Evaluation Report

proficiency test

DLA 55/2019

Contact Material I:

Release of Elements from Food Contact Material

DLA - Proficiency Tests GmbH
Kalte Weide 21
24641 Sievershütten/Germany

proficiency-testing@dla-lvu.de *www.dla-lvu.de*

Coordinator of this PT:
Matthias Besler-Scharf, PhD.

1st Correction 10/02/2020:
In the overview table of z-scores (p. 52) was a mistake:
The z-scores for the evaluation numbers 9, 10 and 11 for aluminium (1st eluate) were wrong. This has been corrected.
The z-scores in the result section were not affected (p. 18).

Allgemeine Informationen zur Eignungsprüfung (EP)
General Information on the proficiency test (PT)

EP-Anbieter *PT-Provider*	**DLA - Proficiency Tests GmbH** Kalte Weide 21, 24641 Sievershütten, Germany Geschäftsführer/CEO: Dr. Matthias Besler-Scharf Stellv. Leitung/Deputy Lead: Alexandra Scharf MSc. Tel. ++49-(0)4532-9183358 Mob. ++49(0)171-1954375 Fax. ++49(0)4102-9944976 eMail. proficiency-testing@dla-lvu.de
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EP-Koordinator *PT-Coordinator*	Dr. Matthias Besler-Scharf
Status des EP-Bericht *Status of PT-Report*	Abschlussbericht / Final report (18 February 2020) 1. Korrektur / 1st Correction Gültig ist die jeweils letzte Version/Korrektur des Berichts. Sie ersetzt alle vorangegangenen Versionen. Only the latest version/correction of the report is valid. It replaces all preceding versions.
EP-Bericht Freigabe *PT-Report Authorization*	Dr. Matthias Besler-Scharf (Technischer Leiter / Technical Manager) *- gezeichnet / signed M. Besler-Scharf* Alexandra Scharf MSc. (QM-Beauftragte / Quality Manager) *- gezeichnet / signed A. Scharf* Datum / Date: 10 February 2020
Unteraufträge *Subcontractors*	Im Rahmen dieser Eignungsprüfung wurden nachstehende Leistungen im Unterauftrag vergeben: Homogenitätsprüfung der EP-Parameter As part of the present proficency test the following services were subcontracted: Homogeneity tests of PT-parameter(s)
Vertraulichkeit *Confidentiality*	Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben. Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.

Contents

1. Introduction

The participation in proficiency testing schemes is an essential element of the quality-management-system of every laboratory testing food and feed, cosmetics and food contact materials. The implementation of proficiency tests enables the participating laboratories to prove their own analytical competence under realistic conditions. At the same time they receive valuable data regarding the verification and/or validation of the particular testing method [1, 5].
The purpose of DLA is to offer proficiency tests for selected parameters in concentrations with practical relevance.
Realisation and evaluation of the present proficiency test follows the technical requirements of DIN EN ISO/IEC 17043 (2010) and DIN ISO 13528:2009 / ISO 13528:2015 [2, 3].

2. Realisation

2.1 Test material

The test material is a fillable food contact material made of metal: Stainless steel cup (0.08 liter). The material was purchased in the trade by DLA from one production unit. The scope of determination based on preliminary investigations in accordance with the Resolution of the Council of Europe on metals and alloys in food contact materials and articles (CM/Res(2013)9, (18)) was narrowed on 6 elements (Al, Cr, Fe, Mn, Ni, Pb), with determinable levels in the migrate solution.

Three samples each were packed into LDPE plastic bags and then labelled.

2.1.1 Homogeneity

The suitability of the test material was checked by 5-fold determination of chromium and nickel in the respective first eluate by ICP-MS. The received repeatability standard deviations of 11 % for chromium and 8,7 % for nickel were in the range of the target standard deviations according to Horwitz. Therefore the homogeneity of the test material was considered acceptable. The results are given in the documentation.

The calculation of the **repeatability standard deviation S_r of the triple determination of the participants** was used as an indicator of homogeneity for this PT. The repeatability standard deviations were 22,6% for aluminium, 15,9% for chromium, 10,2% for iron and 18,6% for nickel and were in the range of the requirements of the Council of Europe Resolution. The repeatability standard deviations of manganese with 24,3% and lead with 41,8% were little or clearly higher.
According to the Council of Europe Resolution the predicted within-laboratory standard deviation based on the Horwitz Equation is for concentrations of 10 mg/L 11,3 %, of 1 mg/L 16,0 % and of 100 µg/L 22,6 % [18].
The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1-4.6.).

In case the criterion for sufficient homogeneity of the test items is not fulfilled the impact on the target standard deviation will be verified. If necessary the evaluation of results will be done considering the standard uncertainty of the assigned value by z'-scores (s. 3.8 and 3.11) [3].

2.1.2 Stability

The test items made of metal (stainless steel) are known to be usually resistant to corrosion at room temperature and dry storage. The stability of the material can be considered given for the examination period of the PT.

2.2 Sample shipment and information to the test

Three samples (A, B, and C) of test material were sent to every participating laboratory in the 40th week of 2019. The testing method was optional. The tests should be finished at 15th November 2019 the latest.

With the cover letter along with the sample shipment the following information was given to participants:

The material is a fillable stainless steel jug (0.08 liters). Three samples are provided. The specific migration of the elements Aluminium, Lead, Chromium, Iron, Manganese and Nickel in part with low contents should be determined.

The analysis conditions according to the Resolution of the Council of Europe on metals and alloys used in food contact materials (CM/Res(2013)9) for "hot fills" should be followed.

The exact conditions are given on the attached information on the proficiency test (see page 2).
(see documentation, section 5.3 Information on the PT)

2.3 Submission of results

The participants submitted their results in standard forms, which have been handed out with the samples (by email).

The finally calculated concentrations of the parameters as average of the determinations of the three samples were used for the statistical evaluation. For the calculation of the repeatability- and reproducibility standard deviation the single values of the triple determination were used.

Queried and documented were single results, recovery and the used testing methods. In case participants submitted several results for the same parameter obtained by different methods these results were evaluated with the same evaluation number with a letter as a suffix and indication of the related method.

All 11 participants submitted their results.

3. Evaluation

3.1 Consensus value from participants (assigned value)

The robust mean of the submitted results was used as assigned value (X_{pt}) („consensus value from participants") providing a normal distribution. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3]. If there are < 12 quantitative results and an increased difference between robust mean and median, the median may be used as the assigned value (criterion: Δ median - rob. mean > 0,3 σ_{pt}) [3].

The condition is that the majority of the participants' results show a normal distribution or are distributed unimodal and symmetrically. To this end, an examination of the distribution is carried out, inter alia, using the kernel density estimate [3, 12].

In case there are indications for sources of higher variability such as a bimodal distribution of results, a cause analysis is performed. Frequently different analytical methods may cause an anomaly in results' distribution. If this is the case, separate evaluations with own assigned values (X_{pti}) are made whenever possible.

The statistical evaluation is carried out for all the parameters for a minimum of 7 values are present, in justified cases, an evaluation may also be carried out from 5 results onwards.

The actual measurement results will be drafted. Individual results, which are outside the specified measurement range of the participating laboratory (for example with the result > 25 mg/kg or < 2,5 mg/kg) or the indicating "0" will not be considered for the statistic evaluation [3].

3.2 Robust standard deviation

For comparison to the target standard deviation σ_{pt} (standard deviation for proficiency assessment) a robust standard deviation (S*) was calculated. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3].

3.3 Repeatability standard deviation

The repeatability standard deviation S_r is based on the laboratory´s standard deviation of (outlier free) individual participant results, each under repeatability conditions, that means analyses was performed on the same sample by the same operator using the same equipment in the same laboratory within a short time. It characterizes the mean deviation of the results within the laboratories [3] and is used by DLA as an indication of the homogeneity of the sample material.

In case single results from participants are available the calculation of the repeatability standard deviation S_r, also known as standard deviation within laboratories S_w, is performed by: [3, 4].

The relative repeatability standard deviation as a percentage of the mean value is indicated as coefficient of variation CV_r in the table of statistical characteristics in the results section in case single results from participants are available.

3.4 Reproducibility standard deviation

The reproducibility standard deviation S_R represents a inter-laboratory estimate of the standard deviation for the determination of each parameter on the bases of (outlier free) individual participant results. It takes into account both the repeatability standard deviation S_r and the within-laboratory standard deviation S_S. Reproducibility standard deviations of PT´s may differ from reproducibility standard deviations of ring trials, because the participating laboratories of a PT generally use different internal conditions and methods for determining the measured values.
In the present evaluation, the specification of the reproducibility standard deviation, therefore, does not refer to a specific method, but characterizes approximately the comparability of results between the laboratories, assumed the effect of homogeneity and stability of the sample are negligible.

In case single results from participants are available the calculation of the reproducibility standard deviation S_R is performed by: [3, 4].

The relative reproducibility standard deviation CV_R in percent of the mean is given as variation coefficient in the statistical data of participant for each parameter. The significance of CV_R is further explained in section 3.9.

3.5 Exclusion of results and outliers

Before statistical evaluation obvious blunders, such as those with incorrect units, decimal point errors, too few significant digits (valid digits) or results for another proficiency test item can be removed from the data set [2]. Even if a result e.g. with a factor >10 deviates significantly from the mean and has an influence on the robust statistics, a result of the statistical evaluation can be excluded [3].

All results should be given at least with 2 significant digits. Specifying 3 significant digits is usually sufficient.

Results obtained by different analytical methods causing an increased variability and/or a bi- or multimodal distribution of results, are treated separately or could be excluded in case of too few numbers of results. For this results are checked by kernel density estimation [3, 12].

Results are tested for outliers by the use of robust statistics (algorithm A): If a value deviates from the robust mean by more than 3 times the robust standard deviation, it can be classified as an outlier (see above) [3]. Due to the use of robust statistics outliers are not excluded, provided that no other reasons are present [3]. Detected outliers are only mentioned in the results section, if they have been excluded from the statistical evaluation.

3.6 Target standard deviation (for proficiency assessment)

The target standard deviation of the assigned value σ_{pt} (= standard deviation for proficiency assessment) can be determined according to the following methods.

If an acceptable quotient S^*/σ_{pt} is present, the target standard deviation of the general model by Horwitz is preferably used for the proficiency assessment. It is usually suitable for evaluation of interlaboratory studies, where different methods are applied by the participants. On the other hand the target standard deviation from the evaluation of precision data of an precision experiment is derived from collaborative studies with specified analytical methods.

In cases where both above-mentioned models are not suitable, the target standard deviation is determined based on values by perception, see under 3.6.3.

For information, the z-scores of both models are given in the evaluation, if available.

Depending on the concentration range in the present PT for valuation of chromium, iron and nickel the target standard deviation according to the general model of Horwitz was applied and for aluminium, manganese and lead the target standard deviation according to the general model of Horwitz/Thompson was used (see 3.6.1).

Additionally for aluminium and lead the standard uncertainty was considered and the results were evaluated using z'-scores (s. 3.8).

3.6.1 General model (Horwitz)

Based on statistical characteristics obtained in numerous PTs for different parameters and methods Horwitz has derived a general model for estimating the reproducibility standard deviation σ_R [6]. Later the model was modified by Thompson for certain concentration ranges [10]. The reproducibility standard deviation σ_R can be applied as the relative target standard deviation σ_{pt} in % of the assigned values and calculated according to the following equations [3]. For this the assigned value X_{pt} is used for the concentration c.

Equations	**Range of concentrations**	**corresponds to**
$\sigma_R = 0{,}22c$	$c < 1{,}2 \times 10^{-7}$	< 120 µg/kg
$\sigma_R = 0{,}02c^{0{,}8495}$	$1{,}2 \times 10^{-7} \leq c \leq 0{,}138$	≥ 120 µg/kg
$\sigma_R = 0{,}01c^{0{,}5}$	$c > 0{,}138$	> 13,8 g/100g

with c = mass content of analyte (as relative size, e.g. 1 mg/kg = 1 ppm = 10^{-6} kg/kg)

3.6.2 Value by precision experiment

Using the reproducibility standard deviation σ_R and the repeatability standard deviation σ_r of a precision experiment (collaborative trial or proficiency test) the target standard deviation σ_{pt} can be derived considering the number of replicate measurements m of participants in the present PT [3]:

$$\sigma_{pt} = \sqrt{\sigma_R^2 - \sigma_r^2 \left(m-1 / m \right)}$$

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) given in Table 1 were determined in ring tests using the indicated methods. There were no appropriate precision data given in the Resolution of the Council of Europe [18]. For comparison precision data for the determination of water were given [19, 20].

