the soil sample.

5.2 Participants and used analytical methods

Participating laboratories in alphabetical order (*not identical with the order of code numbers given in Annex A*):

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)

BAM participated with two laboratories. Participants with code numbers 05 and 12 applied two different analytical methods for the determination of Hg in the extraction solutions. Their results obtained with CV-AAS are marked in the respective tables and figures in Annex A with an ‘a’.

Not all of the participants did apply both extraction methods under investigation; some of them did also not determine the full range of relevant elements.

For determination of element concentrations in the aqua regia extraction solutions the following analytical methods were used by the participants:

- Cold-vapour atomic absorption spectrometry (CV-AAS)
- Cold-vapour atomic fluorescence spectrometry (CV-AFS)
- Flame atomic absorption spectrometry (F-AAS)
- Inductively coupled plasma optical emission spectrometry (ICP-OES)
- Inductively coupled plasma mass spectrometry (ICP-MS)

5.3 Statistical evaluation of ILC-results

The measurement results obtained by participating laboratories are compiled in Annex A. The bars in the graphic presentations indicate the standard deviation of individual results, the bars associated with the also plotted certified values represent the corresponding expanded uncertainties.

Statistical tests and data evaluation were performed using software SoftCRM [8]. The following tests were carried out:

Scheffé’s multiple t-test: All data sets compatible two-by-two?

Grubbs test, Dixon test and Nalimov test: Outlying means?

Cochran test: Outlying variances?

Bartlett test: Variances homogeneous?

Snedecor F-test: Differences between data sets statistically significant?

Kolmogorov-Smirnov-Lilliefors test and skewness & kurtosis test: Normality of the distribution of the means?

The results of these tests are summarised in Annex B in Tables B.1a to B.20c (with indication of the respective level of significance, α = 0.05 or α = 0.01).

Data sets identified as Nalimov-outliers at a level of significance α = 0.01 were excluded from further data processing. The same applies for data sets identified as Cochran-outliers at a level of significance α = 0.01 in case that the relative standard deviation of submitted results was larger than 10 %. Thereby the results of the homogeneity study performed by BAM were taken into account (see Annex C).

Furthermore it was decided to reject all results of lab 04 due to an inadequate overall performance in the inter-laboratory comparison, expressed both in terms of poor repeatability as well as apparently biased measurement results for a comparatively large number of elements under investigation (see Annex B).

6 Certified values and uncertainties

The unweighted means of accepted data set means were taken as the best estimates w_{char} for the values to be certified. They are expressed on a dry mass basis corresponding to a drying temperature of (105 ± 2) °C.

The standard deviations of the means of accepted data set means ($SD_M/\sqrt{N}$; see Annex A) were taken as uncertainty contributions u_{ILC} resulting from the inter-laboratory comparison.

Additionally, the following uncertainty components were taken into account:

u_{bb} uncertainty due to potentially hidden inhomogeneity of the material (see Clause 3),

u_{prec} uncertainty reflecting the average precision of accepted laboratory means and being calculated according to Eq. (5):

$$u_{\mathrm{prec}} = \sqrt{\sum_{i=1}^{N} (SD_i)^2 / nN} \qquad (5)$$

where SD_i is the standard deviation of the results of an individual participant, N is the number of accepted individual data sets, and n is the number of replicate analyses performed by each participant.

The different contributions to the overall uncertainties of the certified mass fractions were combined using the following Eq. (6):

$$u_{\mathrm{CRM}} = \sqrt{u_{\mathrm{ILC}}^2 + u_{\mathrm{bb}}^2 + u_{\mathrm{prec}}^2} \qquad (6)$$

Calculated aqua regia extractable mass fractions w_{char} and absolute values of the different uncertainty components referring to extraction according to EN 16174, Method A and Method B, are given in Table 3 and Table 4, respectively.

Table 3: Mass fractions and uncertainty components for the relevant analytes in CRM BAM-U112a; extraction according to EN 16174, Method A (before rounding)

Element	w_{char} in mg/kg	u_{ILC} in mg/kg	u_{prec} in mg/kg	u_{bb} in mg/kg	u_{CRM} in mg/kg
As	10.27	0.124	0.197	0.083	0.247
Cd	4.118	0.034	0.054	0.039	0.073
Co	3.582	0.082	0.063	0.035	0.109
Cr	80.11	0.730	0.792	0.561	1.214
Cu	75.53	0.707	1.091	0.778	1.515
Hg	16.33	0.222	0.338	0.226	0.463
Ni	10.07	0.178	0.131	0.113	0.248
Pb	197.8	1.803	2.941	2.018	3.997
V	12.65	0.303	0.198	0.091	0.373
Zn	198.1	1.771	1.792	1.387	2.876

Table 4: Mass fractions and uncertainty components for the relevant analytes in CRM BAM-U112a; extraction according to EN 16174, Method B (before rounding)

Element	w_{char} in mg/kg	u_{ILC} in mg/kg	u_{prec} in mg/kg	u_{bb} in mg/kg	u_{CRM} in mg/kg
As	10.43	0.159	0.252	0.085	0.310
Cd	4.089	0.047	0.058	0.039	0.084
Co	3.943	0.138	0.103	0.038	0.176
Cr	81.92	0.883	0.728	0.573	1.280
Cu	75.06	0.851	1.223	0.773	1.679
Hg	15.86	0.300	0.346	0.259	0.526
Ni	11.22	0.236	0.340	0.126	0.432
Pb	198.8	1.910	2.432	2.028	3.698
V	13.96	0.308	0.285	0.101	0.432
Zn	199.6	1.766	2.357	1.397	3.260

The expanded uncertainty U was obtained by multiplying the combined uncertainty u_{CRM} by a coverage factor k = 2, giving a level of confidence of approximately 95 % to be associated with the interval ± U around the certified mass fraction [11].

The certified aqua regia extractable mass fractions of trace elements in CRM BAM-U112a and their corresponding expanded uncertainties were rounded according to DIN 1333 [12] giving the values summarised in Table 5.

Table 5: Certified aqua regia extractable mass fractions and their expanded uncertainties for trace elements in CRM BAM-U112a (*after rounding*)

Element	Extraction according to **EN 16174, Method A**		Extraction according to **EN 16174, Method B**	
	Mass fraction in mg/kg	**Uncertainty *U*** in mg/kg	**Mass fraction** in mg/kg	**Uncertainty *U*** 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

7 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.

8 Additional material information

The main matrix constituents of the bottled CRM BAM-U112a were determined by semi-quantitative X-ray fluorescence analysis. Obtained non-certified results are given in Table 6 (*for information only*).

Table 6: Matrix composition of CRM BAM-U112a (semi-quantitative results)

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

Further informative analytical results obtained in the course of sample characterisation are summarised in Table 7.

Table 7: Informative secondary parameters characterising CRM-BAM-U112a

Parameter	Mass fraction in %	Analytical method
Dry mass content at 105 °C	99.5	ISO 11465 [10]
Loss on ignition at 550 °C	2.4	EN 12879 [13]
Total organic carbon (TOC)	1.4	ISO 10694 [14]
Total inorganic carbon (TIC)	0.3	ISO 10694 [14]

9 Information on the proper use of CRM BAM-U112a

9.1 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 [2].

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.

9.2 Transport, storage and handling

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.

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 (Method A and Method B) 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.

9.3 Shelf life

The initial stability study after storage of selected units at different temperatures did not reveal any statistically significant deterioration of the certified properties. However, starting with dispatch of the material from BAM the validity of the certificate expires after 36 months. Post-certification measurements will be conducted in appropriate periods to keep this information up to date.

9.4 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. It is strongly recommended to handle and dispose the reference material in accordance with the guidelines for hazardous materials legally in force at the site of end use and disposal.

9.5 Legal notice

Neither BAM, its contractors nor any person acting on their behalf:

(a) make any warranty or representation, express or implied, that the use of any information, material, apparatus, method or process disclosed in this document does not infringe any privately owned intellectual property rights; or

(b) assume any liability with respect to, or for damages resulting from, the use of any information, material, apparatus, method or process disclosed in this document.

10 References

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[2] ISO 11466 (1995): Soil quality – Extraction of trace elements soluble in aqua regia

[3] 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)

[4] ISO Guide 31 (2000): Reference materials – Contents of certificates and labels

[5] ISO Guide 34 (2009): General requirements for the competence of reference material producers

[6] ISO Guide 35 (2006): Reference materials – General and statistical principles for certification

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[9] EN ISO/IEC 17025 (2005): General requirements for the competence of testing and calibration laboratories

[10] ISO 11465 (1993): Soil quality – Determination of dry matter and water content on a mass basis. Gravimetric method

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

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[14] ISO 10694 (1995): Soil quality – Determination of organic and total carbon after dry combustion (elementary analysis)

11 Annexes

Annex A: Measurement results of ILC-participants

Annex B: Outcome of statistical tests on ILC-results

Annex C: Homogeneity study (measurement results)

Annex D: Stability study (measurement results)

Annex E: Extraction procedures according to EN 16174

List of used abbreviations
(if not explained elsewhere in the report)

M	arithmetic mean of means
N	number of individual data sets
SD	standard deviation of an individual data set
SD_M	standard deviation of the mean of data set means

Table A.1a: Measurement results of ILC-participants **As (A)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
03	9.777	9.477	9.567	9.61	0.154	ICP-MS
07	9.91	9.75	9.2	9.62	0.372	ICP-MS
18	9.74	9.77	9.64	9.72	0.068	ICP-OES
02	9.79	9.68	9.79	9.75	0.064	ICP-MS
05	9.838	9.965	9.996	9.93	0.084	ICP-OES
01	9.95	10.1	10.2	10.08	0.126	ICP-OES
15	10.8	9.92	9.86	10.19	0.526	ICP-OES
06	10.08	10.21	10.35	10.21	0.135	ICP-MS
09	10.02	9.994	10.99	10.33	0.568	ICP-MS
16	10.40	10.79	10.45	10.55	0.212	ET-AAS
08	10.5	10.5	10.8	10.60	0.173	ICP-MS
17	11.3	10.4	10.2	10.63	0.586	ICP-OES
12	11.1	10.7	10.8	10.87	0.208	ICP-MS
11	11.16	10.78	10.82	10.92	0.209	ICP-MS
10	10.3	11.6	11.1	11.00	0.656	ICP-OES
04	11.95	11.89	12.05	11.96	0.081	ICP-MS

M (mg/kg): **10.27**

SD_M (mg/kg): 0.480

$SD_M/\sqrt{N}$ (mg/kg): 0.124

Data set of lab 04 was not taken into account (see Chapter 5.2 and Annex B).

Table A.1b: Measurement results of ILC-participants **As (B)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
07	9.85	9.35	9.28	9.49	0.311	ICP-MS
18	9.77	9.63	9.74	9.71	0.074	ICP-OES
13	10.57	9.66	8.95	9.73	0.812	ICP-OES
02	9.68	9.91	9.92	9.84	0.136	ICP-MS
06	10.22	10.30	10.43	10.32	0.106	ICP-MS
03	10.6	10.2	10.4	10.40	0.200	ICP-MS
08	10.5	10.5	10.3	10.43	0.115	ICP-MS
12	10.3	10.5	10.5	10.43	0.115	ICP-MS
01	10.4	10.7	10.5	10.53	0.153	ICP-OES
09	10.26	11.06	10.40	10.57	0.427	ICP-MS
15	11.72	10.83	9.56	10.70	1.086	ICP-OES
10	10.7	9.28	12.8	10.93	1.771	ICP-OES
16	10.91	11.62	10.45	10.99	0.589	ET-AAS
05	11.31	11.30	11.45	11.35	0.084	ICP-OES
11	11.53	11.20	11.69	11.47	0.250	ICP-MS
04	10.78	12.41	11.93	11.71	0.838	ICP-MS

M (mg/kg): **10.43**

SD_M (mg/kg): 0.596

$SD_M/\sqrt{N}$ (mg/kg): 0.159

Data sets of labs 04 and 10 were not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **As**