Table 1: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) according to selected evaluations of tests for precision and the resulting target standard deviation σ_{pt} [19, 20]

Parameter	**Matrix**	**Mean** [mg/L]	**RSD_r**	**RSD_R**	**σ_{pt}**	**Method / Literature**
Lead	Waste water	0,147	2,9%	7,2%	6,8%	ICP-OES [20]
	Drinking water	0,0184	8,2%	15,8%	14,3%	ICP-OES [20]
	Surface water	0,0576	3,4%	9,2%	8,7%	ICP-OES [20]
Chromium	Waste water	0,0983	3,3%	5,6%	4,9%	ICP-OES [20]
	Drinking water	0,0162	3,1%	9,0%	8,6%	ICP-OES [20]
	Surface water	0,0302	2,1%	5,8%	5,4%	ICP-OES [20]
Iron	Waste water	0,817	1,8%	5,9%	5,7%	ICP-OES [20]
	Drinking water	0,196	1,7%	4,7%	4,5%	ICP-OES [20]
	Surface water	0,302	1,9%	4,8%	4,5%	ICP-OES [20]
	Mineral water	13,798	2,58%	4,84%	4,4%	ICP-OES [19]
Copper	Waste water	1,957	1,4%	4,4%	4,3%	ICP-OES [20]
	Drinking water	0,634	1,2%	3,7%	3,6%	ICP-OES [20]
	Surface water	0,802	3,9%	4,8%	3,6%	ICP-OES [20]
Nickel	Waste water	0,192	1,6%	5,1%	4,9%	ICP-OES [20]
	Drinking water	0,0259	3,8%	7,4%	6,7%	ICP-OES [20]
	Surface water	0,0523	2,8%	6,4%	6,0%	ICP-OES [20]
Zinc	Waste water	1,210	2,4%	5,9%	5,6%	ICP-OES [20]
	Drinking water	0,124	1,9%	4,9%	4,6%	ICP-OES [20]
	Surface Water	0,187	1,5%	4,9%	4,7%	ICP-OES [20]

3.6.3 Value by perception

The target standard deviation for proficiency assessment can be set at a value that corresponds to the level of performance that the coordinator would wish laboratories to be able to achieve [3].

For the present evaluation the target standard deviation according to 3.6.1 was regarded suitable.

Table 2 shows selected characteristics of participants results of the present PT in comparison to the previous year.

3.7 z-Score

To assess the results of the participants the z-score is used. It indicates about which multiple of the target standard deviation (σ_{pt}) the result (x_i) of the participant is deviating from the assigned value (X_{pt}) [3].
Participants' z-scores are derived from:

$$z_i = \frac{\left(x_i - x_{pt}\right)}{\sigma_{pt}}$$

The requirements for the analytical performance are generally considered as fulfilled if

$$-2 \leq z \leq 2 \ .$$

The valid z-Score for each parameter is indicated as z-Score (σ_{pt}). The value indicated as z-Score (Info) only obtains a informative character. The both z-Scores were calculated with the different target standard deviations in accordance with 3.6.

3.7.1 Warning and action signals

In accordance with the norm ISO 13528 it is recommended that a result that gives rise to a *z*-score above 3,0 or below −3,0, shall be considered to give an "action signal" [3]. Likewise, a *z*-score above 2,0 or below −2,0 shall be considered to give a "warning signal". A single "action signal", or "warning signal" in two successive PT-rounds, shall be taken as evidence that an anomaly has occurred which requires investigation.

An error or cause analysis can be carried out by checking the analysis process including understanding and implementation of the measurement by the staff, details of the measurement procedure, calibration of equipment and composition of reagents, transmission error or an error in the calculation, in the trueness and precision and use of reference material. If necessary, the problems must be addressed through appropriate corrective action [3].

In the figures of z-scores DLA gives the limits of warning and action signals as yellow and red lines respectively. According to ISO 13528 the signals are valid only in case of a number of ≥ 10 results [3].

Table 2: Characteristics of the present PT (on dark gray) in comparison to previous PTs since 2015 (SD = standard deviation, CV = coefficient of variation)

Parameter	**Matrix**	**rob. Mean [mg/L]**	**rob. SD (S*) [mg/L]**	**rel. SD (VK_{S*}) [%]**	**Quotient S^*/σ_{pt}**	**DLA-Report**
Aluminium	Mug stainless steel	0,0424	0,0260	61,3	1,9°	DLA 48/2015
Aluminium	Mug stainless steel	0,0863	0,321	37,1	1,4°	DLA 55/2019
Lead	Jug chromium steel	0,0148	0,00527	35,6	1,6	DLA 69/2016
Lead	Mug stainless steel	0,000938	0,000513	54,8	1,6°	DLA 55/2019
Chromium	Mug stainless steel	0.116	0.0226	19.5	0.88	DLA 48/2015
Chromium	Jug chromium steel	0.262	0.0404	15.4	0.79	DLA 69/2016
Chromium	Mug stainless steel	0,251	0,0539	21,5	1,1	DLA 55/2019
Iron	Mug stainless steel	5.13	1.55	30.2	2.0	DLA 48/2015
Iron	Jug chromium steel	7.84	1.30	16.6	1.4	DLA 69/2016
Iron	Mug stainless steel	5,88	1,02	17,3	1,4	DLA 55/2019
Manganese	Mug stainless steel	0,667	0,166	24,9	1,5	DLA 48/2015
Manganese	Mug stainless steel	0,0803	0,0187	23,3	1,1	DLA 55/2019
Nickel	Jug chromium steel	0,0240	0,00529	22,0	1,0	DLA 69/2016
Nickel	Mug stainless steel	0,159	0,0280	17,6	0,83	DLA 55/2019

° *Calculated with z'-Score*

3.8 z'-Score

The z'-score can be used for the valuation of the results of the participants, in cases the standard uncertainty has to be considered (s. 3.11). The z'-score represents the relation of the deviation of the result (x_i) of the participant from the respective consensus value (X) to the square root of quadrat sum of the target standard deviation (σ_{pt}) and the standard uncertainty ($U_{(xpt)}$) [3].

The calculation is performed by:

$$z'_i = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u_{(x_{pt})}^2}}$$

If carried out an evaluation of the results by means of z 'score, we have defined below the expression in the denominator as a target standard deviation σ_{pt}'.

The requirements for the analytical performance are generally considered as fulfilled if

$$-2 \leq z' \leq 2 \ .$$

For warning and action signals see 3.7.1.

3.9 Reproducibility cofficient of variation (CV_R)

The variation coefficient (CV_R) of the reproducibility (= *relative reproducibility standard deviation)* is calculated from the standard deviation and the mean as follows [4, 13]:

$$CV_R = \frac{S_R * 100}{X}$$

In contrast to the standard deviation as a measure of the absolute variability the CV_R gives the relative variability within a data region. While a low CV_R, e.g. <5-10% can be taken as evidence for a homogeneous set of results, a CV_R of more than 50% indicates a "strong inhomogeneity of statistical mass", so that the suitability for certain applications such as the assessment of exceeded maximum levels or the performance evaluation of the participating laboratories possibly can not be done [3].

3.10 Quotient S*/σ_{pt}

Following the HorRat-value the results of a proficiency-test (PT) can be considered convincing, if the quotient of robust standard deviation S* and target standard deviation σ_{pt} does not exceed the value of 2.
A value > 2 means an insufficient precision, i.e. the analytical method is too variable, or the variation between the test participants is higher than estimated. Thus the comparability of the results is not given [3].

3.11 Standard uncertainty of the assigned value

Every assigned value has a standard uncertainty that depends on the analytical method, differences between the analytical methods used, the test material, the number of participating laboratories (P) and on other factors. The standard uncertainty ($U_{(Xpt)}$) for this PT is calculated as follows [3]:

$$u_{(x_{pt})} = 1{,}25 \times \frac{s^*}{\sqrt{p}}$$

If $U_{(Xpt)} \leq 0{,}3\ \sigma_{pt}$ the standard uncertainty of the assigned value needs not to be included in the interpretation of the results of the PT [3]. Values exceeding 0,3 imply, that the target standard deviation could be too low with respect to the standard uncertainty of the assigned value.

The traceability of the assigned value is ensured on the basis of the consensus value as a robust mean of the participant results.

4. Results

All following tables are anonymized. With the delivering of the evaluation report the participants are informed about their individual evaluation number.

In the first table the characteristics are listed:

Statistic Data
Number of results
Number of outliers
Mean
Median
Robust mean(X_{pt})
Robust standard deviation **(S*)**
Number with m replicate measurements
Repeatability standard deviation (S_r)
Coefficient of Variation (CV_r)in %
Reproducibility standard deviation (S_R)
Coefficient of Variation (CV_R)in %
Target range:
Target standard deviation σ_{pt} or σ_{pt}'
lower limit of target range ($X_{pt} - 2\sigma_{pt}$) or ($X_{pt} - 2\sigma_{pt}$') *
upper limit of target range ($X_{pt} + 2\sigma_{pt}$) or ($X_{pt} + 2\sigma_{pt}$´) *
Quotient S/σ_{pt} or S*/σ_{pt}'*
Standard uncertainty U(X_{pt})
Number of results in the target range
Percent in the target range

* Target range is calculated with z-score or z'-score

In the table below, the results of the participating laboratories are formatted in 3 valid digits**:

Auswerte-nummer **Evaluation number**	**Parameter [Einheit / Unit]**	**Abweichung** **Deviation**	**z-Score σpt**	**z-Score** (Info)	**Hinweis** **Remark**

*** In the documentation part, the results are given as they were transmitted by the participants.*

4.1 Aluminium in mg/L

1. Eluat /1st Eluate

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	0,0863
Median	0,0800
Robust Mean (X_{pt})	**0,0863**
Robust standard deviation (S*)	**0,0321**
Number with 3 replicates	9
Repeatability SD (S_r)	0,0184
Repeatability (CV_r)	22,6%
Reproducibility SD (S_R)	0,0284
Reproducibility (CV_R)	34,9%
Target range:	
Target standard deviation σ_{pt}'	**0,0228**
lower limit of target range	**0,041**
upper limit of target range	**0,132**
Quotient S/σ_{pt}'*	*1,4*
Standard uncertainty $U_{(Xpt)}$	*0,0127*
Results in the target range	*10*
Percent in the target range	*100%*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz/Thompson considering the standard uncertainty.

The distribution of results showed a slightly increased variability compared to the standard target deviation according to Thompson. Thus, the standard target deviation σ_{pt}' and the z'-score were used for evaluation. The quotient S/σ_{pt}' was below 2,0. The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The repeatability and reproducibility standard deviation are above the range of established values gained by determination methods in water (s. 3.6.2). However, the comparability of results is given for used methods and the present range of concentrations.*

100% of results were in the target range.