(graphical presentation) Aqua regia extractable mass fraction

Fig. A.1a: Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

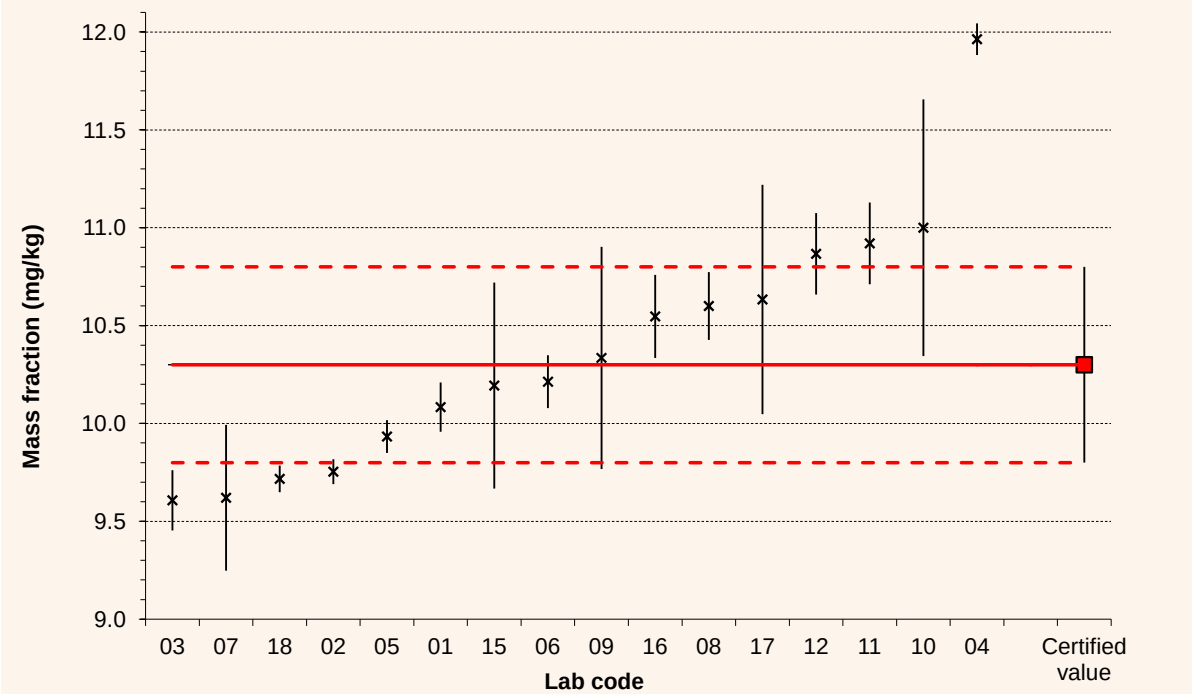


Fig. A.1b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

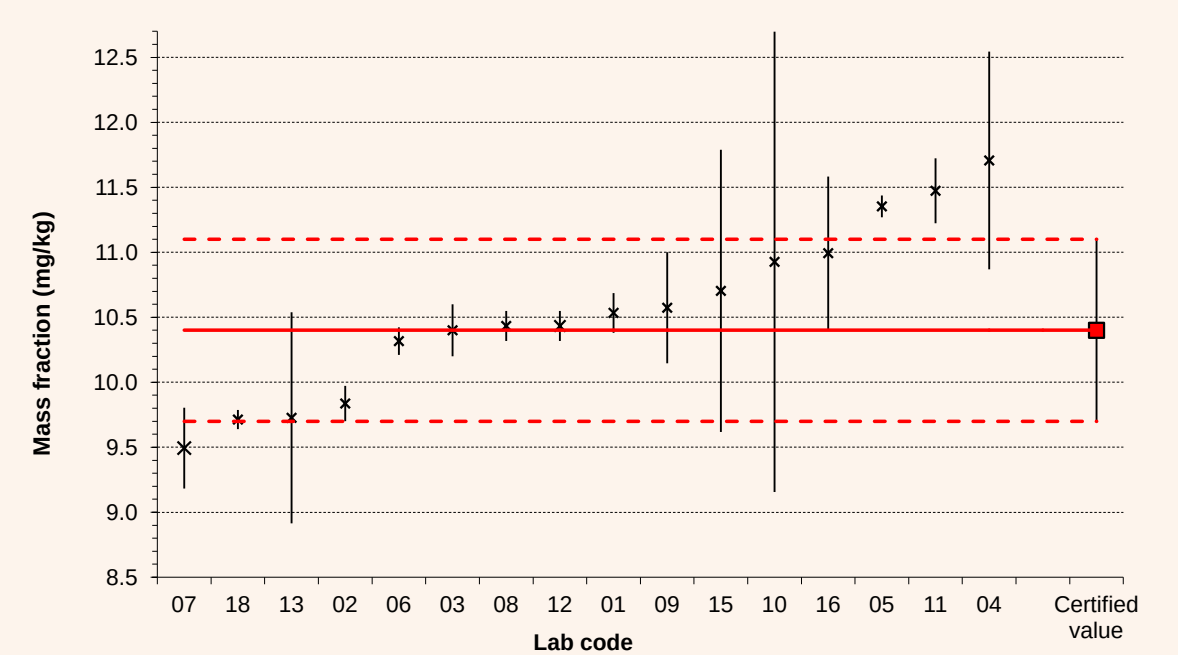


Table A.2a: Measurement results of ILC-participants **Cd (A)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Lab Code	**Sub-sample 1** (mg/kg)	**Sub-sample 2** (mg/kg)	**Sub-sample 3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)	**Analytical method**
03	3.580	3.637	3.641	3.619	0.034	ICP-MS
16	3.97	3.92	3.98	3.957	0.032	ET-AAS
07	4.12	3.96	3.82	3.967	0.150	ICP-MS
06	4.019	3.916	3.966	3.967	0.052	ICP-MS
04	3.94	3.80	4.17	3.970	0.187	ICP-MS
01	3.95	4.05	4.05	4.017	0.058	ICP-OES
18	4.02	4.04	4.08	4.047	0.031	ICP-OES
02	4.14	4.12	4.07	4.110	0.036	ICP-MS
15	4.145	4.171	4.020	4.112	0.081	ICP-OES
10	4.30	3.82	4.23	4.117	0.259	ICP-OES
08	4.06	4.17	4.19	4.140	0.070	ICP-MS
11	4.114	4.156	4.152	4.141	0.023	ICP-MS
05	4.118	4.090	4.254	4.154	0.088	ICP-OES
09	3.797	4.003	4.789	4.196	0.523	ICP-MS
19	4.28	4.28	4.26	4.273	0.012	ICP-OES
17	4.27	4.28	4.32	4.290	0.026	ICP-OES
12	4.39	4.33	4.38	4.367	0.032	ICP-MS

M (mg/kg): **4.118**

SD_M (mg/kg): 0.126

$SD_M/\sqrt{N}$ (mg/kg): 0.034

Data sets of labs 03, 04 and 09 were not taken into account (see Chapter 5.2 and Annex B).

Table A.2b: Measurement results of ILC-participants **Cd (B)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
16	3.55	3.47	3.49	3.503	0.042	ET-AAS
13	4.019	3.758	3.593	3.790	0.215	ICP-OES
07	4.01	3.87	3.77	3.883	0.121	ICP-MS
10	3.98	3.85	3.86	3.897	0.072	ICP-OES
03	3.98	3.89	3.88	3.917	0.055	ICP-MS
12	3.99	3.96	3.95	3.967	0.021	ICP-MS
09	4.041	4.145	3.941	4.042	0.102	ICP-MS
06	4.041	3.933	4.193	4.056	0.131	ICP-MS
02	4.18	4.03	4.03	4.080	0.087	ICP-MS
18	4.10	4.12	4.05	4.090	0.036	ICP-OES
04	3.84	4.23	4.24	4.103	0.228	ICP-MS
05	4.287	4.005	4.114	4.135	0.142	ICP-OES
01	4.14	4.17	4.13	4.147	0.021	ICP-OES
19	4.29	4.27	4.33	4.297	0.031	ICP-OES
08	4.40	4.29	4.26	4.317	0.074	ICP-MS
11	4.400	4.302	4.364	4.355	0.050	ICP-MS
15	4.290	4.283	4.513	4.362	0.131	ICP-OES

M (mg/kg): **4.089**

SD_M (mg/kg): 0.182

$SD_M/\sqrt{N}$ (mg/kg): 0.047

Data sets of labs 04 and 16 were not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **Cd**

(graphical presentation) Aqua regia extractable mass fraction

Fig. A.2a: Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

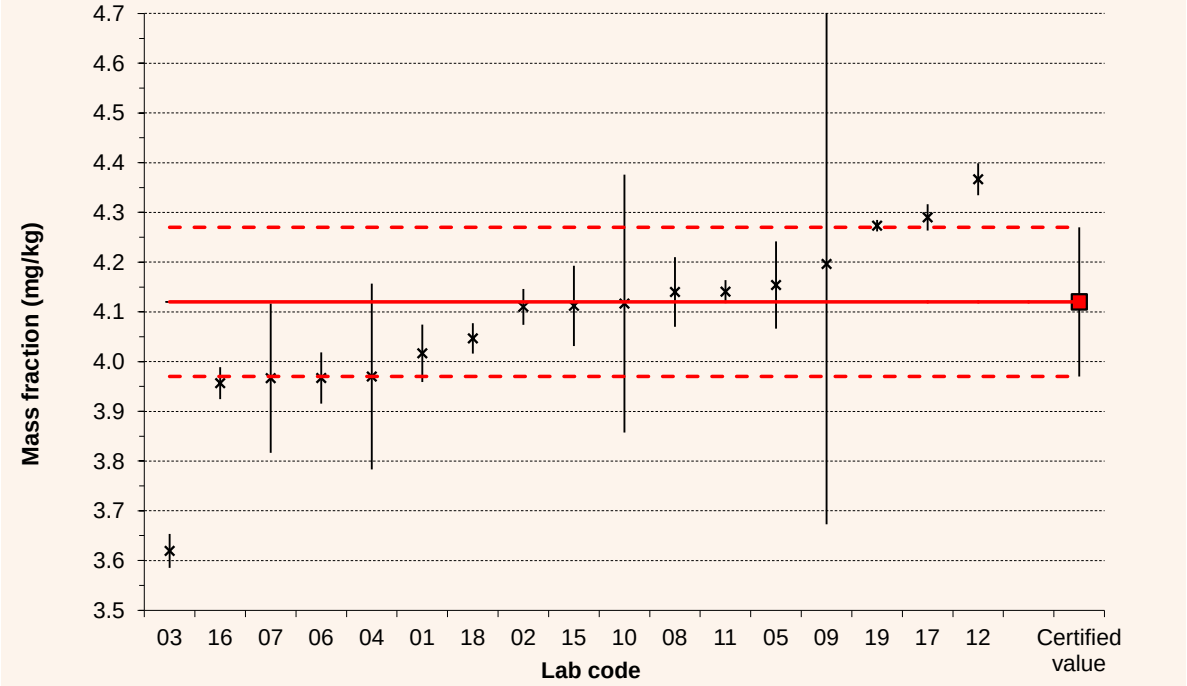


Fig. A.2b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

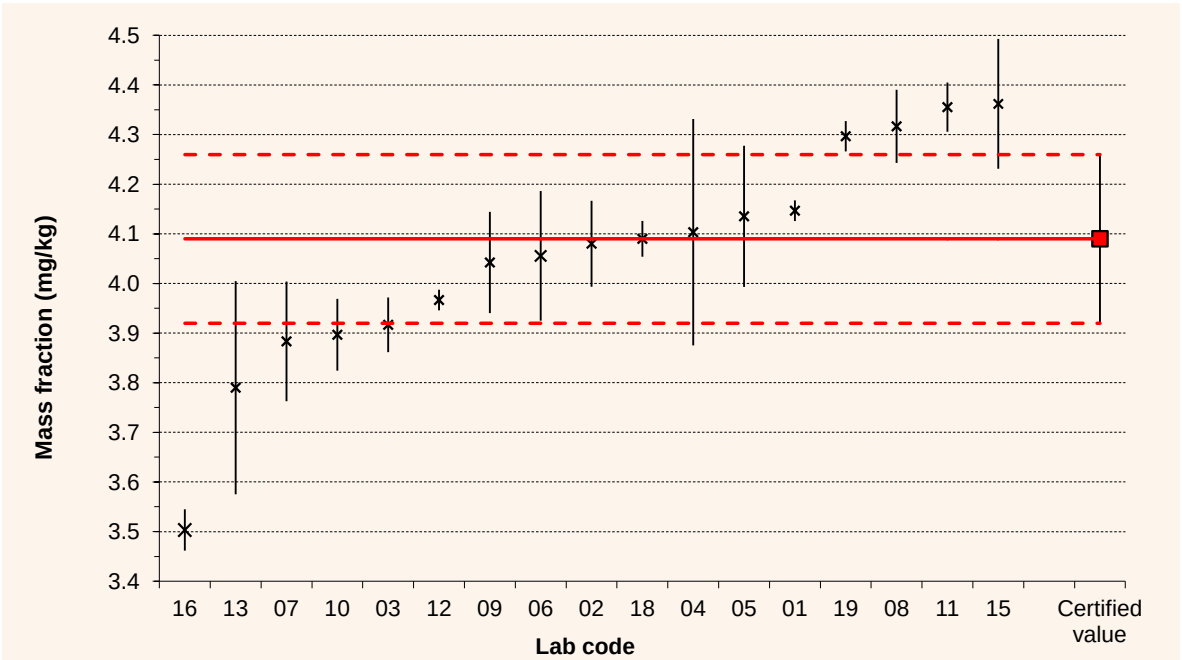


Table A.3a: Measurement results of ILC-participants **Co (A)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
11	2.674	3.166	3.030	2.957	0.254	ICP-MS
09	2.901	3.024	2.977	2.967	0.062	ICP-MS
07	3.42	3.27	3.12	3.270	0.150	ICP-MS
03	3.339	3.327	3.409	3.358	0.044	ICP-MS
02	3.41	3.38	3.43	3.407	0.025	ICP-MS
18	3.41	3.58	3.47	3.487	0.086	ICP-OES
06	3.548	3.561	3.549	3.553	0.007	ICP-MS
08	3.57	3.61	3.67	3.617	0.050	ICP-MS
14	3.79	3.68	3.39	3.620	0.207	ICP-OES
04	3.72	3.59	3.67	3.660	0.066	ICP-MS
16	3.58	3.68	3.72	3.660	0.072	ET-AAS
17	3.78	3.80	3.74	3.773	0.031	ICP-OES
12	3.84	4.04	3.74	3.873	0.153	ICP-MS
05	3.936	3.811	3.932	3.893	0.071	ICP-OES
01	3.90	3.90	3.99	3.930	0.052	ICP-OES
15	4.036	3.981	3.883	3.967	0.078	ICP-OES
10	3.95	4.04	3.97	3.987	0.047	ICP-OES

M (mg/kg): **3.582**

SD_M (mg/kg): 0.328

$SD_M/\sqrt{N}$ (mg/kg): 0.082

Data set of lab 04 was not taken into account (see Chapter 5.2 and Annex B).

Table A.3b: Measurement results of ILC-participants **Co (B)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
13	2.80	3.12	3.01	2.977	0.163	ICP-OES
18	3.51	3.35	3.60	3.487	0.127	ICP-OES
04	3.26	3.79	3.50	3.517	0.265	ICP-MS
01	3.47	3.60	3.54	3.537	0.065	ICP-OES
09	3.645	3.547	3.501	3.564	0.074	ICP-MS
03	3.618	3.800	3.430	3.616	0.185	ICP-MS
14	3.19	3.95	3.80	3.647	0.403	ICP-OES
16	3.58	3.75	3.70	3.677	0.087	ET-AAS
08	3.81	4.06	3.63	3.833	0.216	ICP-MS
02	3.99	3.76	3.76	3.837	0.133	ICP-MS
07	3.99	3.89	3.73	3.870	0.131	ICP-MS
12	3.47	4.11	4.05	3.877	0.353	ICP-MS
06	4.174	4.118	4.176	4.156	0.033	ICP-MS
11	4.534	4.316	4.555	4.468	0.132	ICP-MS
10	4.73	4.73	4.64	4.700	0.052	ICP-OES
05	4.744	4.890	4.749	4.794	0.083	ICP-OES
15	5.112	4.908	5.106	5.042	0.116	ICP-OES

M (mg/kg): **3.943**

SD_M (mg/kg): 0.553

$SD_M/\sqrt{N}$ (mg/kg): 0.138

Data set of lab 04 was not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **Co**