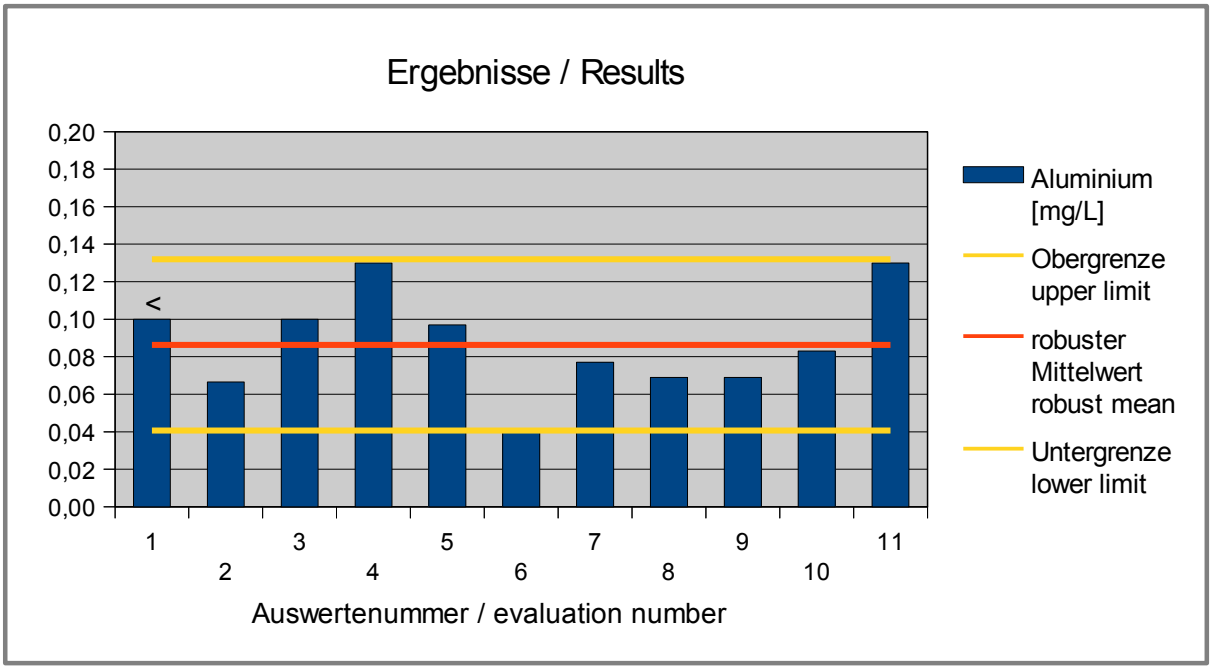

Abb. / Fig. 1: Results Aluminium

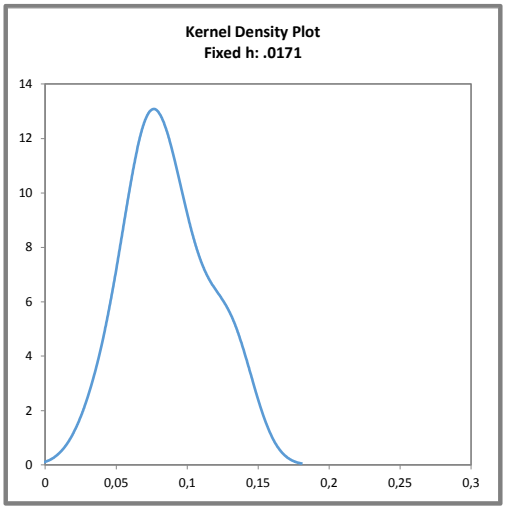

Abb. / Fig. 2:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results (with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows almost a symmetrical distribution of results with a slight shoulder.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Aluminium [mg/L]	Abweichung [mg/L] Deviation [mg/L]	z'-Score (σ_{pt})	Hinweis Remark
1	< 0,100			
2	0,067	-0,0197	-0,86	
3	0,100	0,0137	0,60	
4	0,130	0,0437	1,9	
5	0,097	0,0107	0,47	
6	0,042	-0,0446	-2,0	
7	0,077	-0,0093	-0,41	
8	0,069	-0,0173	-0,76	
9	0,069	-0,0173	-0,76	
10	0,083	-0,0033	-0,15	
11	0,130	0,0437	1,9	

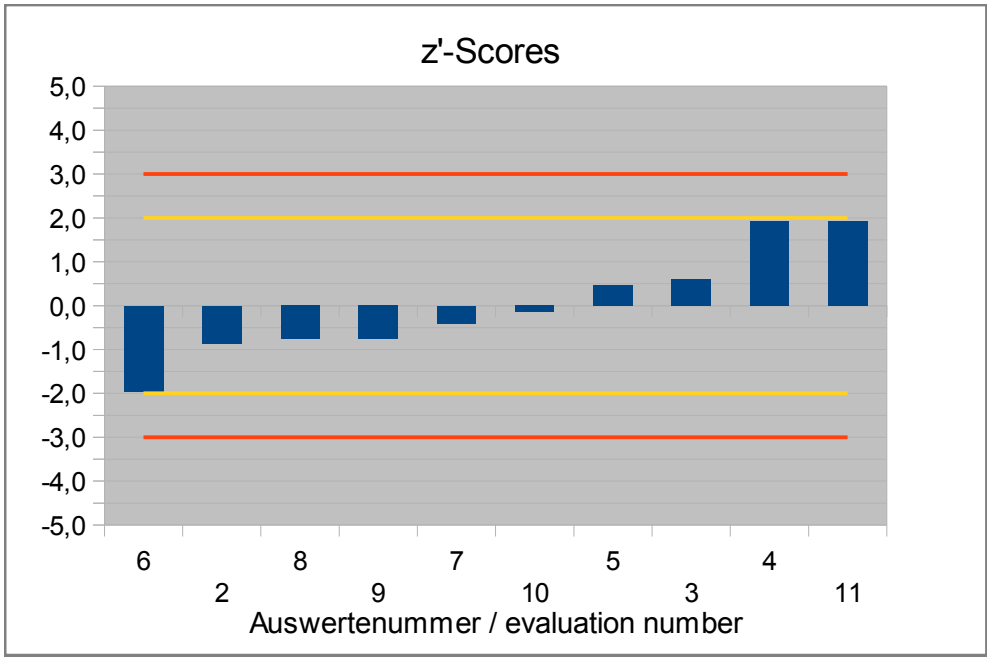

Abb. / Fig. 3: z'-Scores Aluminium

Summe der Eluate 1 + 2 + 3 / Sum of Eluates 1 + 2 + 3

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	6
Number of outliers	1
Mean	0,119
Median	0,104
Robust Mean (X_{pt})	**0,119**
Robust standard deviation (S*)	**0,0572**
Number with 3 replicates	9
Repeatability SD (S_r)	0,0184
Repeatability (CV_r)	22,6%
Reproducibility SD (S_R)	0,0284
Reproducibility (CV_R)	34,9%
Target range:	
Target standard deviation σ_{pt}'	**0,0393**
lower limit of target range	**0,0408**
upper limit of target range	**0,198**
Quotient S/σ_{pt}'*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*0,0292*
Results in the target range	*6*
Percent in the target range	*100%*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz/Thompson considering the standard uncertainty.

The distribution of results showed a slightly increased variability compared to the standard target deviation according to Thompson. Thus, the standard target deviation σ_{pt}' and the z'-Score were used for evaluation. The quotient S/σ_{pt}' was below 2,0. Thus the comparability of results is given for used methods and the present range of concentrations.*

100% of results were in the target range.

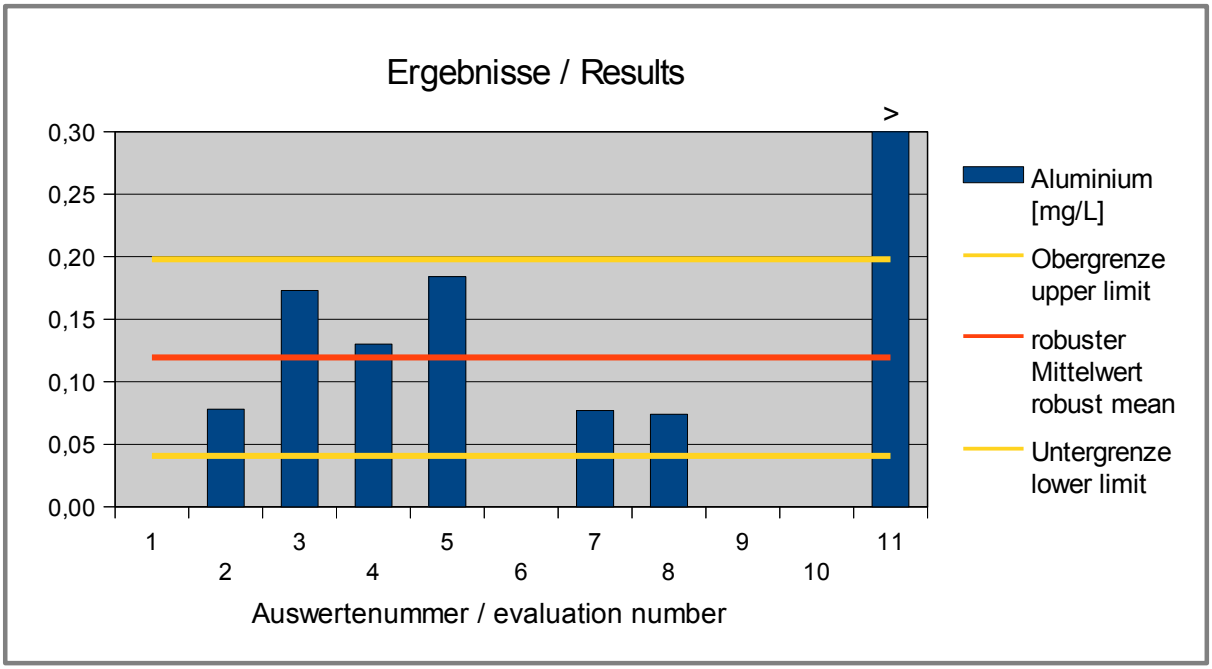

Abb. / Fig. 4: Results Aluminium (Sum of Eluates 1 + 2 + 3)

Comment:
A kernel density estimation was not made due to the number of <8 results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Aluminium [mg/L]	Abweichung [mg/L] Deviation [mg/L]	z'-Score (σ_{pt})	Hinweis Remark
1				
2	0,0781 *	-0,0412	-1,0	
3	0,173 *	0,0536	1,4	
4	0,130 *	0,0106	0,3	
5	0,184 *	0,0646	1,6	
6				
7	0,0770 *	-0,0424	-1,1	
8	0,0740 *	-0,0454	-1,2	
9				
10				
11	0,380 *			Ergebnis ausgeschlossen / Result excluded

* The sums of the eluates were calculated by DLA

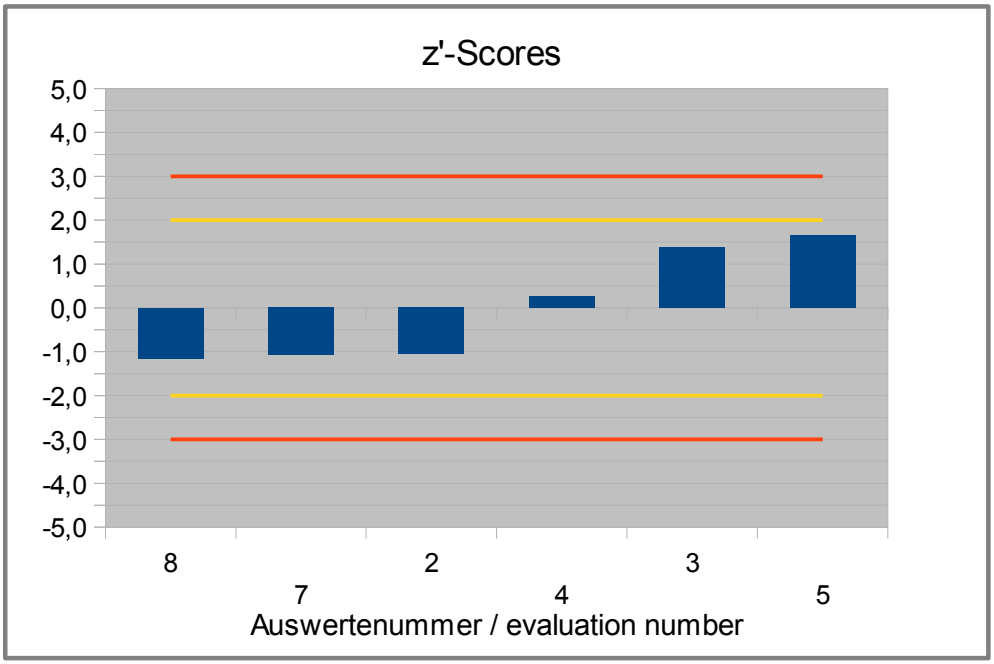

Abb. / Fig. 5: z'-Scores Aluminium (Sum of Eluates 1 + 2 + 3)

4.2 Chromium in mg/L

1. Eluat /1st Eluate

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	0,253
Median	0,241
Robust Mean (x_{pt})	**0,251**
Robust standard deviation (S*)	**0,0539**
Number with 3 replicates	9
Repeatability SD (S_r)	0,0378
Repeatability (CV_r)	15,9%
Reproducibility SD (S_R)	0,0478
Reproducibility (CV_R)	20,1%
Target range:	
Target standard deviation σ_{pt}	**0,0495**
lower limit of target range	**0,152**
upper limit of target range	**0,350**
Quotient S/σ_{pt}*	*1,1*
Standard uncertainty $U_{(Xpt)}$	*0,0203*
Results in the target range	*11*
Percent in the target range	*100%*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz.