(graphical presentation) Aqua regia extractable mass fraction

Fig. A.3a: Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

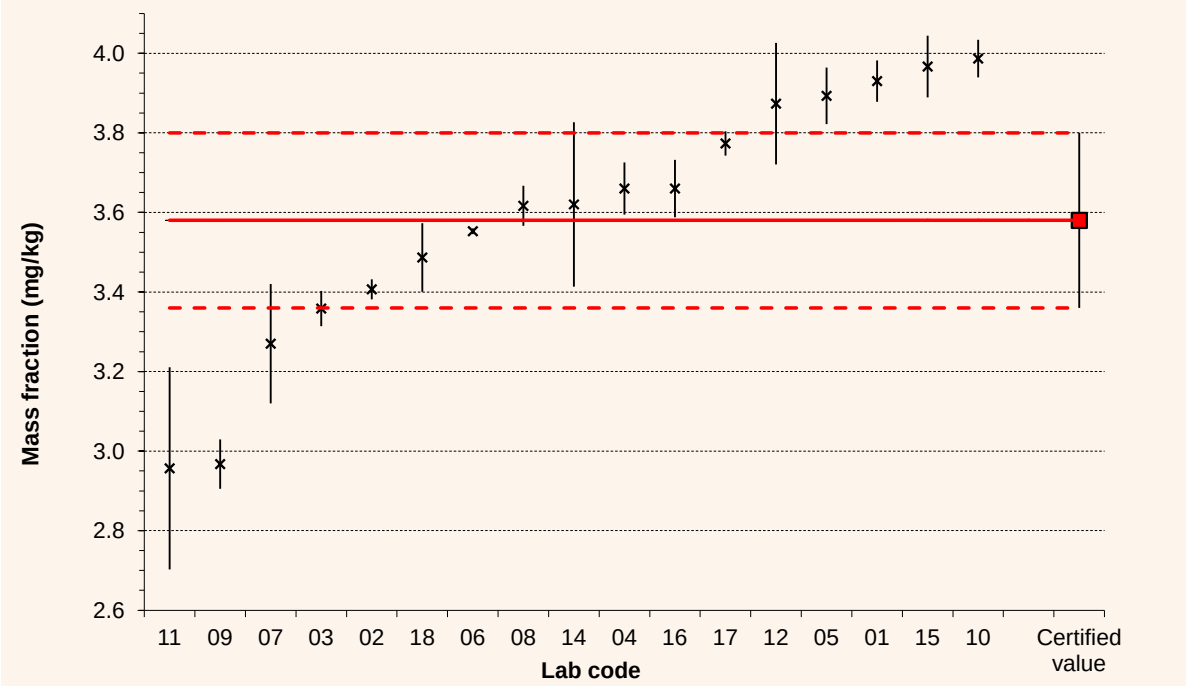


Fig. A.3b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

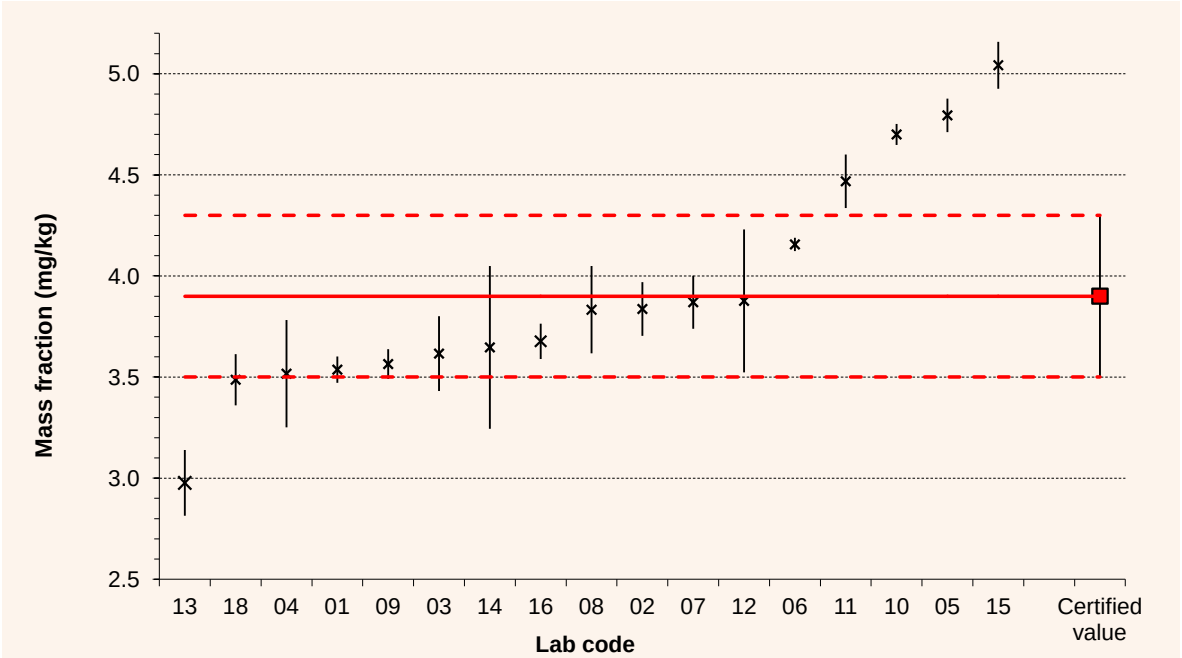


Table A.4a: Measurement results of ILC-participants **Cr (A)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
10	72.4	79.3	74.1	75.27	3.595	ICP-OES
02	75.3	75.4	75.8	75.50	0.265	ICP-MS
11	76.63	77.97	76.68	77.09	0.760	ICP-MS
14	76.9	77.7	78.0	77.53	0.569	ICP-OES
07	80.3	79.0	74.4	77.90	3.100	ICP-MS
06	78.60	78.75	79.25	78.87	0.340	ICP-MS
09	79.6	79.0	78.6	79.07	0.503	ICP-OES
15	78.63	80.22	79.90	79.58	0.841	ICP-OES
17	79.9	78.9	81.2	80.00	1.153	ICP-OES
05	79.75	79.84	81	80.20	0.697	ICP-OES
03	80.83	80.83	79.32	80.33	0.872	ICP-OES
19	81.1	80.8	81.2	81.03	0.208	ICP-OES
08	81.8	82.2	83.3	82.43	0.777	ICP-MS
01	82.8	84.6	83.5	83.63	0.907	ICP-OES
18	83.0	84.5	83.6	83.70	0.755	ICP-OES
16	84.2	83.4	86.4	84.67	1.553	F-AAS
12	85.3	85.5	84.3	85.03	0.643	ICP-MS
04	88.86	87.18	89.51	88.52	1.202	ICP-MS

M (mg/kg): **80.11**

SD_M (mg/kg): 3.009

$SD_M/\sqrt{N}$ (mg/kg): 0.730

Data set of lab 04 was not taken into account (see Chapter 5.2 and Annex B).

Table A.4b: Measurement results of ILC-participants **Cr (B)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
13	76.91	73.17	73.41	74.50	2.093	ICP-OES
02	75.1	77.4	76.2	76.23	1.150	ICP-MS
07	77.8	75.9	78.7	77.47	1.429	ICP-MS
14	77.7	77.0	79.2	77.97	1.124	ICP-OES
03	81.8	80.1	81.3	81.07	0.874	ICP-OES
10	81.5	80.7	82.1	81.43	0.702	ICP-OES
12	78.3	83.2	84.1	81.87	3.121	ICP-MS
09	82.8	81.8	81.6	82.07	0.643	ICP-OES
15	83.16	82.71	81.72	82.53	0.737	ICP-OES
08	83.0	82.2	82.4	82.53	0.416	ICP-MS
05	83.21	81.20	84.05	82.82	1.464	ICP-OES
19	83.2	84.2	81.9	83.10	1.153	ICP-OES
06	83.65	83.91	83.41	83.66	0.250	ICP-MS
18	85.5	84.2	85.4	85.03	0.723	ICP-OES
04	76.17	89.35	89.69	85.07	7.710	ICP-MS
11	86.14	84.80	86.59	85.84	0.931	ICP-MS
01	86.1	87.0	87.0	86.70	0.520	ICP-OES
16	88.7	87.2	87.4	87.77	0.814	F-AAS

M (mg/kg): **81.92**

SD_M (mg/kg): 3.640

$SD_M/\sqrt{N}$ (mg/kg): 0.883

Data set of lab 04 was not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **Cr**

(graphical presentation) Aqua regia extractable mass fraction

Fig. A.4a: Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

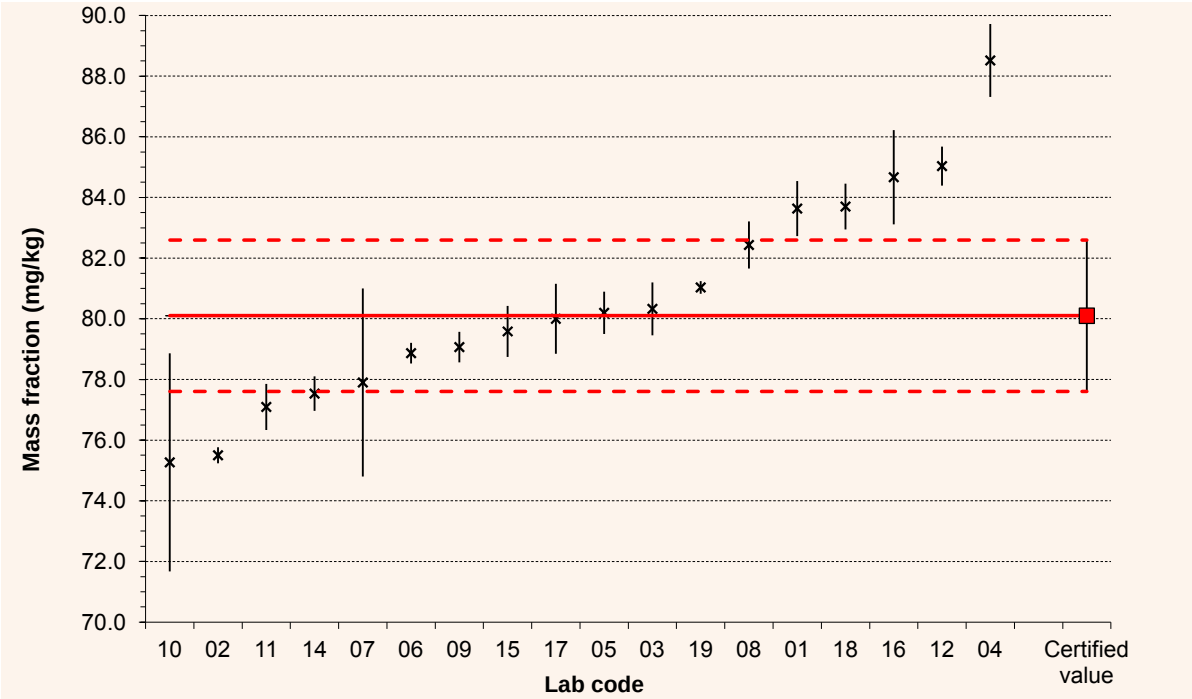


Fig. A.4b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

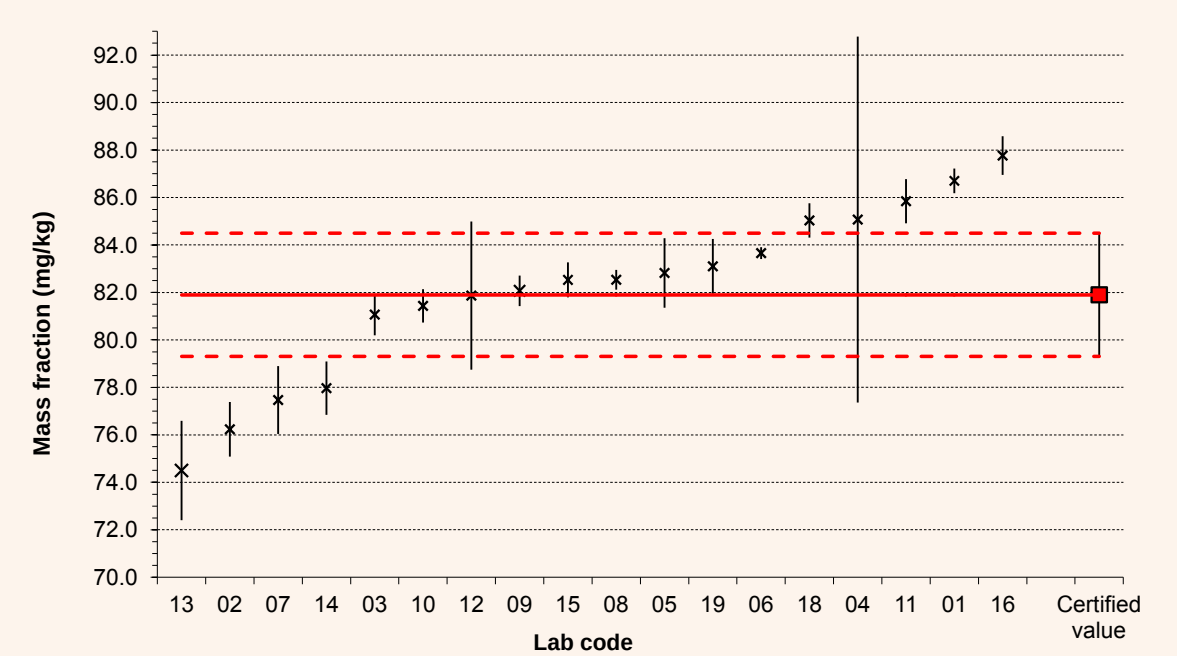


Table A.5a: Measurement results of ILC-participants **Cu (A)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
10	70.8	70.9	71.0	70.90	0.100	ICP-OES
02	71.6	71.6	71.5	71.57	0.058	ICP-MS
11	72.52	75.37	71.73	73.21	1.915	ICP-MS
06	74.39	72.64	73.32	73.45	0.882	ICP-MS
05	73.78	74.38	73.40	73.85	0.494	ICP-OES
07	78.4	73.9	69.3	73.87	4.550	ICP-MS
17	71.9	76.5	74.6	74.33	2.312	ICP-OES
03	75.11	77.62	73.61	75.45	2.026	ICP-OES
16	75.9	74.9	75.9	75.57	0.577	F-AAS
09	76.8	75.1	76.1	76.00	0.854	ICP-OES
12	75.6	76.2	77.2	76.33	0.808	ICP-MS
18	77.0	79.9	74.8	77.23	2.558	ICP-OES
15	74.09	77.57	80.98	77.55	3.445	ICP-OES
08	78.1	77.6	78.3	78.00	0.361	ICP-MS
01	81.6	79.1	79.9	80.20	1.277	ICP-OES
04	80.07	80.49	80.23	80.26	0.212	ICP-MS
19	81.1	81.6	80.4	81.03	0.603	ICP-OES

M (mg/kg): **75.53**

SD_M (mg/kg): 2.827

$SD_M/\sqrt{N}$ (mg/kg): 0.707

Data set of lab 04 was not taken into account (see Chapter 5.2 and Annex B).

Table A.5b: Measurement results of ILC-participants **Cu (B)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

Lab Code	**Sub-sample 1** (mg/kg)	**Sub-sample 2** (mg/kg)	**Sub-sample 3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)	**Analytical method**
16	68.0	69.3	71.2	69.50	1.609	F-AAS
10	71.3	72.5	70.0	71.27	1.250	ICP-OES
07	73.3	69.6	71.8	71.57	1.861	ICP-MS
14	74.8	71.3	69.5	71.87	2.695	ICP-OES
02	71.4	72.8	71.5	71.90	0.781	ICP-MS
13	75.10	70.45	71.41	72.32	2.455	ICP-OES
15	73.47	74.22	74.05	73.91	0.393	ICP-OES
03	75.5	72.5	75.1	74.37	1.629	ICP-OES
05	74.84	75.22	74.46	74.84	0.380	ICP-OES
06	73.68	77.50	74.68	75.29	1.981	ICP-MS
12	72.1	75.6	79.1	75.60	3.500	ICP-MS
08	76.0	75.4	76.3	75.90	0.458	ICP-MS
11	75.57	74.52	83.13	77.74	4.697	ICP-MS
19	79.3	76.8	78.1	78.07	1.250	ICP-OES
18	79.3	83.6	77.8	80.23	3.011	ICP-OES
09	80.2	80.1	81.3	80.53	0.666	ICP-OES
01	79.8	80.5	82.7	81.00	1.513	ICP-OES
04	75.78	92.36	86.97	85.04	8.457	ICP-MS

M (mg/kg): **75.06**

SD_M (mg/kg): 3.507

$SD_M/\sqrt{N}$ (mg/kg): 0.851

Data set of lab 04 was not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **Cu**