The distribution of results showed a normal variability. The quotient S/σ_{pt} was below 2,0. The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The repeatability and reproducibility standard deviation are in the range of established values gained by determination methods in water (s. 3.6.2). Thus the comparability of results is given for used methods and the present range of concentrations.*

All results were in the target range.

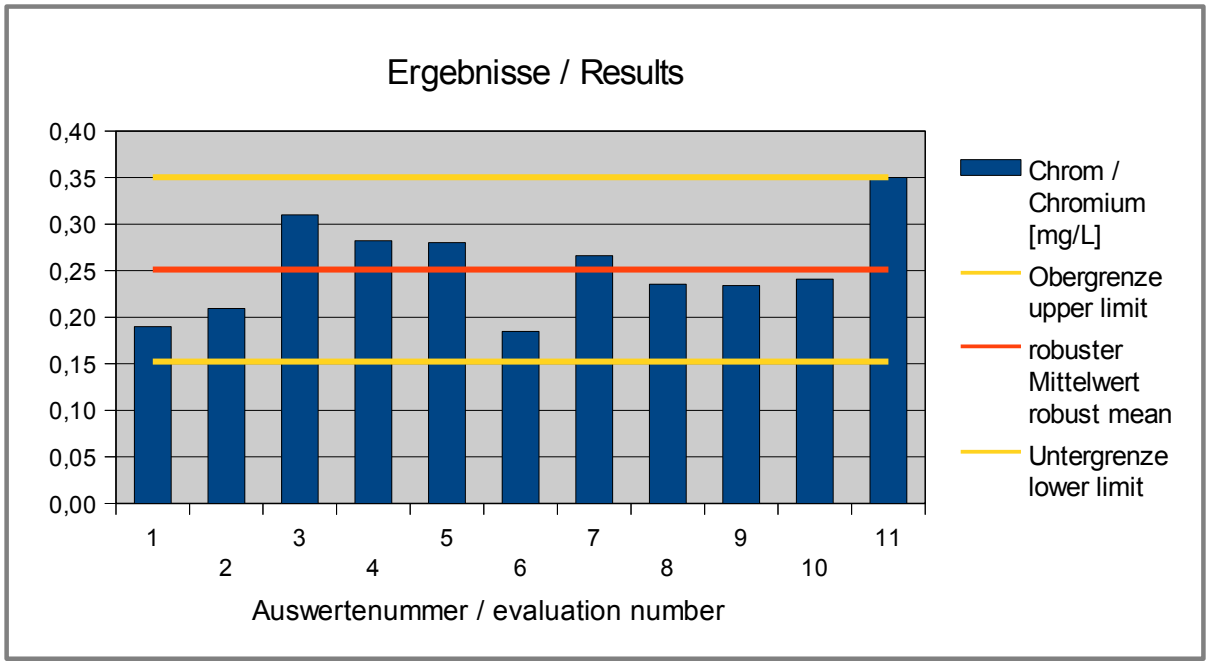

Abb. / Fig. 6: Results Chromium

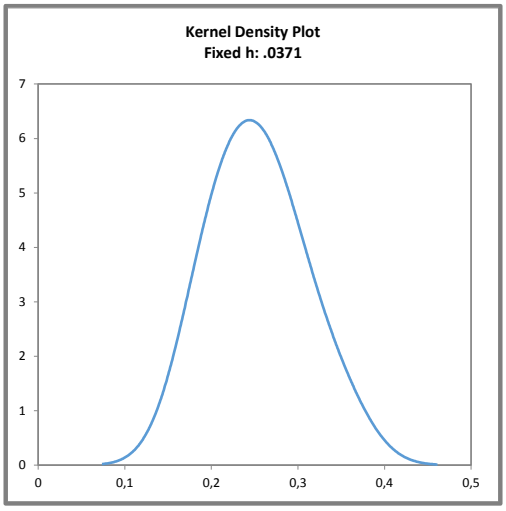

Abb. / Fig. 7:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results (with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows almost a symmetrical distribution of results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Chrom / Chromium [mg/L]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	0,190	-0,0613	-1,2	
2	0,209	-0,0420	-0,8	
3	0,310	0,0587	1,2	
4	0,282	0,0307	0,6	
5	0,280	0,0287	0,6	
6	0,185	-0,0666	-1,3	
7	0,266	0,0147	0,3	
8	0,236	-0,0158	-0,3	
9	0,234	-0,0173	-0,4	
10	0,241	-0,0103	-0,2	
11	0,350	0,0987	2,0	

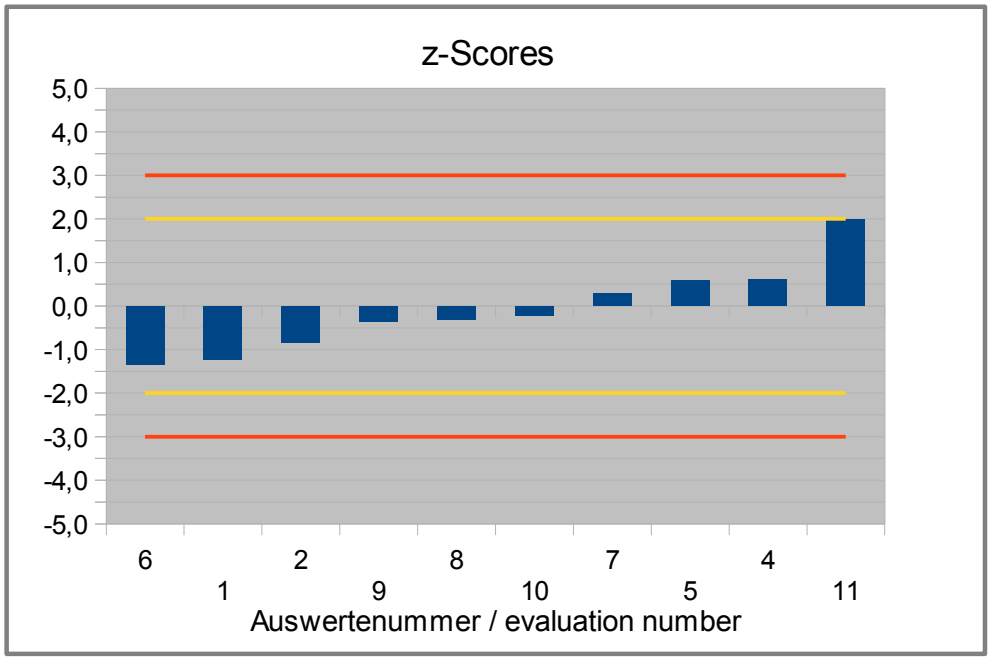

Abb. / Fig. 8: z-Scores Chromium

Summe der Eluate 1 + 2 + 3 / Sum of Eluates 1 + 2 + 3

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	6
Number of outliers	1
Mean	0,421
Median	0,437
Robust Mean (X_{pt})	**0,421**
Robust standard deviation (S*)	**0,0740**
Number with 3 replicates	6
Repeatability SD (S_r)	0,0821
Repeatability (CV_r)	19,7%
Reproducibility SD (S_R)	0,0852
Reproducibility (CV_R)	20,4%
Target range:	
Target standard deviation σ_{pt}	**0,0768**
lower limit of target range	**0,268**
upper limit of target range	**0,575**
Quotient S/σ_{pt}*	*0,96*
Standard uncertainty $U_{(Xpt)}$	*0,0378*
Results in the target range	*6*
Percent in the target range	*100%*

° without result No. 11 (outlier excluded)

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz.

The distribution of results showed a normal variability. The quotient S/σ_{pt} was below 2,0. The comparability of results is given for used methods and the present range of concentrations.*

100% of results were in the target range.

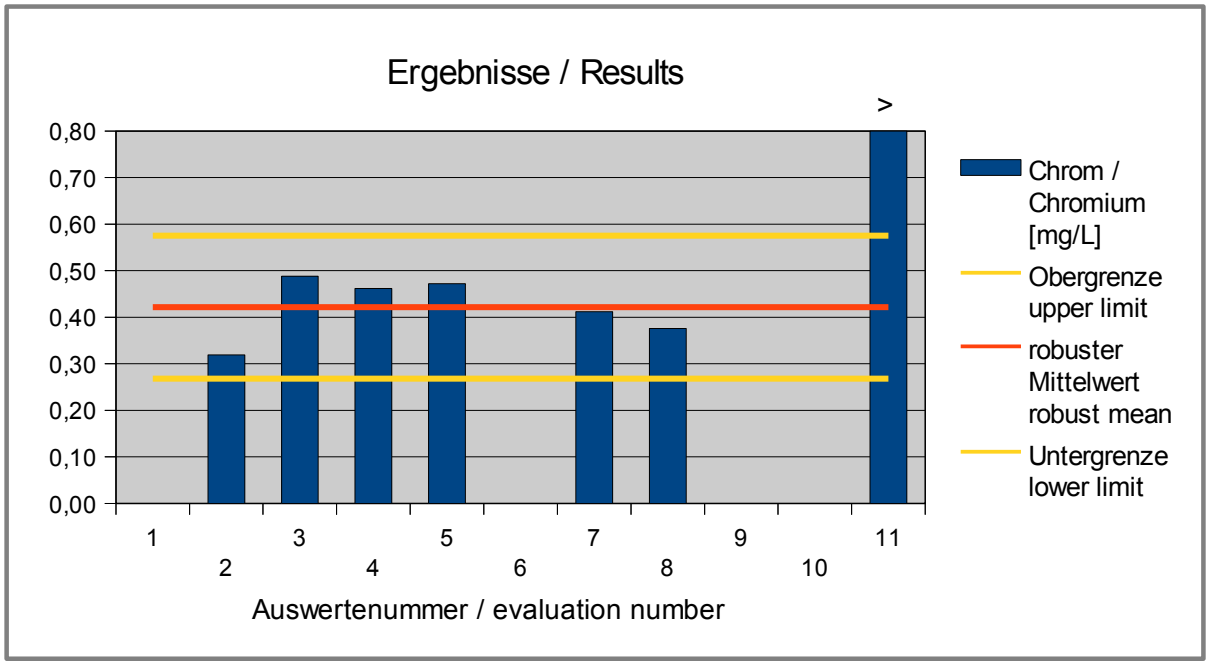

Abb. / Fig. 9: Results Chromium (Sum of Eluates 1 + 2 + 3)

Comment:
A kernel density estimation was not made due to the number of <8 results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Chrom / Chromium [mg/L]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1				
2	0,319 *	-0,1023	-1,3	
3	0,488 *	0,0666	0,87	
4	0,462 *	0,0406	0,53	
5	0,472 *	0,0506	0,66	
6				
7	0,412 *	-0,0094	-0,12	
8	0,376 *	-0,0459	-0,60	
9				
10				
11	0,980 *			Ergebnis ausgeschlossen / Result excluded

* The sums of the eluates were calculated by DLA

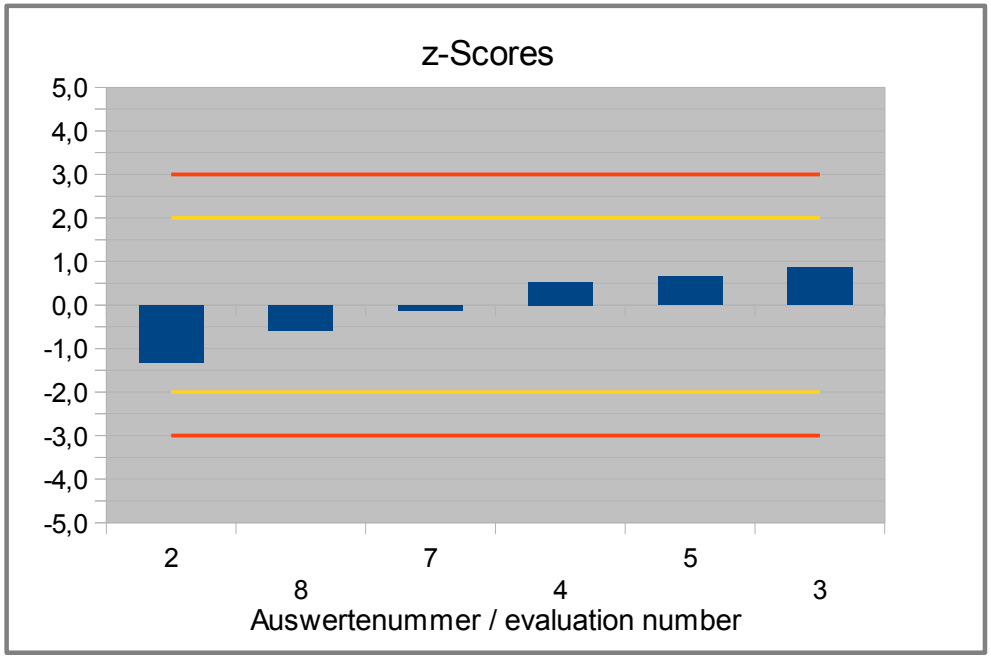

Abb. / Fig. 10: z-Scores Chromium (Sum of Eluates 1 + 2 + 3)

4.3 Iron in mg/L

1. Eluat /1st Eluate

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	5,93
Median	5,86
Robust Mean (X_{pt})	**5,88**
Robust standard deviation (S*)	**1,02**
Number with 3 replicates	10
Repeatability SD (S_r)	0,583
Repeatability (CV_r)	10,2%
Reproducibility SD (S_R)	0,882
Reproducibility (CV_R)	15,4%
Target range:	
Target standard deviation σ_{pt}	**0,721**
lower limit of target range	**4,44**
upper limit of target range	**7,32**
Quotient S/σ_{pt}*	*1,4*
Standard uncertainty $U_{(Xpt)}$	*0,384*
Results in the target range	*10*
Percent in the target range	*91%*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz.

The distribution of results showed a normal variability. The quotient S/σ_{pt} was below 2,0. The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The repeatability and reproducibility standard deviation are in the range of established values gained by determination methods in water (s. 3.6.2). Thus the comparability of results is given for used methods and the present range of concentrations.*

91% of the results were in the target range.

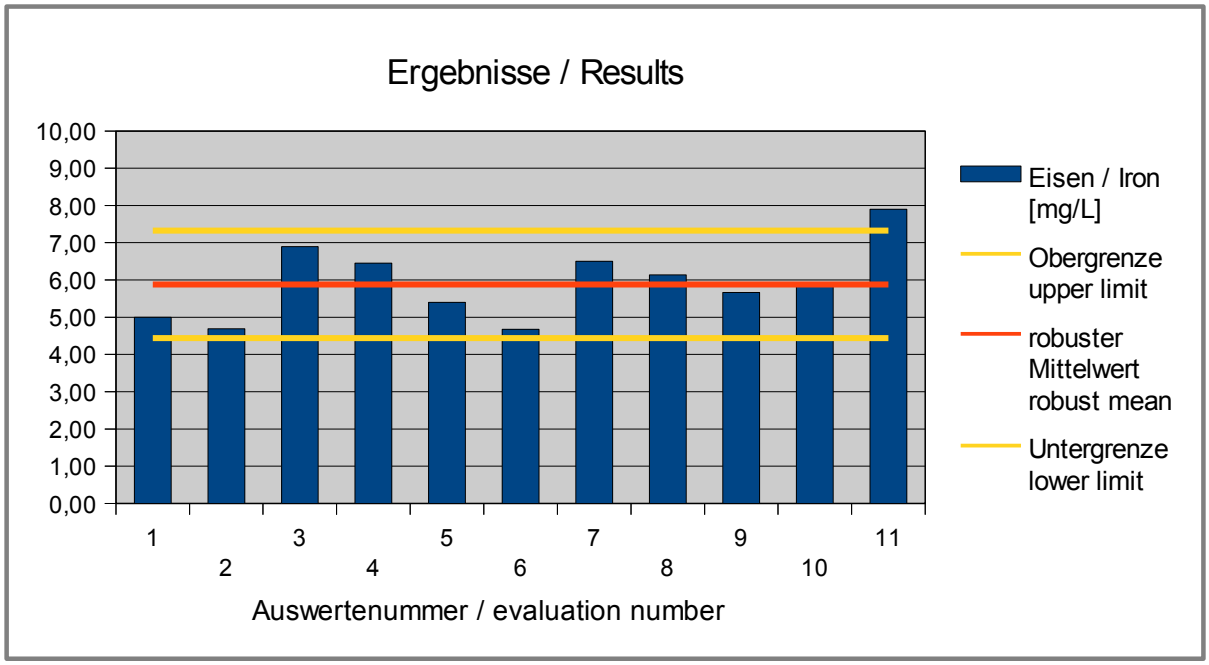

Abb. / Fig. 11: Results Iron

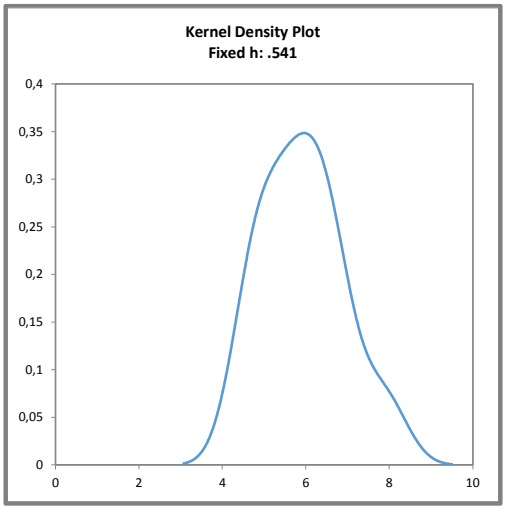

Abb. / Fig. 12:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results (with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows almost a symmetrical distribution of results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Eisen / Iron [mg/L]	Abweichung [mg/L] Deviation [mg/L]	z-Score (σ_{pt})	Hinweis Remark
1	5,00	-0,881	-1,2	
2	4,69	-1,189	-1,6	
3	6,90	1,019	1,4	
4	6,45	0,572	0,8	
5	5,40	-0,481	-0,7	
6	4,67	-1,208	-1,7	
7	6,50	0,619	0,9	
8	6,14	0,255	0,4	
9	5,67	-0,214	-0,3	
10	5,86	-0,021	0,0	
11	7,90	2,019	2,8	

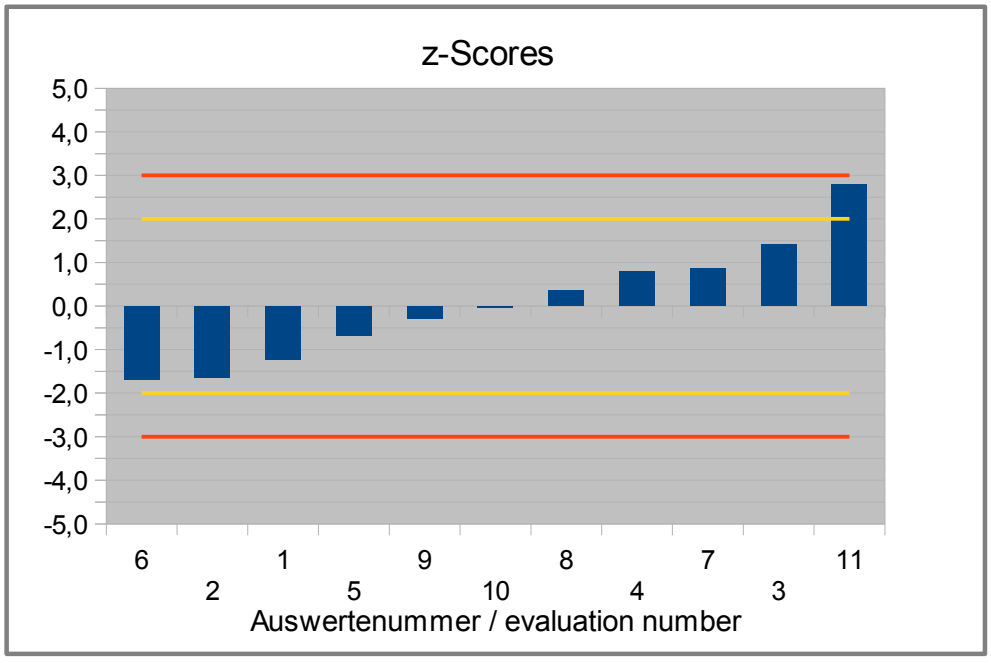

Abb. / Fig. 13: z-Scores Iron

Summe der Eluate 1 + 2 + 3 / Sum of Eluates 1 + 2 + 3

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	7
Number of outliers	1
Mean	7,20
Median	7,21
Robust Mean (X)	**7,20**
Robust standard deviation (S*)	**1,269**
Number with 3 replicates	7
Repeatability SD (S_r)	1,05
Repeatability (CV_r)	14,7%
Reproducibility SD (S_R)	1,29
Reproducibility (CV_R)	18,1%
Target range:	
Target standard deviation σ_{pt}	**0,856**
lower limit of target range	**5,49**
upper limit of target range	**8,91**
Quotient S/σ_{pt}*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*0,600*
Results in the target range	*7*
Percent in the target range	*100%*

° without result No. 11 (outlier excluded)

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz

The distribution of results showed a normal variability. The quotient S/σ_{pt} was below 2,0. The comparability of results is given for used methods and the present range of concentrations.*

100% of results were in the target range.

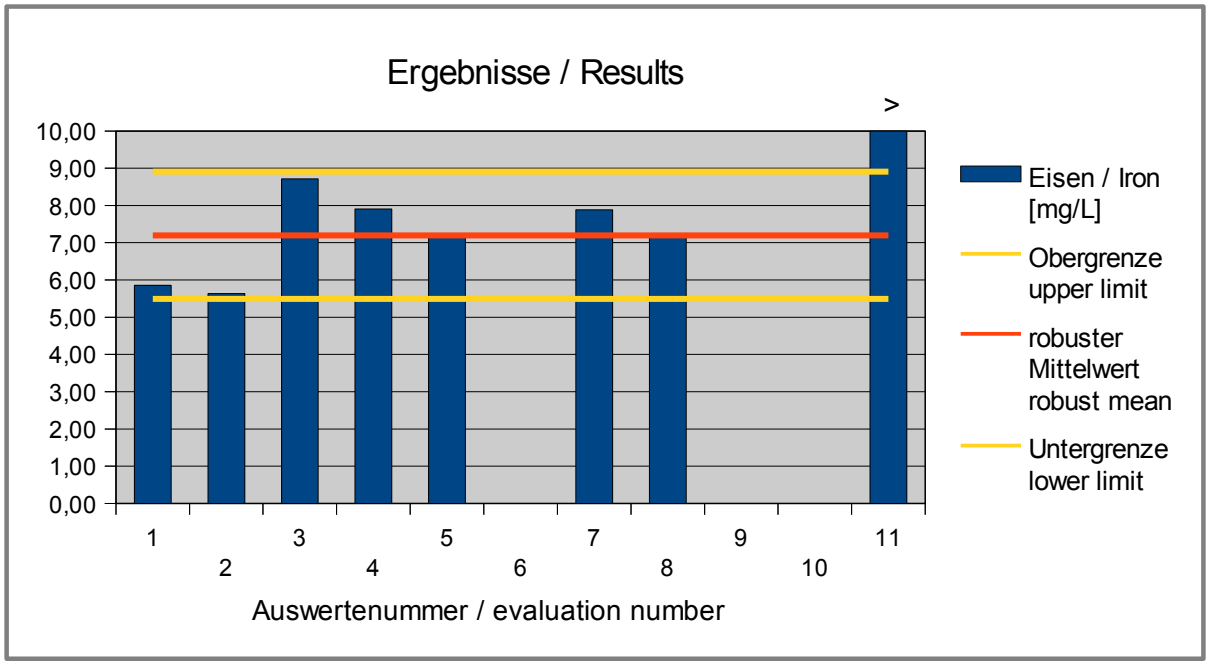

Abb. / Fig. 14: Results Iron (Sum of Eluates 1 + 2 + 3)