(graphical presentation) Aqua regia extractable mass fraction

Fig. A.5a: Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

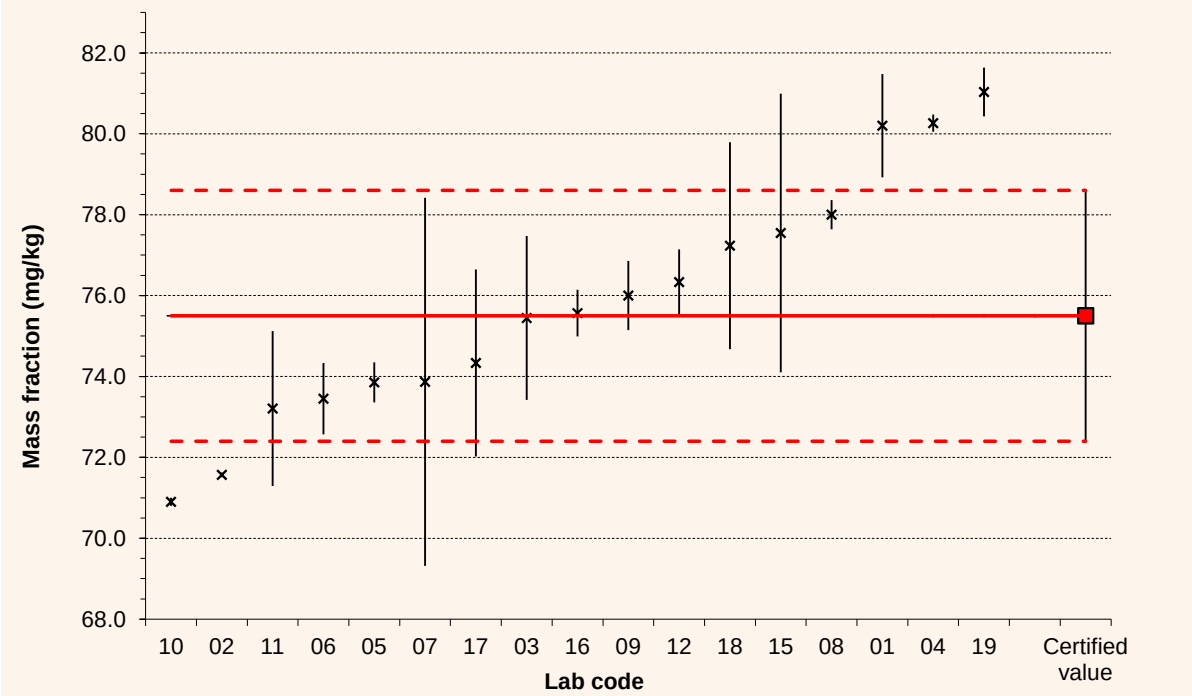


Fig. A.5b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

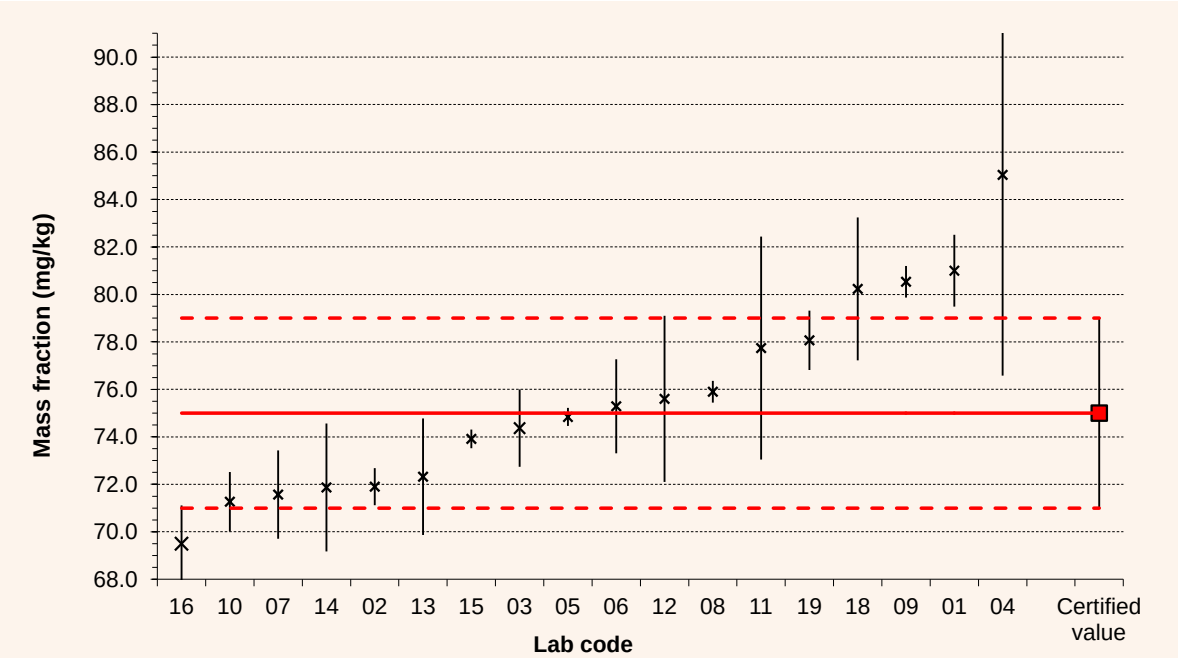


Table A.6a: Measurement results of ILC-participants **Hg (A)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Lab Code	**Sub-sample 1** (mg/kg)	**Sub-sample 2** (mg/kg)	**Sub-sample 3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)	**Analytical method**
01	15.1	14.8	14.8	14.90	0.173	CV-AAS
17	15.0	15.6	14.7	15.10	0.458	CV-AAS
19	15.3	15.4	15.3	15.33	0.058	CV-AFS
07	16.0	15.5	14.8	15.43	0.603	ICP-MS
12	16.9	15.3	14.9	15.70	1.058	ICP-MS
16	15.73	15.45	16.04	15.74	0.295	CV-AAS
18	16.4	16.2	14.9	15.83	0.814	ICP-OES
08	15.3	16.6	16.3	16.07	0.681	ICP-MS
05	16.23	16.36	16.08	16.22	0.140	ICP-OES
02	16.2	15.6	16.9	16.23	0.651	CV-AAS
06	16.00	16.16	16.75	16.30	0.395	ICP-MS
05a	16.31	16.80	16.38	16.50	0.265	CV-AAS
11	16.23	16.76	16.77	16.59	0.309	ICP-MS
14	16.4	17.7	16.9	17.00	0.656	CV-AAS
03	17.45	18.15	16.75	17.45	0.700	CV-AAS
10	17.8	16.4	18.5	17.57	1.069	CV-AAS
15	17.89	17.96	17.56	17.80	0.214	CV-AAS
12a	18.5	18.4	17.4	18.10	0.608	CV-AAS

M (mg/kg): **16.33**

SD_M (mg/kg): 0.941

$SD_M/\sqrt{N}$ (mg/kg): 0.222

Table A.6b: Measurement results of ILC-participants **Hg (B)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
13	13.21	14.91	14.21	14.11	0.854	CV-AAS
03	15.20	13.40	13.80	14.13	0.945	CV-AAS
07	14.90	14.50	13.70	14.37	0.611	ICP-MS
19	14.4	14.5	14.4	14.43	0.058	CV-AFS
02	15.2	14.7	14.7	14.87	0.289	CV-AAS
16	15.13	14.78	15.42	15.11	0.320	CV-AAS
04	14.17	15.57	16.74	15.49	1.287	ICP-MS
08	16.1	15.3	15.6	15.67	0.404	ICP-MS
12	16.8	15.7	15.5	16.00	0.700	ICP-MS
18	16.0	16.3	15.9	16.07	0.208	ICP-OES
06	16.34	16.90	15.31	16.18	0.806	ICP-MS
14	16.5	15.5	16.6	16.20	0.608	CV-AAS
11	16.56	16.10	16.53	16.40	0.257	ICP-MS
05a	16.23	16.09	17.23	16.52	0.622	CV-AAS
05	16.42	17.19	16.26	16.62	0.497	ICP-OES
01	17.2	17.0	16.7	16.97	0.252	CV-AAS
10	17.5	17.1	17.9	17.50	0.400	CV-AAS
15	19.84	17.93	17.82	18.53	1.136	CV-AAS
12a	20.9	18.9	19.5	19.77	1.026	CV-AAS

M (mg/kg): **15.86**

SD_M (mg/kg): 1.238

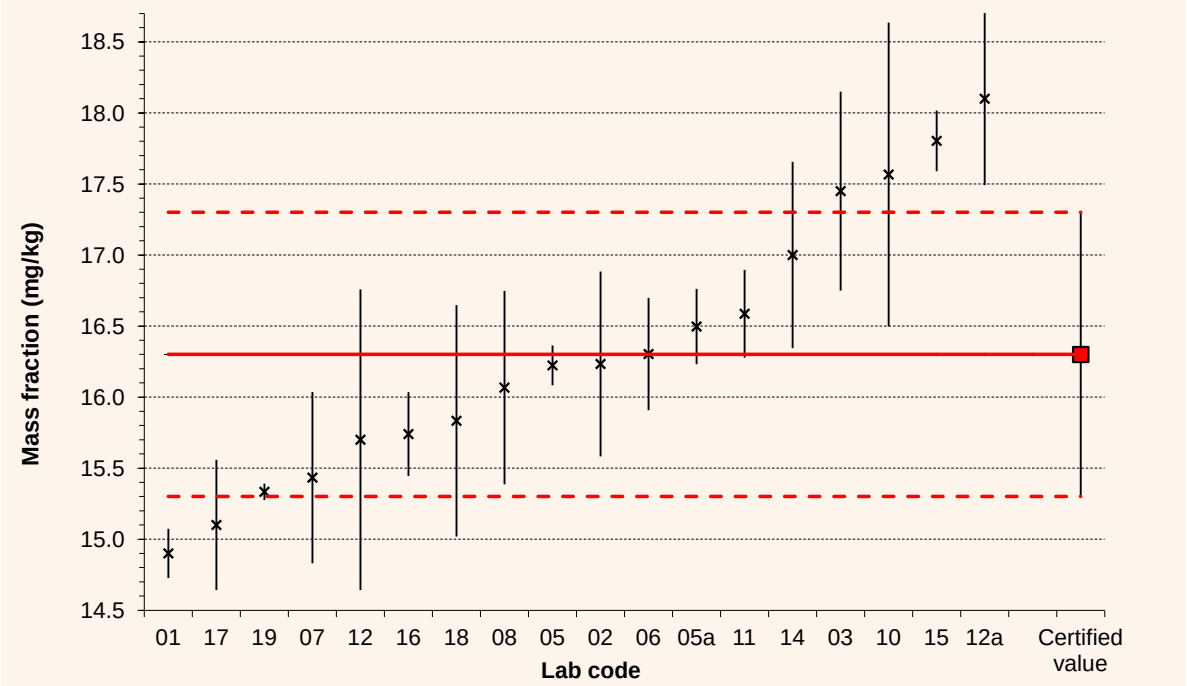
$SD_M/\sqrt{N}$ (mg/kg): 0.300

Data sets of labs 04 and 12a were not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **Hg**

(graphical presentation) Aqua regia extractable mass fraction

Fig. A.6a: Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)



Fif. A.6b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

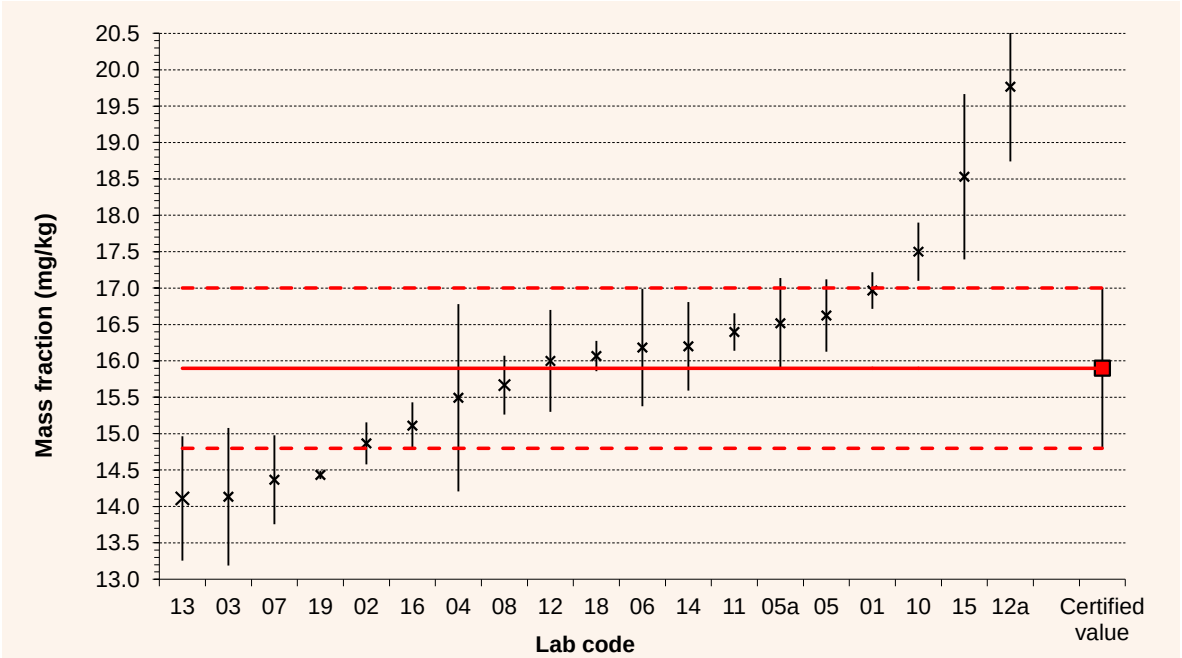


Table A.7a: Measurement results of ILC-participants **Ni (A)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
14	8.98	9.08	8.40	8.82	0.367	ICP-OES
09	9.05	9.05	9.07	9.06	0.012	ICP-MS
11	7.940	8.836	10.89	9.22	1.512	ICP-MS
17	9.76	8.96	9.50	9.41	0.408	ICP-OES
07	9.81	9.69	9.10	9.53	0.380	ICP-MS
06	9.656	9.521	9.528	9.57	0.076	ICP-MS
16	9.43	9.72	9.68	9.61	0.157	ET-AAS
02	9.52	9.97	9.75	9.75	0.225	ICP-MS
10	10.4	9.75	9.91	10.02	0.339	ICP-OES
05	10.17	10.09	10.30	10.19	0.106	ICP-OES
15	10.39	10.43	10.13	10.32	0.163	ICP-OES
03	10.45	10.43	10.23	10.37	0.122	ICP-OES
01	10.6	10.7	10.9	10.73	0.153	ICP-OES
08	10.6	10.8	11.0	10.80	0.200	ICP-MS
12	10.8	11.0	10.6	10.80	0.200	ICP-MS
18	10.9	11.1	11.0	11.00	0.100	ICP-OES
19	11.2	11.0	11.2	11.13	0.115	ICP-OES
04	12.28	11.28	11.47	11.68	0.531	ICP-MS

M (mg/kg): **10.07**

SD_M (mg/kg): 0.710

$SD_M/\sqrt{N}$ (mg/kg): 0.178

Data sets of labs 04 and 11 were not taken into account (see Chapter 5.2 and Annex B).

Table A.7b: Measurement results of ILC-participants **Ni (B)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
13	8.22	7.44	6.66	7.44	0.780	ICP-OES
14	8.44	9.15	9.74	9.11	0.651	ICP-OES
09	10.69	10.37	10.05	10.37	0.320	ICP-MS
16	10.6	10.3	11.0	10.63	0.351	ET-AAS
10	10.5	10.6	11.1	10.73	0.321	ICP-OES
01	11.1	10.8	10.4	10.77	0.351	ICP-OES
12	9.55	11.5	11.6	10.88	1.156	ICP-MS
07	11.0	10.9	11.1	11.00	0.100	ICP-MS
06	11.14	11.36	10.68	11.06	0.347	ICP-MS
05	11.04	10.86	11.34	11.08	0.242	ICP-OES
03	10.3	12.0	11.4	11.23	0.862	ICP-OES
18	11.5	10.9	11.4	11.27	0.321	ICP-OES
02	11.1	11.8	11.3	11.40	0.361	ICP-MS
15	12.09	12.12	11.76	11.99	0.200	ICP-OES
08	12.4	12.9	11.4	12.23	0.764	ICP-MS
11	14.13	12.20	12.21	12.85	1.111	ICP-MS
19	13.5	12.4	12.9	12.93	0.551	ICP-OES
04	15.38	15.28	13.29	14.65	1.179	ICP-MS

M (mg/kg): **11.22**

SD_M (mg/kg): 0.945

$SD_M/\sqrt{N}$ (mg/kg): 0.236

Data sets of labs 04 and 13 were not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **Ni**