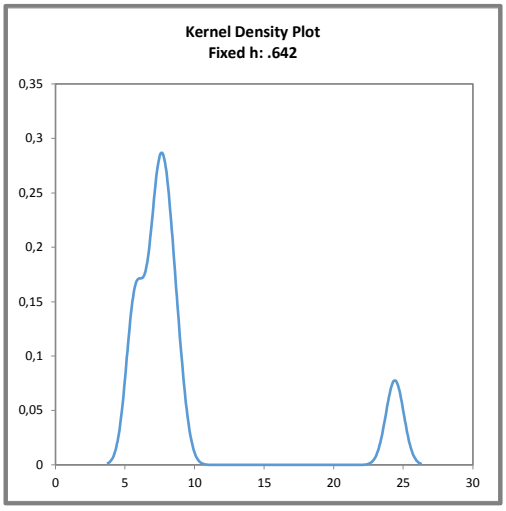

Abb. / Fig. 15:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results (with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows almost a symmetrical distribution of results with a shoulder and a secondary peak, due to a single value above the target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Eisen / Iron [mg/L]	Abweichung [mg/L] Deviation [mg/L]	z-Score (σ_{pt})	Hinweis Remark
1	5,86 *	-1,340	-1,6	
2	5,64 *	-1,564	-1,8	
3	8,72 *	1,517	1,8	
4	7,91 *	0,709	0,83	
5	7,21 *	0,010	0,01	
6				
7	7,89 *	0,685	0,80	
8	7,18 *	-0,018	-0,02	
9				
10				
11	24,4 *			Ergebnis ausgeschlossen / Result excluded

* The sums of the eluates were calculated by DLA

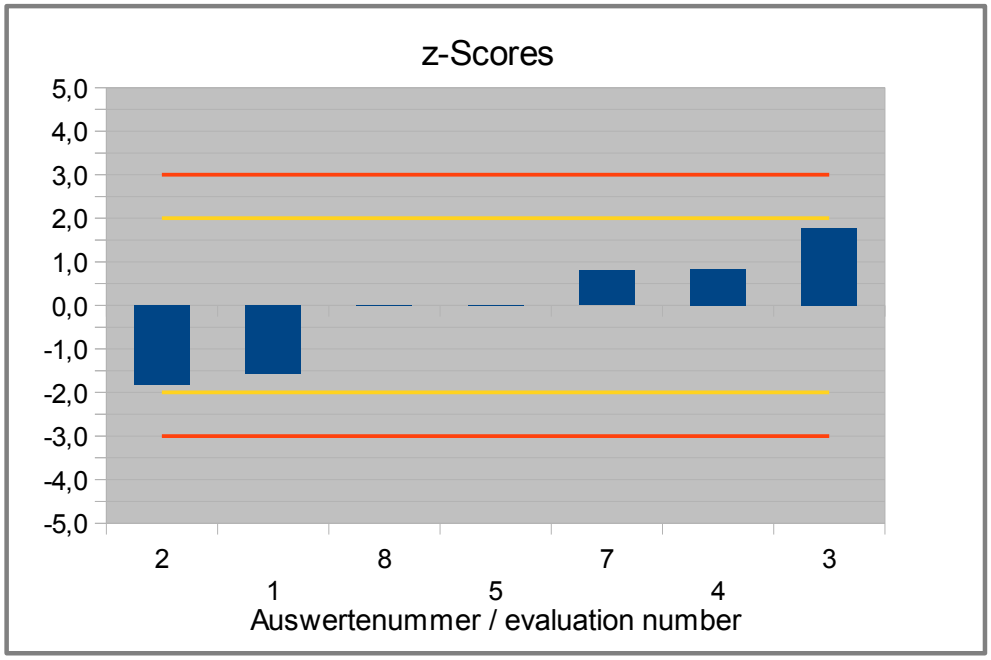

Abb. / Fig. 16: z-Scores Iron (Sum of Eluates 1 + 2 + 3)

4.4 Manganese in mg/L

1. Eluat /1st Eluate

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	0,0805
Median	0,0800
Robust Mean (X_{pt})	**0,0803**
Robust standard deviation (S*)	**0,0187**
Number with 3 replicates	10
Repeatability SD (S_r)	0,0190
Repeatability (CV_r)	24,3%
Reproducibility SD (S_R)	0,0204
Reproducibility (CV_R)	26,1%
Target range:	
Target standard deviation σ_{pt}	**0,0177**
lower limit of target range	**0,0450**
upper limit of target range	**0,116**
Quotient S/σ_{pt}*	*1,1*
Standard uncertainty $U_{(Xpt)}$	*0,0071*
Results in the target range	*11*
Percent in the target range	*100%*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz.

The distribution of results showed a normal variability. The quotient S/σ_{pt} was below 2,0. The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The repeatability and reproducibility standard deviation are in the range of established values gained by determination methods in water (s. 3.6.2). Thus the comparability of results is given for used methods and the present range of concentrations.*

All results were in the target range.

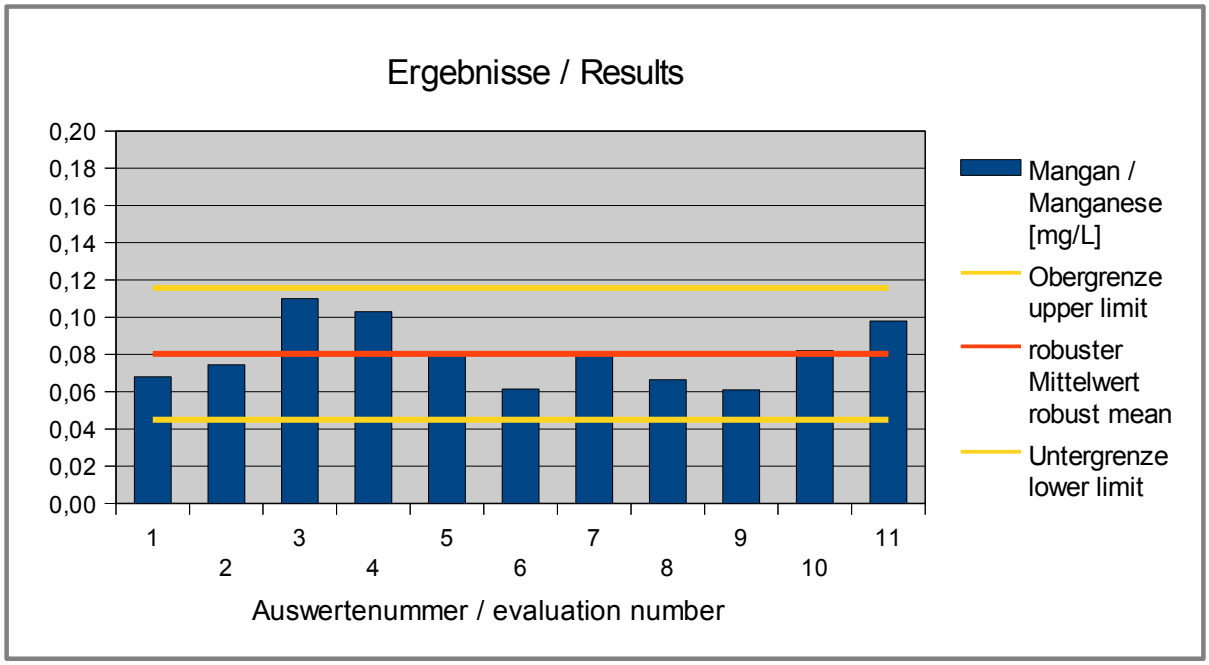

Abb. / Fig. 17: Results Manganese

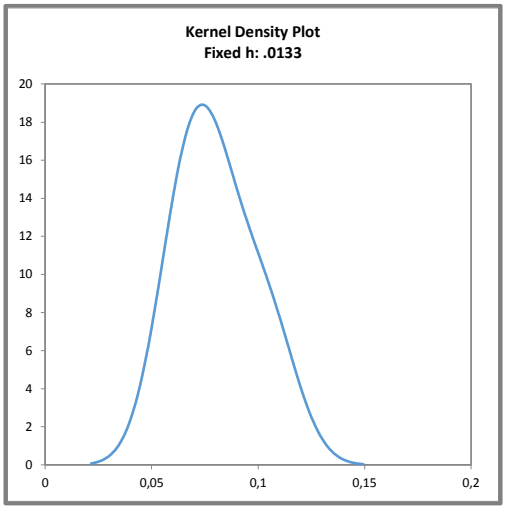

Abb. / Fig. 18:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results (with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows almost a symmetrical distribution of results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Mangan / Manganese [mg/L]	Abweichung [mg/L] Deviation [mg/L]	z-Score (σ_{pt})	Hinweis Remark
1	0,0680	-0,0123	-0,70	
2	0,0745	-0,0059	-0,33	
3	0,110	0,0297	1,7	
4	0,103	0,0227	1,3	
5	0,0810	0,0007	0,04	
6	0,0614	-0,0190	-1,1	
7	0,0800	-0,0003	-0,02	
8	0,0665	-0,0138	-0,78	
9	0,0610	-0,0193	-1,1	
10	0,0820	0,0017	0,09	
11	0,0980	0,0177	1,0	

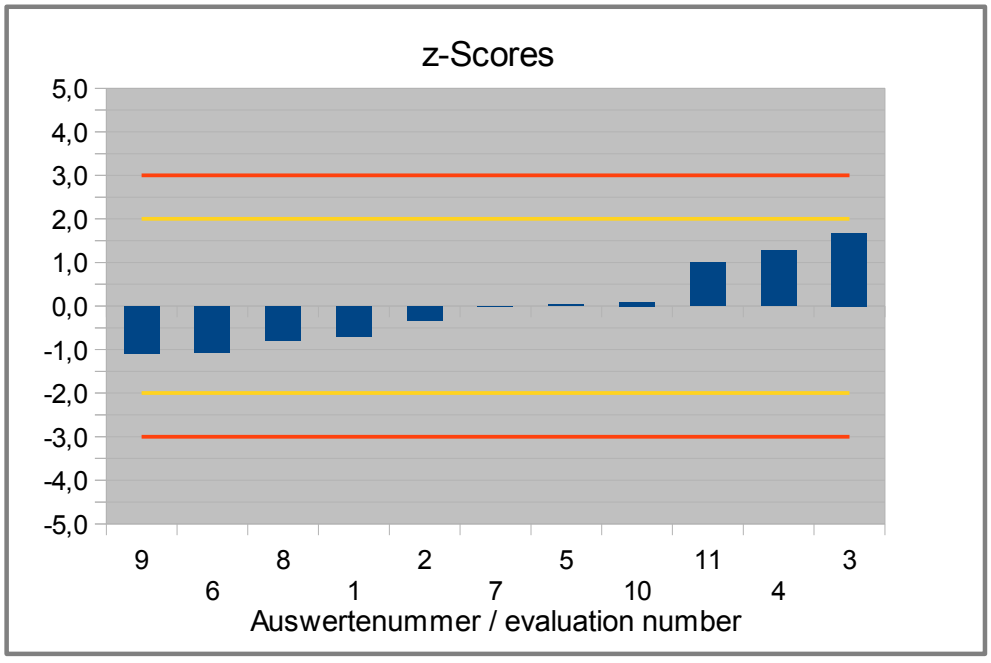

Abb. / Fig. 19: z-Scores Manganese

Summe der Eluate 1 + 2 + 3 / Sum of Eluates 1 + 2 + 3

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	6
Number of outliers	1
Mean	0,115
Median	0,100
Robust Mean (X_{pt})	**0,107**
Robust standard deviation (S*)	**0,0306**
Number with 3 replicates	5
Repeatability SD (S_r)	0,0229
Repeatability (CV_r)	24,2%
Reproducibility SD (S_R)	0,0252
Reproducibility (CV_R)	26,6%
Target range:	
Target standard deviation σ_{pt}	**0,0236**
lower limit of target range	**0,0600**
upper limit of target range	**0,154**
Quotient S^*/σ_{pt}	*1,3*
Standard uncertainty $U_{(Xpt)}$	*0,0156*
Results in the target range	*5*
Percent in the target range	*83%*

° without result No. 11 (outlier excluded)

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz/Thompson.

The distribution of results showed a normal variability. The quotient S^*/σ_{pt} was below 2,0. The comparability of results is given for used methods and the present range of concentrations.

83% of results were in the target range.

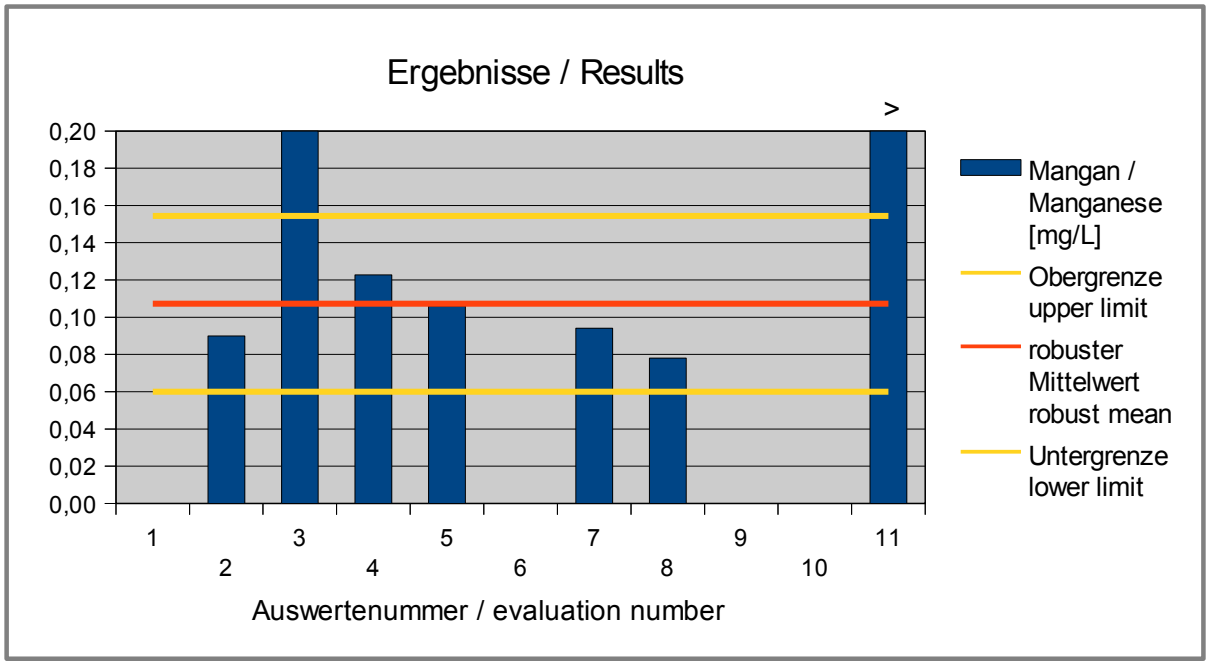

Abb. / Fig. 20: Results Manganese (Sum of Eluates 1 + 2 + 3)

Comment:
A kernel density estimation was not made due to the number of <8 results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Mangan / Manganese [mg/L]	Abweichung [mg/L] Deviation [mg/L]	z-Score (σ_{pt})	Hinweis Remark
1				
2	0,0899 *	-0,0173	-0,74	
3	0,200 *	0,0928	3,9	
4	0,123 *	0,0155	0,66	
5	0,106 *	-0,0012	-0,05	
6				
7	0,0940 *	-0,0132	-0,56	
8	0,0780 *	-0,0292	-1,24	
9				
10				
11	0,368 *			Ergebnis ausgeschlossen / Result excluded

* The sums of the eluates were calculated by DLA

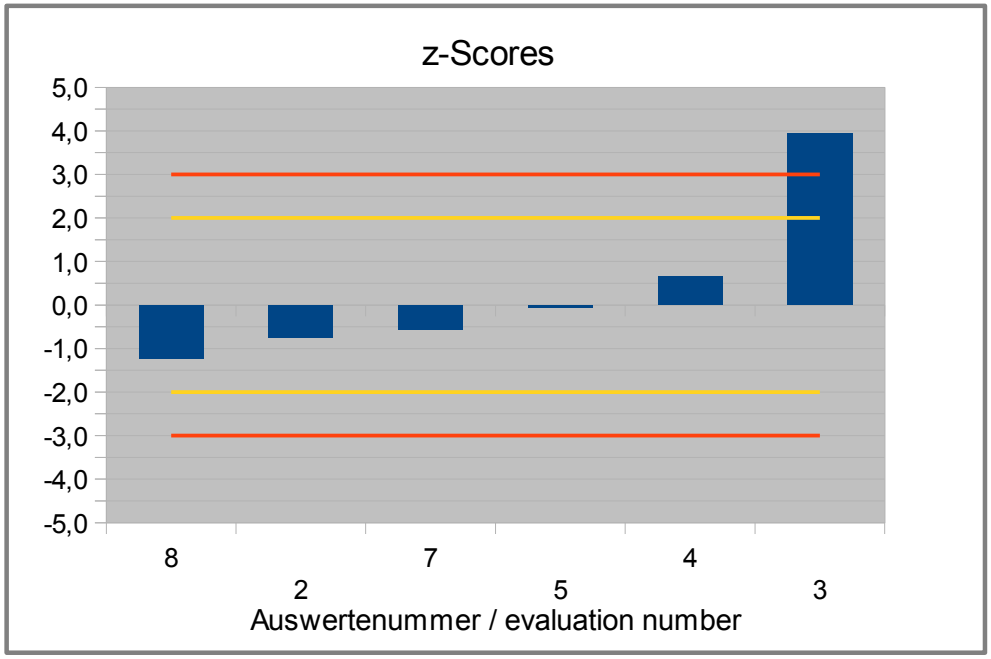

Abb. / Fig. 21: z-Scores Manganese (Sum of Eluates 1 + 2 + 3)

4.5 Nickel in mg/L

1. Eluat /1st Eluate

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	0,159
Median	0,153
Robust Mean (X_{pt})	**0,159**
Robust standard deviation (S*)	**0,0280**
Number with 3 replicates	10
Repeatability SD (S_r)	0,0287
Repeatability (CV_r)	18,6%
Reproducibility SD (S_R)	0,0293
Reproducibility (CV_R)	18,9%
Target range:	
Target standard deviation σ_{pt}	**0,0336**
lower limit of target range	**0,0920**
upper limit of target range	**0,226**
Quotient S/σ_{pt}*	*0,83*
Standard uncertainty $U_{(Xpt)}$	*0,0106*
Results in the target range	*11*
Percent in the target range	*100%*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz.

The distribution of results showed a normal variability. The quotient S/σ_{pt} was below 2,0. The robust standard deviation is comparable to those of prior PTs (see 3.6.3).The repeatability and reproducibility standard deviation are in the range of established values gained by determination methods in water (s. 3.6.2). Thus the comparability of results is given for used methods and the present range of concentrations.*

All results were in the target range.

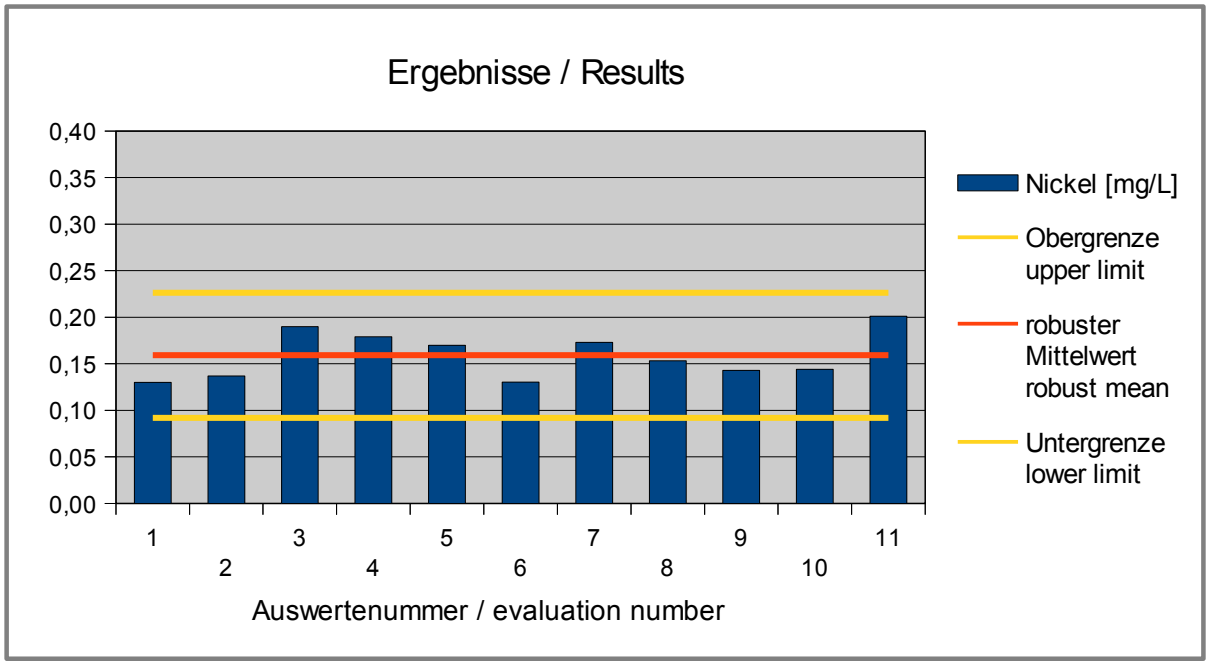

Abb. / Fig. 22: Results Nickel

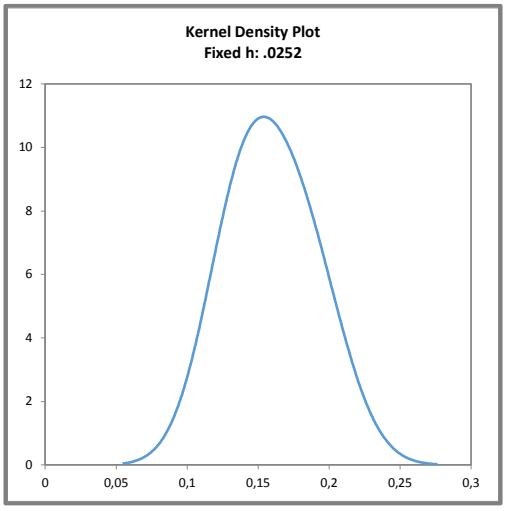

Abb. / Fig. 23:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results
(with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows almost a symmetrical distribution of results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Nickel [mg/L]	Abweichung [mg/L] Deviation [mg/L]	z-Score (σ_{pt})	Hinweis Remark
1	0,130	-0,0291	-0,87	
2	0,137	-0,0222	-0,66	
3	0,190	0,0309	0,92	
4	0,179	0,0199	0,59	
5	0,170	0,0109	0,32	
6	0,130	-0,0289	-0,86	
7	0,173	0,0139	0,41	
8	0,153	-0,0059	-0,18	
9	0,143	-0,0161	-0,48	
10	0,144	-0,0151	-0,45	
11	0,201	0,0419	1,2	

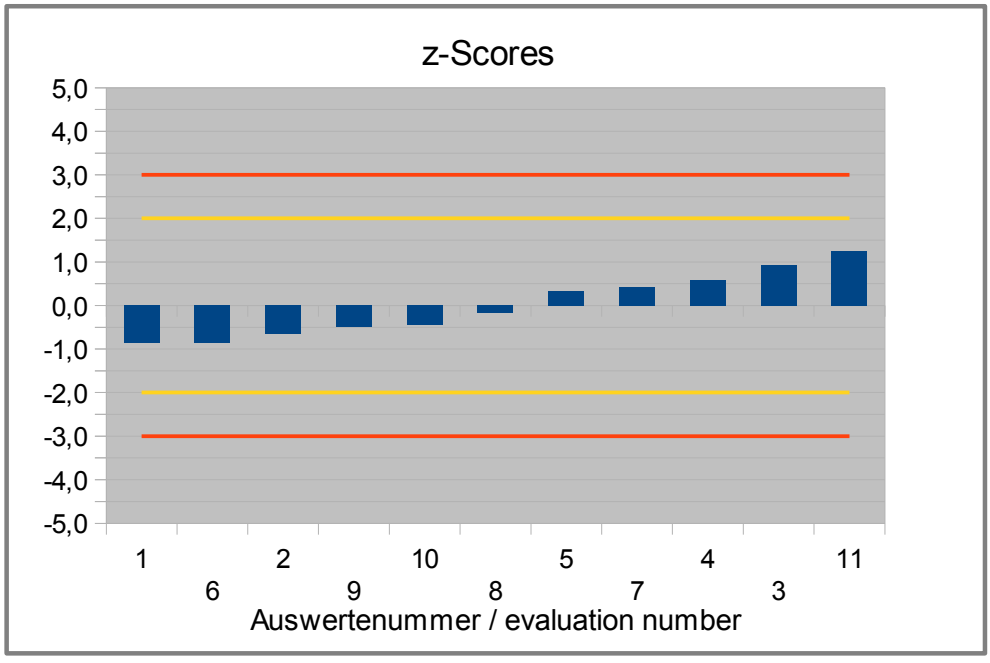

Abb. / Fig. 24: z-Scores Nickel

Summe der Eluate 1 + 2 + 3 / Sum of Eluates 1 + 2 + 3

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	7
Number of outliers	1
Mean	0,204
Median	0,223
Robust Mean (X_{pt})	**0,205**
Robust standard deviation (S*)	**0,0435**
Number with 3 replicates	7
Repeatability SD (S_r)	0,0444
Repeatability (CV_r)	21,7%
Reproducibility SD (S_R)	0,0515
Reproducibility (CV_R)	25,2%
Target range:	
Target standard deviation σ_{pt}	**0,0417**
lower limit of target range	**0,1220**
upper limit of target range	**0,289**
Quotient S^*/σ_{pt}	*1,0*
Standard uncertainty $U_{(Xpt)}$	*0,0206*
Results in the target range	*7*
Percent in the target range	*100%*

° without result No. 11 (outlier excluded)

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz.