(graphical presentation) Aqua regia extractable mass fraction

Fig. A.7a: Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

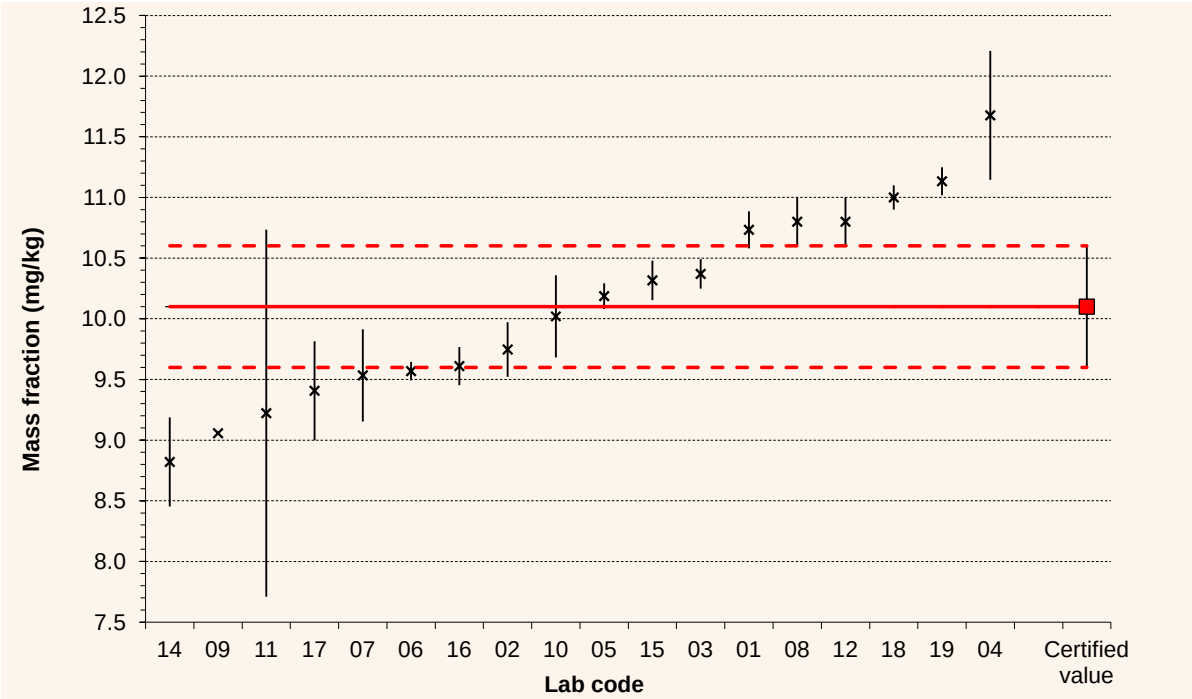


Fig. A.7b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

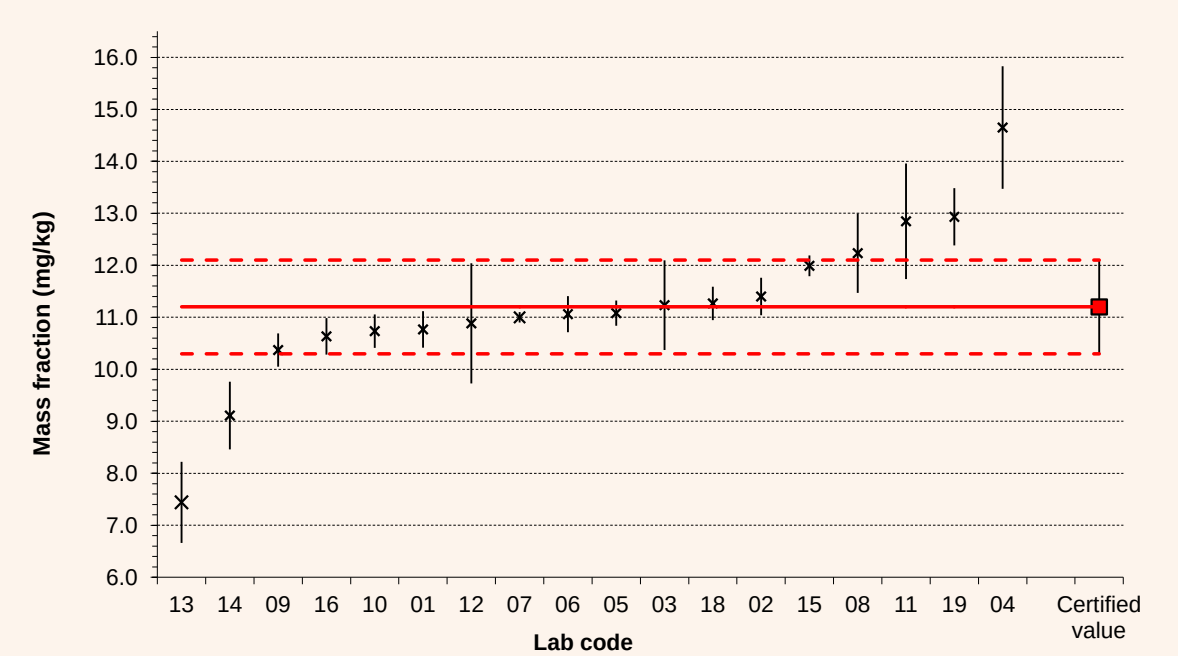


Table A.8a: Measurement results of ILC-participants **Pb (A)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
04	167.7	159.7	157.7	161.7	5.29	ICP-MS
14	188	176	185	183.0	6.24	ICP-OES
16	187	181	182	183.3	3.21	F-AAS
15	185.5	186.4	190.8	187.6	2.84	ICP-OES
12	196	193	192	193.7	2.08	ICP-MS
02	195	196	194	195.0	1.00	ICP-MS
09	203	192	191	195.3	6.66	ICP-OES
10	188	209	196	197.7	10.60	ICP-OES
18	197	201	196	198.0	2.65	ICP-OES
05	200.0	197.2	203.8	200.3	3.31	ICP-OES
19	200	201	200	200.3	0.58	ICP-OES
08	199	202	204	201.7	2.52	ICP-MS
01	205	200	201	202.0	2.65	ICP-OES
06	204.8	200.8	201.2	202.3	2.20	ICP-MS
17	202	205	202	203.0	1.73	ICP-OES
03	208.6	206.6	199.7	205.0	4.67	ICP-OES
07	219	206	194	206.3	12.50	ICP-MS
11	206.1	210.1	207.7	208.0	2.01	ICP-MS

M (mg/kg): **197.8**

SD_M (mg/kg): 7.43

$SD_M/\sqrt{N}$ (mg/kg): 1.80

Data set of lab 04 was not taken into account (see Chapter 5.2 and Annex B).

Table A.8b: Measurement results of ILC-participants **Pb (B)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
16	164	166	169	166.3	2.52	F-AAS
14	183	180	187	183.3	3.51	ICP-OES
12	196	187	181	188.0	7.55	ICP-MS
07	195	187	191	191.0	4.00	ICP-MS
02	193	195	191	193.0	2.00	ICP-MS
13	196.5	191.1	196.2	194.6	3.03	ICP-OES
18	194	197	202	197.7	4.04	ICP-OES
08	200	197	199	198.7	1.53	ICP-MS
03	201	196	200	199.0	2.65	ICP-OES
19	202	197	199	199.3	2.52	ICP-OES
15	198.6	203.1	197.8	199.8	2.86	ICP-OES
04	173.9	226.4	203.1	201.1	26.31	ICP-MS
09	204	203	198	201.7	3.21	ICP-OES
06	207.7	203.7	199.7	203.7	4.00	ICP-MS
05	198.4	209.0	203.8	203.7	5.30	ICP-OES
11	200.2	203.3	211.8	205.1	6.01	ICP-MS
01	205	210	211	208.7	3.21	ICP-OES
10	219	206	215	213.3	6.66	ICP-OES

M (mg/kg): **198.8**

SD_M (mg/kg): 7.64

$SD_M/\sqrt{N}$ (mg/kg): 1.91

Data sets of labs 04 and 16 were not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **Pb**

(graphical presentation) Aqua regia extractable mass fraction

Fig. A.8a: Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

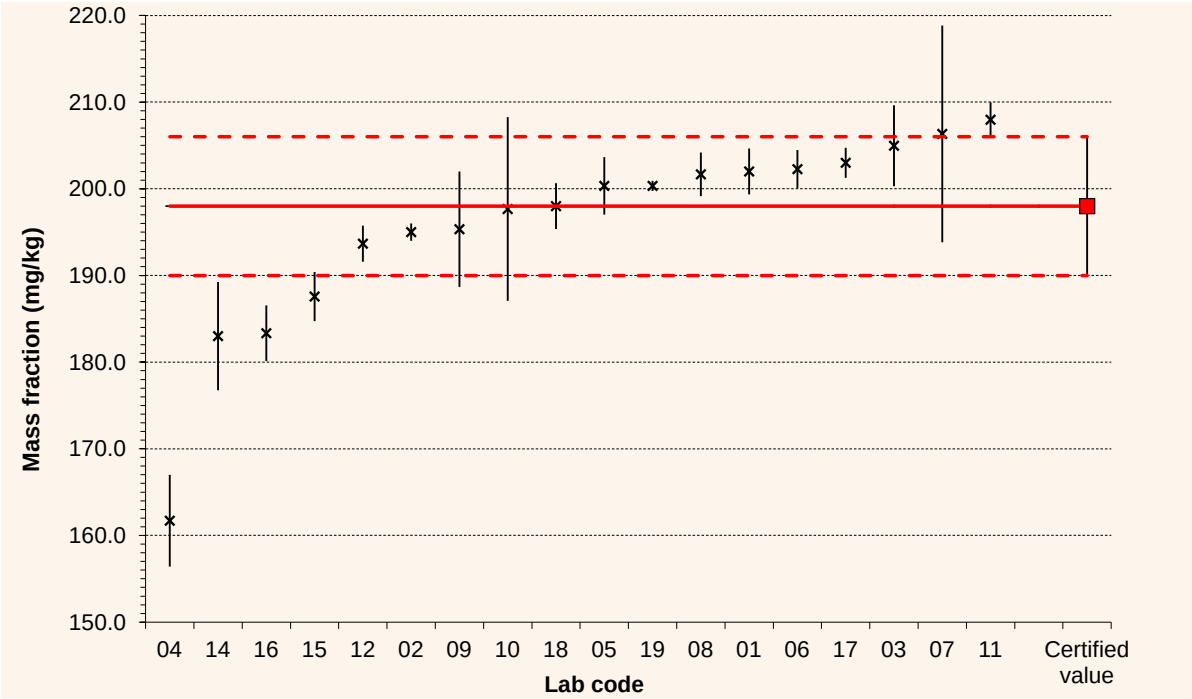


Fig. A.8b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

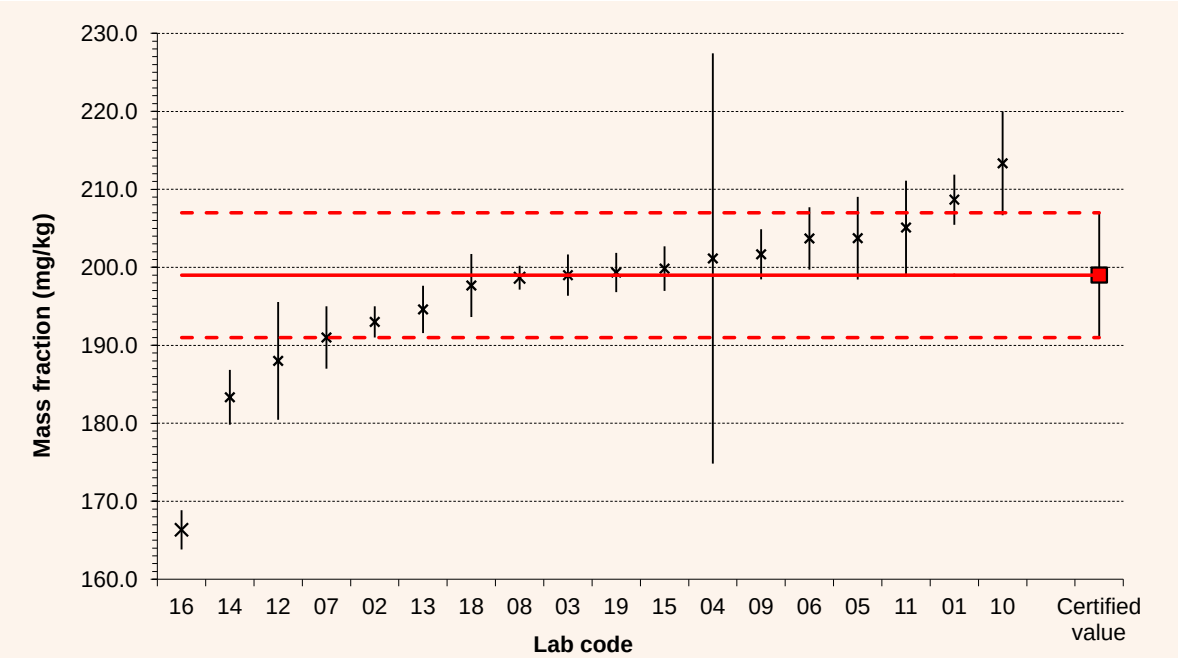


Table A.9a: Measurement results of ILC-participants **V (A)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
11	9.171	10.91	10.82	10.30	0.979	ICP-MS
09	10.67	10.49	10.73	10.63	0.125	ICP-MS
07	11.4	11.4	10.8	11.20	0.346	ICP-MS
16	12.3	11.7	11.7	11.90	0.346	ET-AAS
19	12.0	11.8	11.9	11.90	0.100	ICP-OES
03	12.33	12.13	12.54	12.33	0.205	ICP-OES
05	12.36	12.99	13.13	12.83	0.410	ICP-OES
15	13.01	12.86	12.82	12.90	0.100	ICP-OES
10	13.0	12.9	12.8	12.90	0.100	ICP-OES
08	13.0	13.0	13.1	13.03	0.058	ICP-MS
06	12.91	13.06	13.25	13.07	0.170	ICP-MS
18	12.9	13.4	13.4	13.23	0.289	ICP-OES
01	13.3	13.8	13.4	13.50	0.265	ICP-OES
12	14.1	14.1	13.3	13.83	0.462	ICP-MS
04	14.28	13.97	14.10	14.12	0.156	ICP-MS
02	14.3	14.0	14.1	14.13	0.153	ICP-MS
17	14.7	14.7	14.5	14.63	0.115	ICP-OES

M (mg/kg): **12.65**

SD_M (mg/kg): 1.211

$SD_M/\sqrt{N}$ (mg/kg): 0.303

Data set of lab 04 was not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **V (B)**

Aqua regia extractable mass fraction

Table A.9b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
13	8.95	9.93	9.02	9.30	0.547	ICP-OES
04	11.1	12.7	11.7	11.83	0.808	ICP-MS
16	12.1	11.9	11.6	11.87	0.252	ET-AAS
09	12.86	12.43	12.28	12.52	0.301	ICP-MS
03	12.5	13.5	11.6	12.53	0.950	ICP-OES
18	13.0	12.6	13.1	12.90	0.265	ICP-OES
12	12.1	14.0	13.3	13.13	0.961	ICP-MS
01	12.9	13.6	13.1	13.20	0.361	ICP-OES
07	14.1	13.7	13.9	13.90	0.200	ICP-MS
08	14.0	14.6	13.4	14.00	0.600	ICP-MS
10	14.7	14.1	14.8	14.53	0.379	ICP-OES
19	14.8	14.7	14.5	14.67	0.153	ICP-OES
15	14.34	15.86	14.90	15.03	0.769	ICP-OES
05	15.23	14.98	15.08	15.10	0.126	ICP-OES
02	14.8	15.7	15.0	15.17	0.473	ICP-MS
06	15.10	15.31	15.38	15.26	0.146	ICP-MS
11	15.44	15.41	15.83	15.56	0.234	ICP-MS

M (mg/kg): **13.96**

SD_M (mg/kg): 1.191

$SD_M/\sqrt{N}$ (mg/kg): 0.308

Data sets of labs 04 and 13 were not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **V**

(graphical presentation) Aqua regia extractable mass fraction

Fig. A.9a: Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Fig. A.9b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

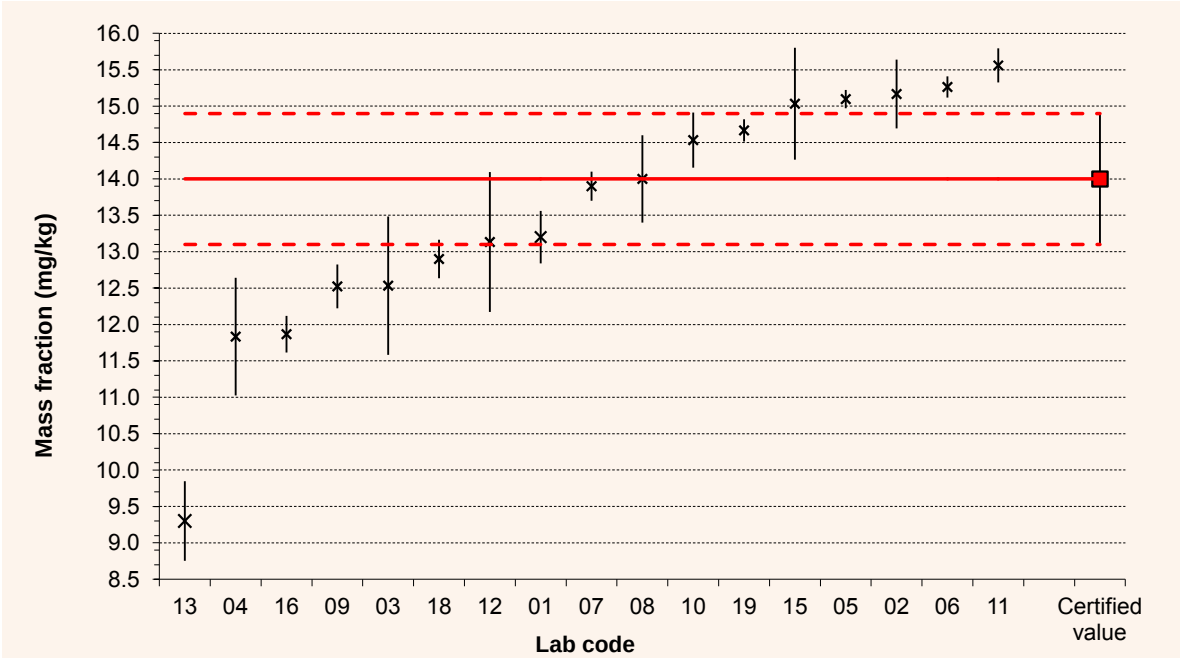


Table A.10a: Measurement results of ILC-participants **Zn (A)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
02	191	189	190	190.0	1.00	ICP-MS
07	193	194	183	190.0	6.08	ICP-MS
16	188	190	192	190.0	2.00	F-AAS
06	190.5	190.3	192.7	191.2	1.33	ICP-MS
10	196	186	194	192.0	5.29	ICP-OES
09	193	190	199	194.0	4.58	ICP-OES
14	191	194	198	194.3	3.51	ICP-OES
05	194.0	192.7	198.6	195.1	3.10	ICP-OES
11	200.2	196.0	195.6	197.3	2.55	ICP-MS
15	195.0	198.6	199.5	197.7	2.38	ICP-OES
03	200.6	199.0	199.9	199.8	0.80	ICP-OES
17	203	203	198	201.3	2.89	ICP-OES
08	200	201	204	201.7	2.08	ICP-MS
01	201	203	205	203.0	2.00	ICP-OES
18	204	207	203	204.7	2.08	ICP-OES
12	210	216	209	211.7	3.79	ICP-MS
19	215	213	214	214.0	1.00	ICP-OES
04	257.5	184.1	224.4	222.0	36.76	ICP-MS

M (mg/kg): **198.1**

SD_M (mg/kg): 7.30

$SD_M/\sqrt{N}$ (mg/kg): 1.77

Data set of lab 04 was not taken into account (see Chapter 5.2 and Annex B).

Table A.10b: Measurement results of ILC-participants **Zn (B)**

Aqua regia extractable mass fraction

Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

Lab Code	Sub-sample 1 (mg/kg)	Sub-sample 2 (mg/kg)	Sub-sample 3 (mg/kg)	Mean (mg/kg)	SD (mg/kg)	Analytical method
16	180	172	174	175.3	4.16	F-AAS
13	185.4	183.3	186.9	185.2	1.81	ICP-OES
07	191	187	193	190.3	3.06	ICP-MS
02	190	192	191	191.0	1.00	ICP-MS
06	192.4	204.1	191.8	196.1	6.93	ICP-MS
10	196	190	206	197.3	8.08	ICP-OES
14	190	198	205	197.7	7.51	ICP-OES
03	199.3	198.8	195.8	198.0	1.89	ICP-OES
12	195	197	202	198.0	3.61	ICP-MS
11	204.7	195.2	199.7	199.9	4.75	ICP-MS
05	199.6	196.3	204.1	200.0	3.92	ICP-OES
09	204	201	202	202.3	1.53	ICP-OES
15	207.6	199.9	202.4	203.3	3.93	ICP-OES
08	206	203	201	203.3	2.52	ICP-MS
18	206	207	209	207.3	1.53	ICP-OES
01	210	208	209	209.0	1.00	ICP-OES
19	215	213	215	214.3	1.15	ICP-OES
04	219.9	288.8	235.9	248.2	36.06	ICP-MS

M (mg/kg): **199.6**

SD_M (mg/kg): 7.28

$SD_M/\sqrt{N}$ (mg/kg): 1.77

Data sets of labs 04 and 16 were not taken into account (see Chapter 5.2 and Annex B).

Measurement results of ILC-participants **Zn**

(graphical presentation) Aqua regia extractable mass fraction

Fig. A.10a: Extraction method: **EN 16174, Method A** (open vessel, reflux conditions)

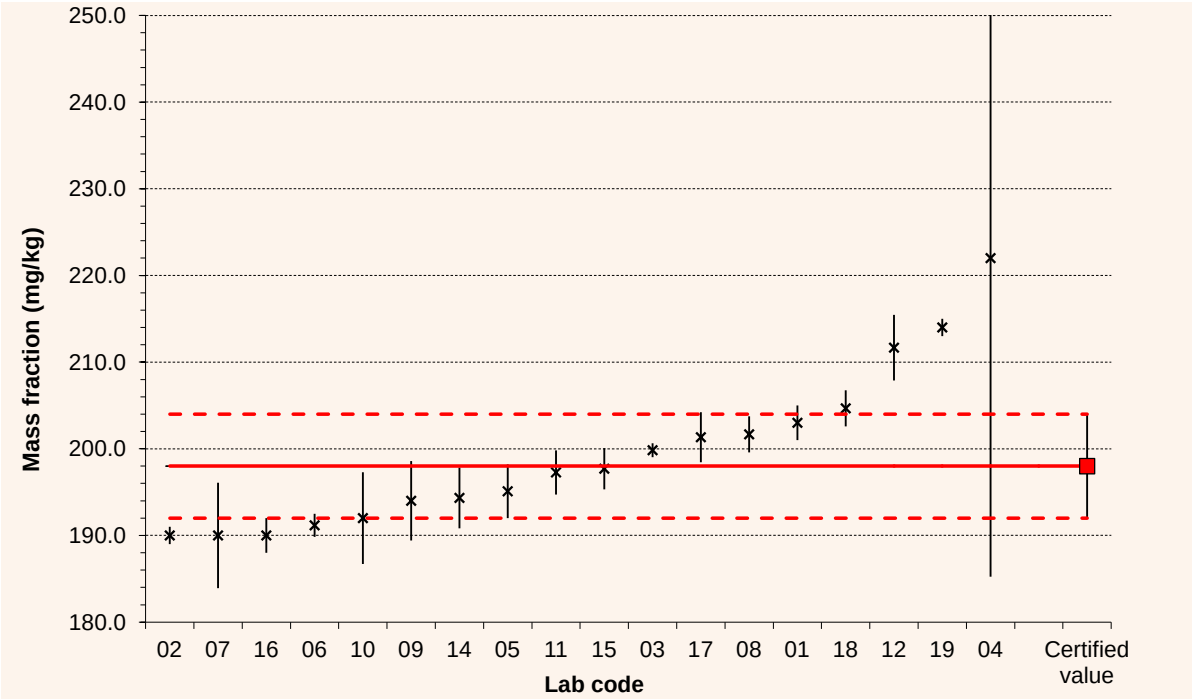


Fig. A.10b: Extraction method: **EN 16174, Method B** (microwave-assisted, 175 °C)

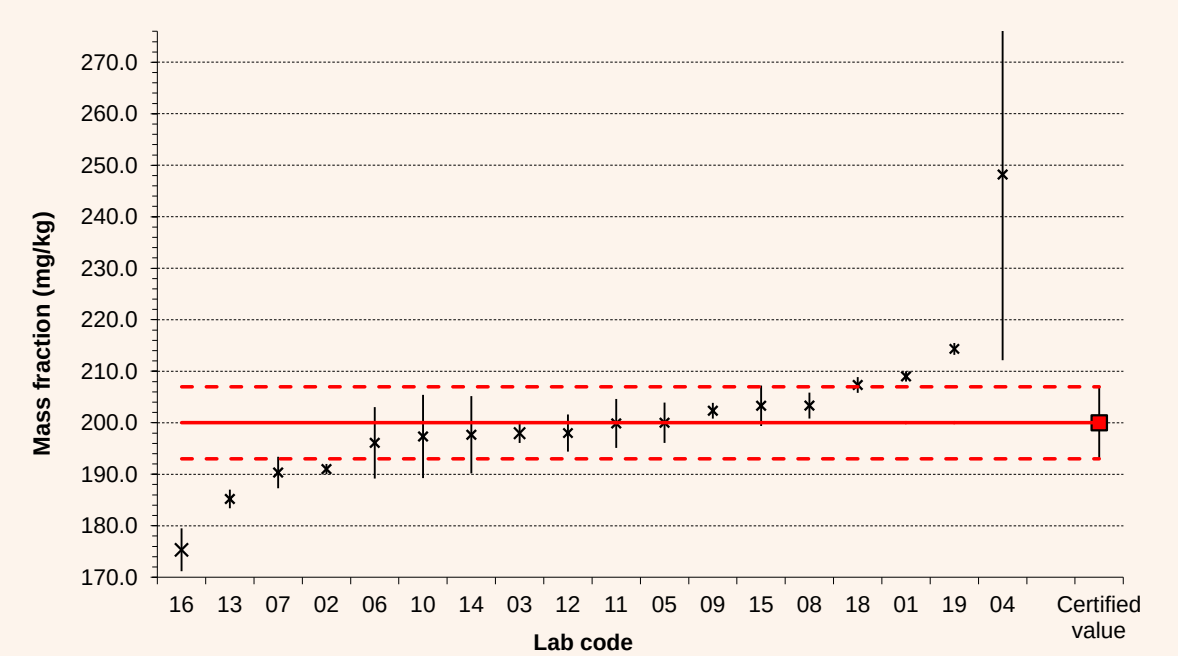


Table B.1a: Outcome of statistical tests on results obtained for **As** (**Extraction Method A**)

Number of evaluated data sets: 16	Outcome of the statistical test
Scheffé's multiple t-test	Data sets are not compatible two-by-two
Grubbs test (α = 0.05)	Lab 04 is an outlier
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 04 is an outlier
Nalimov test (α = 0.01)	Lab 04 is an outlier
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Not normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 04 was rejected as Nalimov-outlier.

Table B.1b: Outcome of statistical tests on results obtained for **As** (**Extraction Method A**) after removal of lab 04

Number of evaluated data sets: 15	Outcome of the statistical test
Scheffé's multiple t-test	Data sets are not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	No outlier detected
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.2a: Outcome of statistical tests on results obtained for **As** (**Extraction Method B**)

Number of evaluated data sets: 16	Outcome of the statistical test
Scheffé's multiple t-test	Data sets are not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	No outlier detected
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 10 and 15 are outliers
Cochran test (α = 0.01)	Lab 10 is an outlier
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 10 was rejected due to RSD > 10 %, data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.2b: Outcome of statistical tests on results obtained for **As** (**Extraction Method B**) after removal of labs 04 and 10

Number of evaluated data sets: 14	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	No outlier detected
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 13 and 15 are outliers
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.3a: Outcome of statistical tests on results obtained for **Cd** (**Extraction Method A**)

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	Lab 03 is an outlier
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	Lab 03 is an outlier
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 03 is an outlier
Nalimov test (α = 0.01)	Lab 03 is an outlier
Cochran test (α = 0.05)	Labs 04, 07, 09 and 10 are outliers
Cochran test (α = 0.01)	Labs 09 and 10 are outliers
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Not normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 03 was rejected as Nalimov-outlier, data set of lab 09 was rejected due to RSD > 10 %, data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.3b: Outcome of statistical tests on results obtained for **Cd** (**Extraction Method A**) after removal of labs 03, 04 and 09

Number of evaluated data sets: 14	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 12 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 07 and 10 are outliers
Cochran test (α = 0.01)	Lab 10 is an outlier
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 10 was not rejected due to RSD < 10 %.

Table B.4a: Outcome of statistical tests on results obtained for **Cd** (**Extraction Method B**)

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	Lab 16 is an outlier
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 16 is an outlier
Nalimov test (α = 0.01)	Lab 16 is an outlier
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 16 was rejected as Nalimov-outlier, data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.4b: Outcome of statistical tests on results obtained for **Cd** (**Extraction Method B**) after removal of labs 04 and 16

Number of evaluated data sets: 15	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	No outlier detected
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.5a: Outcome of statistical tests on results obtained for **Co** (**Extraction Method A**)

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Labs 09 and 11 are outliers
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 11 and 14 are outliers
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.5b: Outcome of statistical tests on results obtained for **Co** (**Extraction Method A**) after removal of lab 04

Number of evaluated data sets: 16	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Labs 09 and 11 are outliers
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 11 and 14 are outliers
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.6a: Outcome of statistical tests on results obtained for **Co** (**Extraction Method B**)

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 15 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Not normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.6b: Outcome of statistical tests on results obtained for **Co** (**Extraction Method B**) after removal of lab 04

Number of evaluated data sets: 16	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 15 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 12 and 14 are outliers
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Not normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.7a: Outcome of statistical tests on results obtained for **Cr** (**Extraction Method A**)

Number of evaluated data sets: 18	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 04 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 07 and 10 are outliers
Cochran test (α = 0.01)	Labs 07 and 10 are outliers
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.7b: Outcome of statistical tests of results obtained for **Cr** (**Extraction Method A**) after removal of lab 04.

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	No outlier detected
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 07 and 10 are outliers
Cochran test (α = 0.01)	Labs 07 and 10 are outliers
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data sets of labs 07 and 10 were not rejected due to RSD < 10 %.

Table B.8a: Outcome of statistical tests on results obtained for **Cr** (**Extraction Method B**)

Number of evaluated data sets: 18	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 13 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 04 and 12 are outliers
Cochran test (α = 0.01)	Lab 04 is an outlier
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.8b: Outcome of statistical tests of results obtained for **Cr** (**Extraction Method B**) after removal of lab 04

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 13 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Lab 12 is an outlier
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.9a: Outcome of statistical tests on results obtained for **Cu** (**Extraction Method A**)

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	No outlier detected
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 07 and 15 are outliers
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.9b: Outcome of statistical tests of results obtained for **Cu** (**Extraction Method A**) after removal of lab 04

Number of evaluated data sets: 16	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 19 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.10a: Outcome of statistical tests on results obtained for **Cu** (**Extraction Method B**)

Number of evaluated data sets: 18	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 04 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Lab 04 is an outlier
Cochran test (α = 0.01)	Lab 04 is an outlier
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.10b: Outcome of statistical tests of results obtained for **Cu** (**Extraction Method B**) after removal of lab 04

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	No outlier detected
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.11: Outcome of statistical tests on results obtained for **Hg** (**Extraction Method A**)

Number of evaluated data sets: 18	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 12a is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

No data sets rejected.

Table B.12a: Outcome of statistical tests on results obtained for **Hg** (**Extraction Method B**)

Number of evaluated data sets: 19	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 12a is an outlier
Nalimov test (α = 0.01)	Lab 12a is an outlier
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Not normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 12a was rejected as Nalimov-outlier, data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.12b: Outcome of statistical tests of results obtained for **Hg** (**Extraction Method B**) after removal of labs 04 and 12a

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 15 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.13a: Outcome of statistical tests on results obtained for **Ni** (**Extraction Method A**)

Number of evaluated data sets: 18	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 04 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Lab 11 is an outlier
Cochran test (α = 0.01)	Lab 11 is an outlier
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 11 was rejected due to RSD > 10 %, data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.13b: Outcome of statistical tests of results obtained for **Ni** (**Extraction Method A**) after removal of labs 04 and 11

Number of evaluated data sets: 16	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	No outlier detected
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.14a: Outcome of statistical tests on results obtained for **Ni** (**Extraction Method B**)

Number of evaluated data sets: 18	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	Lab 13 is an outlier
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Labs 04 and 13 are outliers
Nalimov test (α = 0.01)	Lab 13 is an outlier
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Not normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 13 was rejected as Nalimov-outlier, data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.14b: Outcome of statistical tests of results obtained for **Ni** (**Extraction Method B**) after removal of labs 04 and 13

Number of evaluated data sets: 16	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 14 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.15a: Outcome of statistical tests on results obtained for **Pb** (**Extraction Method A**)

Number of evaluated data sets: 18	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	Lab 04 is an outlier
Grubbs test (α = 0.01)	Lab 04 is an outlier
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 04 is an outlier
Nalimov test (α = 0.01)	Lab 04 is an outlier
Cochran test (α = 0.05)	Labs 07 and 10 are outliers
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Not normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Not normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Not normal distribution of data sets

Data set of lab 04 was rejected as Nalimov-outlier.

Table B.15b: Outcome of statistical tests of results obtained for **Pb** (**Extraction Method A**) after removal of lab 04

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Labs 14 and 16 are outliers
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Labs 07 and 10 are outliers
Cochran test (α = 0.01)	Labs 07 and 10 are outliers
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data sets of labs 07 and 10 were not rejected due to RSD < 10 %.

Table B.16a: Outcome of statistical tests on results obtained for **Pb** (**Extraction Method B**)

Number of evaluated data sets: 18	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	Lab 16 is an outlier
Grubbs test (α = 0.01)	Lab 16 is an outlier
Dixon test (α = 0.05)	Lab 16 is an outlier
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 16 is an outlier
Nalimov test (α = 0.01)	Lab 16 is an outlier
Cochran test (α = 0.05)	Lab 04 is an outlier
Cochran test (α = 0.01)	Lab 04 is an outlier
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Not normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 16 was rejected as Nalimov-outlier, data set of lab 04 was rejected due to RSD > 10 %.

Table B.16b: Outcome of statistical tests of results obtained for **Pb** (**Extraction Method B**) after removal of labs 04 and 16

Number of evaluated data sets: 16	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Labs 10 and 14 are outliers
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.17a: Outcome of statistical tests on results obtained for **V** (**Extraction Method A**)

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 11 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Lab 11 is an outlier
Cochran test (α = 0.01)	Lab 11 is an outlier
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.17b: Outcome of statistical tests of results obtained for **V** (**Extraction Method A**) after removal of lab 04

Number of evaluated data sets: 16	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 11 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	Lab 11 is an outlier
Cochran test (α = 0.01)	Lab 11 is an outlier
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 11 was not rejected due to RSD < 10 %.

Table B.18a: Outcome of statistical tests on results obtained for **V** (**Extraction Method B**)

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	Lab 13 is an outlier
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 13 is an outlier
Nalimov test (α = 0.01)	Lab 13 is an outlier
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 13 was rejected as Nalimov-outlier, data set of lab 04 was rejected due to an inadequate overall performance in the inter-laboratory comparison.

Table B.18b: Outcome of statistical tests of results obtained for **V** (**Extraction Method B**) after removal of labs 04 and 13

Number of evaluated data sets: 15	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	No outlier detected
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.19a: Outcome of statistical tests on results obtained for **Zn** (**Extraction Method A**)

Number of evaluated data sets: 18	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 04 is an outlier
Nalimov test (α = 0.01)	Lab 04 is an outlier
Cochran test (α = 0.05)	Lab 04 is an outlier
Cochran test (α = 0.01)	Lab 04 is an outlier
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Not normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 04 was rejected as Nalimov-outlier.

Table B.19b: Outcome of statistical tests of results obtained for **Zn** (**Extraction Method A**) after removal of lab 04

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 19 is an outlier
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table B.20a: Outcome of statistical tests on results obtained for **Zn** (**Extraction Method B**)

Number of evaluated data sets: 18	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	Lab 04 is an outlier
Grubbs test (α = 0.01)	Lab 04 is an outlier
Dixon test (α = 0.05)	Lab 04 is an outlier
Dixon test (α = 0.01)	Lab 04 is an outlier
Nalimov test (α = 0.05)	Lab 04 is an outlier
Nalimov test (α = 0.01)	Lab 04 is an outlier
Cochran test (α = 0.05)	Lab 04 is an outlier
Cochran test (α = 0.01)	Lab 04 is an outlier
Bartlett test (α = 0.05)	Variances not homogeneous
Bartlett test (α = 0.01)	Variances not homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Not normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Not normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Not normal distribution of data sets

Data set of lab 04 was rejected as Nalimov-outlier.

Table B.20b: Outcome of statistical tests of results obtained for **Zn** (**Extraction Method B**) after removal of lab 04

Number of evaluated data sets: 17	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	Lab 16 is an outlier
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Lab 16 is an outlier
Nalimov test (α = 0.01)	Lab 16 is an outlier
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Data set of lab 16 was rejected as Nalimov-outlier.

Table B.20c: Outcome of statistical tests of results obtained for **Zn** (**Extraction Method B**) after removal of labs 04 and 16

Number of evaluated data sets: 16	Outcome of the statistical test
Scheffé's multiple t-test	Data sets not compatible two-by-two
Grubbs test (α = 0.05)	No outlier detected
Grubbs test (α = 0.01)	No outlier detected
Dixon test (α = 0.05)	No outlier detected
Dixon test (α = 0.01)	No outlier detected
Nalimov test (α = 0.05)	Labs 13 and 19 are outliers
Nalimov test (α = 0.01)	No outlier detected
Cochran test (α = 0.05)	No outlier detected
Cochran test (α = 0.01)	No outlier detected
Bartlett test (α = 0.05)	Variances homogeneous
Bartlett test (α = 0.01)	Variances homogeneous
Snedecor F-test (α = 0.05)	Significant differences between data sets
Snedecor F-test (α = 0.01)	Significant differences between data sets
Summary of Bartlett test and Snedecor F-test	Pooling of individual data not allowed
Kolmogorov-Smirnov-Lilliefors test (α = 0.05)	Normal distribution of data sets
Kolmogorov-Smirnov-Lilliefors test (α = 0.01)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.05)	Normal distribution of data sets
Skewness & kurtosis test (α = 0.01)	Normal distribution of data sets

Table C.1: Homogeneity study; results for **As** (**Extraction Method B**)

Sample intake: approx. 500 mg Analytical method: ICP-OES

Sample I.D.	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
033	10.80	10.66	10.97	10.81	0.16
066	10.84	10.93	10.49	10.75	0.23
099	10.40	10.83	10.85	10.69	0.25
132	11.11	11.07	11.22	11.13	0.08
165	10.90	10.87	10.70	10.82	0.11
198	11.01	11.06	10.41	10.83	0.36
231	11.34	11.00	10.86	11.07	0.25
264	10.61	11.23	11.17	11.00	0.34
297	10.58	10.66	11.10	10.78	0.28
330	11.14	10.36	10.45	10.65	0.43
363	10.68	11.31	11.30	11.10	0.36
396	11.41	11.15	10.77	11.11	0.32

M (mg/kg): **10.90**

SD_M (mg/kg): 0.175

u_{bb} (% rel.): 0.81
(acc. to ISO Guide 35)

Table C.2: Homogeneity study; results for **Cd** (**Extraction Method B**)

Sample intake: approx. 500 mg Analytical method: ICP-MS

Sample I.D.	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
033	4.090	4.094	4.266	4.150	0.100
066	3.887	4.125	4.000	4.004	0.119
099	3.904	4.114	4.104	4.041	0.118
132	4.262	4.302	4.104	4.223	0.105
165	4.034	4.108	4.174	4.105	0.070
198	4.003	4.219	4.042	4.088	0.115
231	3.971	3.995	4.113	4.026	0.076
264	4.042	4.047	4.167	4.085	0.071
297	4.186	4.276	4.020	4.161	0.130
330	4.036	3.948	4.063	4.016	0.060
363	4.005	4.109	4.047	4.054	0.052
396	4.148	4.059	4.190	4.132	0.067

M (mg/kg): **4.090**

SD_M (mg/kg): 0.067

u_{bb} (% rel.): 0.95
(acc. to ISO Guide 35)

Table C.3: Homogeneity study; results for **Co** (**Extraction Method B**)

Sample intake: approx. 500 mg Analytical method: ICP-OES

Sample I.D.	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
033	4.961	4.968	4.981	4.970	0.010
066	4.922	4.955	4.950	4.942	0.018
099	4.997	5.040	4.837	4.958	0.107
132	4.942	4.953	5.026	4.974	0.046
165	4.797	5.046	5.223	5.022	0.214
198	4.735	4.973	4.866	4.858	0.119
231	4.793	5.159	4.983	4.978	0.183
264	4.739	4.735	5.085	4.853	0.201
297	4.755	4.765	4.844	4.788	0.049
330	4.773	4.954	4.862	4.863	0.091
363	4.891	5.034	5.360	5.095	0.240
396	5.105	5.052	5.026	5.061	0.040

M (mg/kg): **4.947**

SD_M (mg/kg): 0.091

u_{bb} (% rel.): 0.97
(acc. to ISO Guide 35)

Table C.4: Homogeneity study; results for **Cr** (**Extraction Method B**)

Sample intake: approx. 500 mg Analytical method: ICP-OES

Sample I.D.	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
033	79.90	84.70	81.64	82.08	2.43
066	81.34	83.45	83.64	82.81	1.28
099	80.20	85.06	83.79	83.02	2.52
132	85.77	83.47	85.35	84.86	1.22
165	81.33	82.89	82.82	82.35	0.88
198	79.95	82.22	84.13	82.10	2.09
231	80.70	85.69	82.08	82.82	2.58
264	82.42	81.81	85.48	83.24	1.97
297	81.67	85.23	82.08	82.99	1.95
330	82.75	81.95	81.50	82.07	0.63
363	82.63	86.47	83.48	84.19	2.02
396	86.46	83.59	86.28	85.44	1.61

M (mg/kg): **83.16**

SD_M (mg/kg): 1.113

u_{bb} (% rel.): 0.70
(acc. to ISO Guide 35)

Table C.5: Homogeneity study; results for **Cu** (**Extraction Method B**)

Sample intake: approx. 500 mg Analytical method: ICP-OES

Sample I.D.	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
033	70.51	74.25	70.87	71.88	2.06
066	73.55	73.73	72.99	73.42	0.39
099	70.06	73.11	73.91	72.36	2.03
132	74.06	71.44	75.78	73.76	2.19
165	70.73	73.75	70.74	71.74	1.74
198	70.91	72.04	76.36	73.10	2.88
231	69.16	73.36	76.73	73.08	3.79
264	72.47	73.33	72.86	72.89	0.43
297	77.52	70.29	72.14	73.32	3.76
330	70.24	73.67	75.31	73.07	2.59
363	72.53	74.21	77.90	74.88	2.75
396	75.59	76.76	73.31	75.22	1.75

M (mg/kg):	**73.23**
SD_M (mg/kg):	1.044
u_{bb} (% rel.): (acc. to ISO Guide 35)	1.03

Table C.6: Homogeneity study; results for **Hg** (**Extraction Method B**)

Sample intake: approx. 500 mg Analytical method: ICP-MS

Sample I.D.	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
033	16.64	15.03	15.09	15.59	0.91
066	14.71	15.77	15.09	15.19	0.54
099	14.51	16.25	16.17	15.64	0.98
132	16.09	16.65	15.03	15.92	0.82
165	15.06	15.87	14.52	15.15	0.68
198	16.73	15.03	15.29	15.68	0.92
231	16.74	14.87	16.18	15.93	0.96
264	14.73	17.04	15.07	15.61	1.25
297	14.97	14.42	14.83	14.74	0.29
330	15.73	15.11	16.10	15.65	0.50
363	14.89	15.85	16.71	15.82	0.91
396	15.42	14.55	15.27	15.08	0.47

M (mg/kg): **15.50**

SD_M (mg/kg): 0.373

u_{bb} (% rel.): 1.63
(acc. to ISO Guide 35)

Table C.7: Homogeneity study; results for **Ni** (**Extraction Method B**)

Sample intake: approx. 500 mg Analytical method: ICP-OES

Sample I.D.	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
033	10.39	10.43	11.22	10.68	0.47
066	10.78	11.42	11.32	11.17	0.34
099	10.97	12.01	11.14	11.37	0.56
132	10.96	10.87	11.64	11.16	0.42
165	10.63	11.12	10.72	10.82	0.26
198	10.41	11.13	11.02	10.85	0.39
231	11.26	11.48	10.90	11.21	0.29
264	11.12	11.37	10.63	11.04	0.38
297	10.74	11.43	11.57	11.25	0.44
330	10.66	10.81	11.46	10.98	0.42
363	10.94	10.69	11.26	10.96	0.29
396	11.10	11.12	11.84	11.35	0.42

M (mg/kg): **11.07**

SD_M (mg/kg): 0.218

u_{bb} (% rel.): 1.12
(acc. to ISO Guide 35)

Table C.8: Homogeneity study; results for **Pb** (**Extraction Method B**)

Sample intake: approx. 500 mg Analytical method: ICP-OES

Sample I.D.	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
033	212.3	208.0	201.8	207.4	5.28
066	204.9	208.5	204.8	206.1	2.11
099	195.7	205.2	205.8	202.2	5.67
132	211.3	210.7	210.8	210.9	0.32
165	203.7	206.6	211.1	207.1	3.73
198	198.4	211.7	202.2	204.1	6.85
231	205.9	205.0	203.4	204.8	1.27
264	208.1	205.4	205.5	206.3	1.53
297	206.2	202.7	196.4	201.8	4.97
330	200.4	203.6	210.9	205.0	5.38
363	205.4	214.3	215.4	211.7	5.48
396	210.9	212.0	208.3	210.4	1.90

M (mg/kg): **206.5**

SD_M (mg/kg): 3.23

u_{bb} (% rel.): 1.02
(acc. to ISO Guide 35)

Table C.9: Homogeneity study; results for **V** (**Extraction Method B**)

Sample intake: approx. 500 mg Analytical method: ICP-OES

Sample I.D.	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
033	15.12	15.57	15.22	15.30	0.24
066	14.88	15.52	14.91	15.10	0.36
099	15.13	15.89	15.45	15.49	0.38
132	15.25	15.33	15.04	15.21	0.15
165	15.53	15.59	14.76	15.29	0.46
198	14.99	15.31	15.35	15.22	0.20
231	14.79	15.71	15.38	15.29	0.47
264	15.27	15.53	15.34	15.38	0.13
297	14.84	15.21	15.69	15.25	0.43
330	14.72	15.41	15.59	15.24	0.46
363	15.32	15.72	15.60	15.55	0.21
396	16.08	15.14	15.88	15.70	0.50

M (mg/kg): **15.34**

SD_M (mg/kg): 0.168

u_{bb} (% rel.): 0.72
(acc. to ISO Guide 35)

Table C.10: Homogeneity study; results for **Zn** (**Extraction Method B**)

Sample intake: approx. 500 mg Analytical method: ICP-OES

Sample I.D.	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
033	200.3	200.6	203.8	201.6	1.94
066	197.7	206.7	203.5	202.6	4.56
099	196.8	208.7	205.2	203.6	6.12
132	205.4	216.5	205.0	209.0	6.53
165	201.0	206.3	202.6	203.3	2.72
198	199.1	211.6	201.1	203.9	6.71
231	203.1	214.5	209.0	208.9	5.70
264	203.8	202.6	203.8	203.4	0.69
297	197.9	206.9	201.1	202.0	4.56
330	205.7	200.9	201.0	202.5	2.74
363	200.9	208.2	201.8	203.6	3.98
396	203.6	211.8	209.8	208.4	4.28

M (mg/kg): **204.4**

SD_M (mg/kg): 2.71

u_{bb} (% rel.): 0.70
(acc. to ISO Guide 35)

Table D.1: Stability study; results for **As** (**Extraction Method A**)

Analytical method: ICP-OES

Storage time	**Storage temperature**	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**#4** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
2 months	-20 °C	10.00	10.55	10.09	10.29	10.23	0.24
	+20 °C	10.08	10.38	10.04	10.25	10.19	0.16
	+40 °C	10.45	10.32	10.02	10.09	10.22	0.20
	+60 °C	10.30	10.33	10.09	10.33	10.26	0.12
4 months	-20 °C	10.26	10.72	10.57	10.49	10.51	0.19
	+20 °C	10.62	10.74	10.27	10.52	10.54	0.20
	+40 °C	10.20	10.63	10.61	10.46	10.48	0.20
	+60 °C	10.79	10.38	10.86	10.84	10.72	0.23
6 months	-20 °C	10.54	10.23	10.14	10.66	10.39	0.25
	+20 °C	10.59	10.42	10.47	10.35	10.46	0.10
	+40 °C	10.56	10.63	10.32	10.62	10.53	0.15
	+60 °C	10.31	10.66	10.37	10.33	10.42	0.16

Table D.2: Stability study; results for **Cd** (**Extraction Method A**)

Analytical method: ICP-MS

Storage time	**Storage temperature**	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**#4** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
2 months	-20 °C	3.950	4.097	3.885	3.992	3.981	0.089
	+20 °C	3.972	3.942	4.103	3.825	3.961	0.114
	+40 °C	3.932	4.149	4.006	3.846	3.983	0.128
	+60 °C	4.046	3.907	4.095	3.928	3.994	0.091
4 months	-20 °C	3.829	3.827	4.027	3.877	3.890	0.094
	+20 °C	3.950	3.868	3.840	3.833	3.873	0.054
	+40 °C	3.908	4.108	3.830	3.936	3.946	0.117
	+60 °C	3.918	3.884	3.954	3.971	3.932	0.039
6 months	-20 °C	3.950	4.028	3.901	4.014	3.973	0.059
	+20 °C	3.888	3.872	4.064	3.996	3.955	0.091
	+40 °C	3.892	3.839	3.845	3.977	3.888	0.064
	+60 °C	3.812	3.991	3.963	3.879	3.911	0.082

Table D.3: Stability study; results for **Co** (**Extraction Method A**)

Analytical method: ICP-OES

Storage time	**Storage temperature**	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**#4** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
2 months	-20 °C	3.825	3.795	3.766	3.752	3.785	0.032
	+20 °C	3.807	3.805	3.692	3.768	3.768	0.054
	+40 °C	3.769	3.851	3.738	3.752	3.778	0.051
	+60 °C	3.718	3.740	3.745	3.909	3.778	0.088
4 months	-20 °C	3.862	3.999	3.980	4.017	3.965	0.070
	+20 °C	3.957	4.038	3.881	3.931	3.952	0.066
	+40 °C	3.975	3.971	4.029	3.905	3.970	0.051
	+60 °C	3.958	3.841	3.984	3.852	3.909	0.073
6 months	-20 °C	3.889	3.603	3.902	4.037	3.858	0.183
	+20 °C	3.764	3.874	3.943	3.940	3.880	0.084
	+40 °C	3.980	3.993	3.982	3.703	3.915	0.141
	+60 °C	3.922	3.884	3.783	3.822	3.853	0.062

Table D.4: Stability study; results for **Cr** (**Extraction Method A**)

Analytical method: ICP-OES

Storage time	**Storage temperature**	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**#4** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
2 months	-20 °C	77.36	81.00	77.07	77.40	78.21	1.87
	+20 °C	77.83	79.51	77.86	77.53	78.18	0.90
	+40 °C	81.11	80.30	77.52	78.18	79.28	1.70
	+60 °C	78.68	78.97	77.85	81.22	79.18	1.44
4 months	-20 °C	77.49	78.34	78.96	79.37	78.54	0.82
	+20 °C	78.99	78.93	76.97	78.25	78.29	0.94
	+40 °C	78.58	80.88	78.57	78.89	79.23	1.11
	+60 °C	78.29	79.13	80.02	80.04	79.37	0.84
6 months	-20 °C	80.17	79.08	79.71	81.05	80.00	0.83
	+20 °C	79.46	78.67	79.81	79.63	79.39	0.50
	+40 °C	80.46	79.79	79.99	79.46	79.93	0.42
	+60 °C	80.01	81.00	79.89	79.02	79.98	0.81

Table D.5: Stability study; results for **Cu** (**Extraction Method A**)

Analytical method: ICP-OES

Storage time	**Storage temperature**	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**#4** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
2 months	-20 °C	71.75	75.97	72.69	72.72	73.28	1.85
	+20 °C	73.73	76.27	71.62	74.15	73.94	1.91
	+40 °C	75.70	76.47	74.23	73.33	74.93	1.42
	+60 °C	73.45	73.06	72.01	76.46	73.75	1.91
4 months	-20 °C	73.44	70.64	74.96	74.07	73.28	1.87
	+20 °C	74.57	73.31	72.21	71.81	72.98	1.24
	+40 °C	72.41	75.11	73.24	73.57	73.58	1.13
	+60 °C	74.44	73.18	75.84	73.01	74.12	1.31
6 months	-20 °C	73.25	75.54	74.24	73.79	74.21	0.98
	+20 °C	73.76	73.64	75.54	74.33	74.32	0.87
	+40 °C	73.62	73.64	73.01	74.28	73.64	0.52
	+60 °C	73.27	74.91	74.23	72.27	73.67	1.15

Table D.6: Stability study; results for **Hg** (**Extraction Method A**)

Analytical method: ICP-MS

Storage time	**Storage temperature**	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**#4** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
2 months	-20 °C	16.22	16.24	15.40	15.95	15.95	0.39
	+20 °C	16.30	15.75	16.03	15.19	15.82	0.47
	+40 °C	15.57	16.29	16.39	17.13	16.35	0.64
	+60 °C	16.16	15.65	15.65	16.21	15.92	0.31
4 months	-20 °C	15.21	15.54	15.07	16.56	15.60	0.67
	+20 °C	16.51	15.52	15.46	16.38	15.97	0.55
	+40 °C	15.07	16.37	16.47	15.74	15.91	0.65
	+60 °C	15.05	16.54	16.72	16.16	16.12	0.75
6 months	-20 °C	15.33	15.07	16.16	15.77	15.58	0.48
	+20 °C	15.59	15.34	16.22	15.33	15.62	0.42
	+40 °C	15.76	16.32	15.46	15.31	15.71	0.45
	+60 °C	14.94	15.36	14.91	15.81	15.26	0.42

Table D.7: Stability study; results for **Ni** (**Extraction Method A**)

Analytical method: ICP-OES

Storage time	**Storage temperature**	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**#4** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
2 months	-20 °C	10.64	10.44	9.846	10.33	10.31	0.34
	+20 °C	9.855	10.09	10.01	10.50	10.11	0.28
	+40 °C	9.899	10.39	10.07	10.24	10.15	0.21
	+60 °C	10.35	10.10	10.11	10.53	10.27	0.21
4 months	-20 °C	9.945	10.13	10.49	9.971	10.13	0.25
	+20 °C	10.06	10.29	10.42	10.15	10.23	0.16
	+40 °C	10.42	10.42	10.28	10.20	10.33	0.11
	+60 °C	10.40	10.27	10.22	10.34	10.31	0.08
6 months	-20 °C	10.39	10.15	10.56	10.38	10.37	0.17
	+20 °C	10.51	10.24	10.32	10.16	10.31	0.15
	+40 °C	10.19	10.17	10.53	10.41	10.33	0.17
	+60 °C	10.40	10.43	10.49	10.07	10.35	0.19

Table D.8: Stability study; results for **Pb** (**Extraction Method A**)

Analytical method: ICP-OES

Storage time	**Storage temperature**	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**#4** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
2 months	-20 °C	201.4	214.9	199.1	200.9	204.1	7.3
	+20 °C	203.6	206.1	203.3	200.5	203.4	2.3
	+40 °C	208.5	203.4	203.2	204.2	204.8	2.5
	+60 °C	204.2	201.1	204.1	208.6	204.5	3.1
4 months	-20 °C	199.3	198.7	203.8	200.8	200.7	2.3
	+20 °C	206.6	203.5	198.6	202.7	202.9	3.3
	+40 °C	197.1	210.3	196.0	202.4	201.5	6.5
	+60 °C	200.4	200.9	200.2	207.2	202.2	3.4
6 months	-20 °C	202.1	193.8	201.8	201.8	199.9	4.1
	+20 °C	196.9	200.5	199.7	199.6	199.2	1.6
	+40 °C	201.5	199.0	197.8	195.9	198.6	2.3
	+60 °C	199.5	200.5	198.9	198.3	199.3	0.9

Table D.9: Stability study; results for **V** (**Extraction Method A**)

Analytical method: ICP-OES

Storage time	**Storage temperature**	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**#4** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
2 months	-20 °C	12.82	13.10	12.69	12.90	12.88	0.17
	+20 °C	13.06	13.09	12.95	12.73	12.96	0.16
	+40 °C	13.04	13.09	12.58	12.69	12.85	0.25
	+60 °C	13.20	13.03	12.72	13.00	12.99	0.20
4 months	-20 °C	12.71	12.78	12.56	12.82	12.72	0.11
	+20 °C	12.72	12.75	12.75	12.89	12.78	0.08
	+40 °C	12.82	13.23	12.90	12.75	12.93	0.21
	+60 °C	12.58	12.66	12.86	12.73	12.71	0.12
6 months	-20 °C	12.92	13.03	12.87	13.00	12.96	0.07
	+20 °C	12.99	12.95	13.19	13.30	13.11	0.17
	+40 °C	12.68	12.84	12.93	12.94	12.85	0.12
	+60 °C	13.00	13.14	12.97	12.83	12.99	0.13

Table D.10: Stability study; results for **Zn** (**Extraction Method A**)

Analytical method: ICP-OES

Storage time	**Storage temperature**	**#1** (mg/kg)	**#2** (mg/kg)	**#3** (mg/kg)	**#4** (mg/kg)	**Mean** (mg/kg)	**SD** (mg/kg)
2 months	-20 °C	194.5	205.5	196.9	195.5	198.1	5.0
	+20 °C	197.9	200.3	198.0	195.5	197.9	2.0
	+40 °C	202.3	202.9	196.8	197.1	199.8	3.3
	+60 °C	198.3	199.4	197.1	201.6	199.1	1.9
4 months	-20 °C	197.1	197.0	198.8	198.5	197.9	0.9
	+20 °C	197.4	197.6	195.3	198.6	197.2	1.4
	+40 °C	195.0	205.8	198.2	196.9	199.0	4.7
	+60 °C	197.0	198.5	199.9	196.7	198.0	1.5
6 months	-20 °C	202.4	196.5	201.3	203.7	201.0	3.1
	+20 °C	199.9	201.2	198.5	198.6	199.6	1.3
	+40 °C	193.9	195.1	197.9	203.9	197.7	4.5
	+60 °C	199.7	199.7	200.6	198.7	199.7	0.8

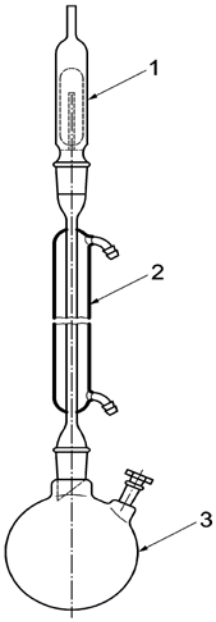
Extraction according to EN 16174, Method A:
(*procedure almost identical to ISO 11466*)

Weigh approximately 3 g, to the nearest 0,01 g, of the soil sample and transfer it to the 250 mL reaction vessel of the apparatus shown in the figure below. Similar equipment can also be used.

Add some acid-washed glass beads (diameter ~ 3 mm) as boiling aids.
In case of dry samples moisten the test portion with about 0.5 mL of water and add, with mixing, 21 mL of concentrated hydrochloric acid followed by 7 mL of concentrated nitric acid.
Connect the reflux condenser to the reaction vessel. Fill the absorption vessel with approx. 15 mL of 0.5 mol/L nitric acid.
Connect the absorption vessel to the reflux condenser, and let stand at room temperature until any effervescence almost ceases to allow for slow oxidation of the organic matter in the sample.

NOTE: The time of standing at room temperature can influence the extraction efficiency of aqua regia. For comparison reasons of the method it is recommended to start heating as soon as possible after the first strong reaction has ceased.

Connect the reaction vessel to the heating device and raise the temperature of the reaction mixture to reflux conditions and maintain for 2 h ensuring that the condensation zone is lower than 1/3 of the height of the reflux condenser, then allow to cool. Add the content of the absorption vessel to the reaction vessel via the reflux condenser, rinsing both the absorption vessel and condenser with further 10 mL of 0.5 mol/L nitric acid.



1 Absorption vessel
2 Reflux condenser
3 Reaction vessel

Add about 20 mL of water, filter the sample through an acid-resistant membrane filter (preferably with a pore size of 0.45 µm) into a 100 mL volumetric flask, wash the filter residue with 0.5 mol/L nitric acid and fill up to volume with water.
The measurement solution is now ready for analysis for elements of interest using appropriate elemental analysis techniques.

Extraction according to EN 16174, Method B:
(*microwave-assisted extraction*)

Operate the microwave equipment according to the instructions of the manufacturer!
Weigh an amount of 0.5 g to 1 g of the soil sample with an accuracy of 0,001 g and transfer it into the extraction vessel. Referring to the manufacturer's instructions, the upper limits of mass of the test portion shall be taken into account.

Moisten the sample with a few drops of water. Add separately 6 mL of concentrated hydrochloric acid and 2 mL of concentrated nitric acid and mix well. If a vigorous reaction occurs, allow the reaction cease before capping the vessel.

Cap the extraction vessel according to the manufacturer's instructions. Weigh the extraction vessel before starting the heating program. The carrousel of the microwave system must be filled with the number of extraction vessels prescribed by the manufacturer. If a lower number of samples should be processed, place extraction vessels filled with the same amount of aqua regia without sample.

The temperature of the extraction mixture in each vessel shall be raised with a heating rate of approx. 10 °C/min to 15 °C/min to (175 ± 5) °C and remain at this temperature for (10 ± 1) min.
At the end of the microwave heating program, allow the vessels to cool before removing them from the microwave system.
Cooling of the vessels may be accelerated by internal or external cooling devices.

After reaching room temperature, check if the microwave vessels maintained their seal throughout the extraction procedure. Weigh the vessels to evaluate seal integrity. If the weight loss of the sample exceeds 10 % of the weight of the added test portion and reagents before the start of the extraction procedure, the respective sample is considered compromised.

Carefully uncap and vent each vessel in a well-ventilated fume hood. Add about 20 mL of water, filter the sample through an acid-resistant membrane filter (preferably with a pore size of 0.45 µm) into a volumetric flask (typically 50 mL or 100 mL), wash the residue with 0.5 mol/L nitric acid and fill up to volume with water.
The measurement solution is now ready for analysis for elements of interest using appropriate elemental analysis techniques.