The distribution of results showed a normal variability. The quotient S^*/σ_{pt} was below 2,0. The comparability of results is given for used methods and the present range of concentrations.

100% of results were in the target range.

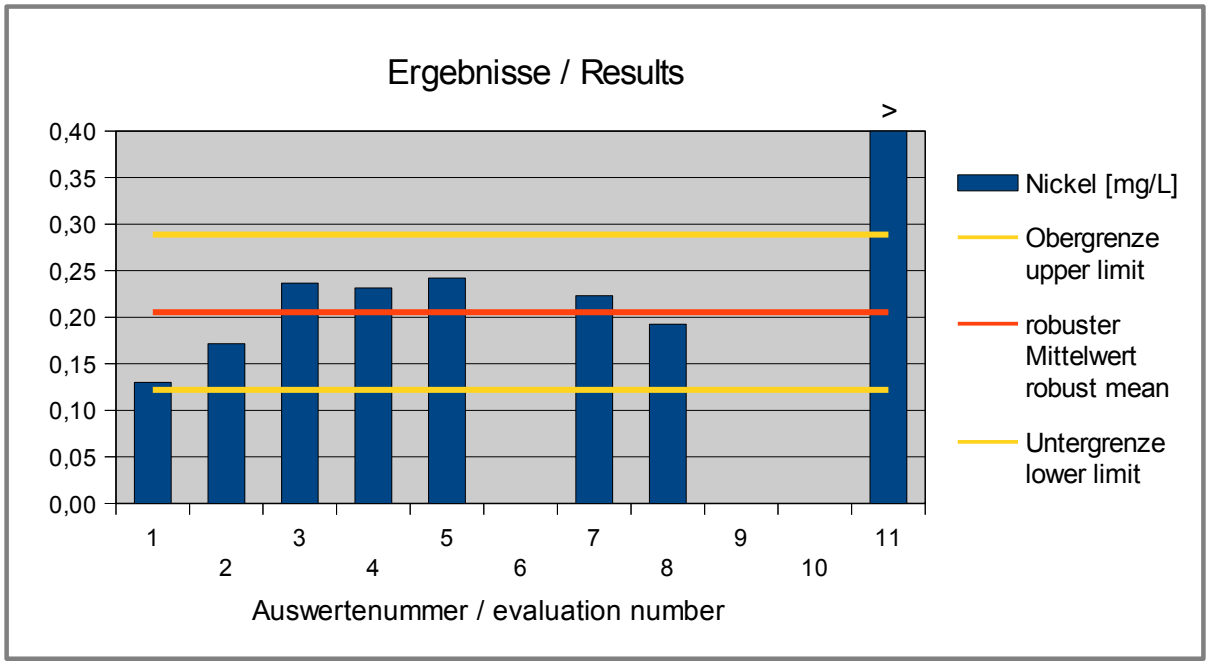

Abb. / Fig. 25: Results Nickel (Sum of Eluates 1 + 2 + 3)

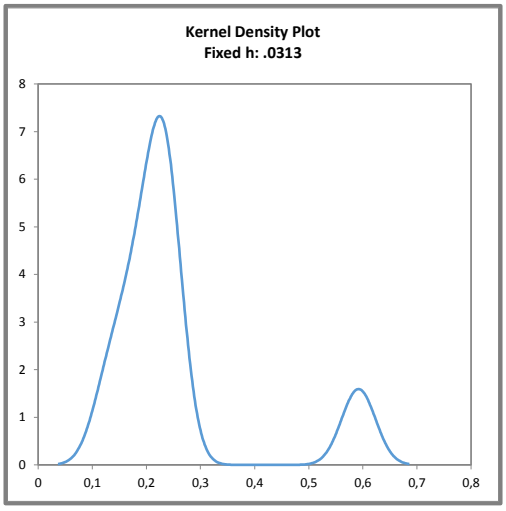

Abb. / Fig. 26:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results
(with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows almost a symmetrical distribution of results with a secondary peak, due to a single value above the target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Nickel [mg/L]	Abweichung [mg/L] Deviation [mg/L]	z-Score (σ_{pt})	Hinweis Remark
1	0,130 *	-0,0754	-1,8	
2	0,172 *	-0,0337	-0,81	
3	0,237 *	0,0313	0,75	
4	0,232 *	0,0261	0,63	
5	0,242 *	0,0366	0,88	
6				
7	0,223 *	0,0176	0,42	
8	0,193 *	-0,0129	-0,31	
9				
10				
11	0,592 *			Ergebnis ausgeschlossen / Result excluded

* The sums of the eluates were calculated by DLA

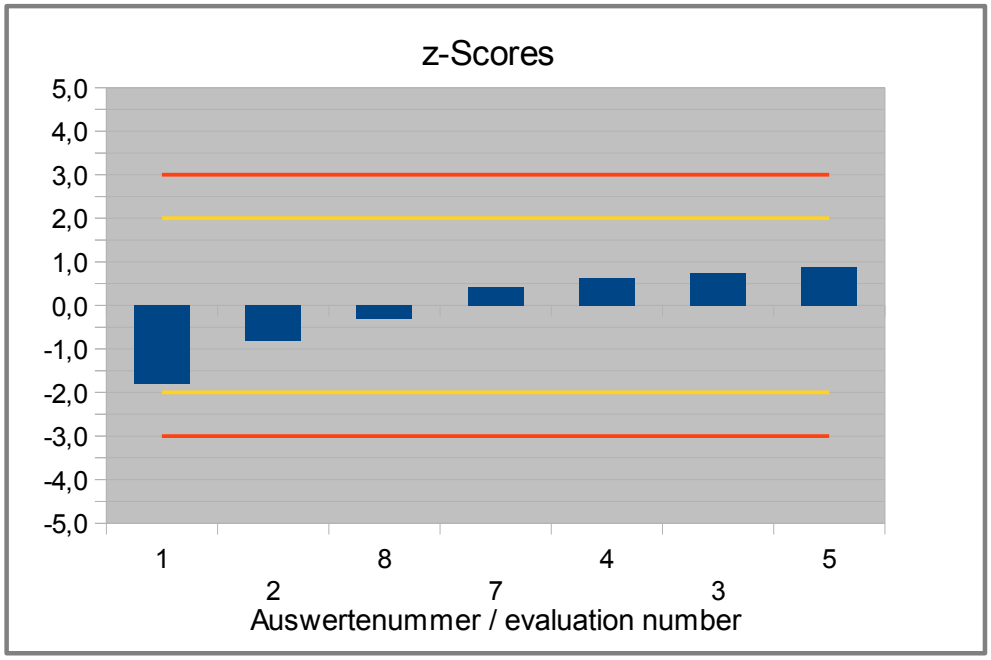

Abb. / Fig. 27: z-Scores Nickel (Sum of Eluates 1 + 2 + 3)

4.6 Lead in mg/L

1. Eluat /1st Eluate

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	7
Number of outliers	2
Mean	0,00121
Median	0,000640
Robust Mean (X_{pt})	**0,000938**
Robust standard deviation (S*)	**0,000513**
Number with 3 replicates	6
Repeatability SD (S_r)	0,0003
Repeatability (CV_r)	41,8%
Reproducibility SD (S_R)	0,0004
Reproducibility (CV_R)	53,3%
Target range:	
Target standard deviation σ_{pt}'	**0,000318**
lower limit of target range	**0,000301**
upper limit of target range	**0,00157**
Quotient S/σ_{pt}'*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*0,0002*
Results in the target range	*6*
Percent in the target range	*86%*

° without results No. 4 and 5 (outlier excluded)

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz/Thompson considering the standard uncertainty.

The distribution of results showed an increased variability. Thus, the standard target deviation σ_{pt}' and the z'-Score were used for evaluation. The quotient S/σ_{pt}' was below 2,0. The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The repeatability and reproducibility standard deviation are above the range of established values gained by determination methods in water (s. 3.6.2). However, the comparability of results is given for the used methods and the present range of concentrations.*

86% of results were in the target range.

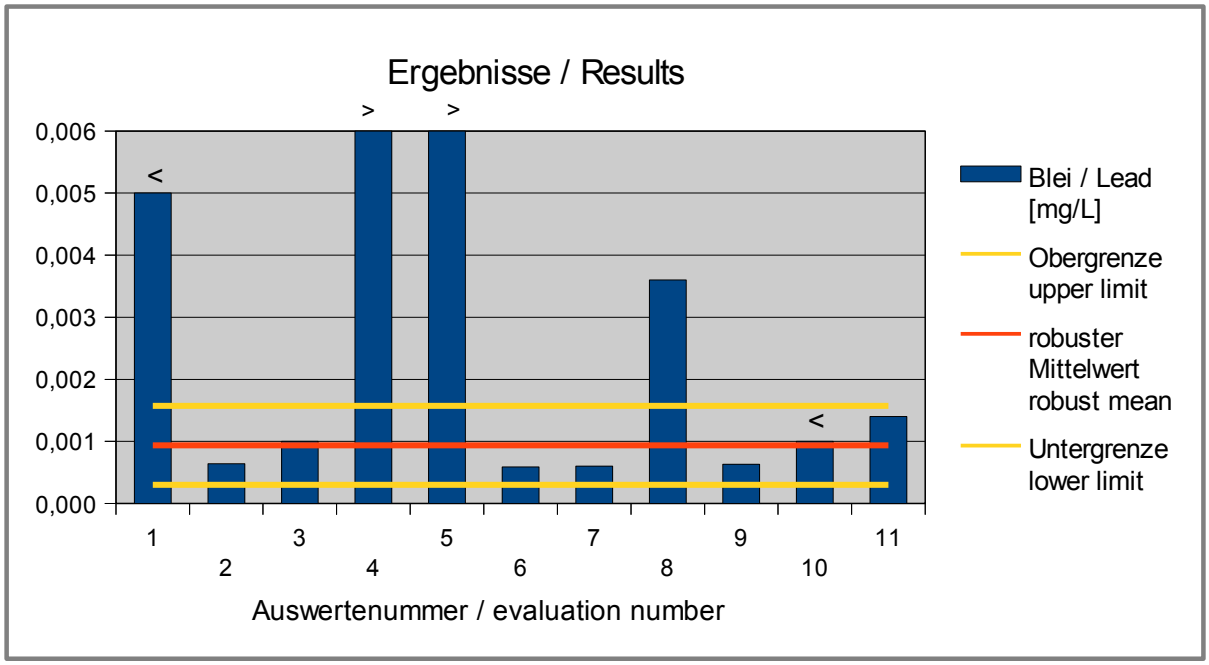

Abb. / Fig. 28: Results Lead

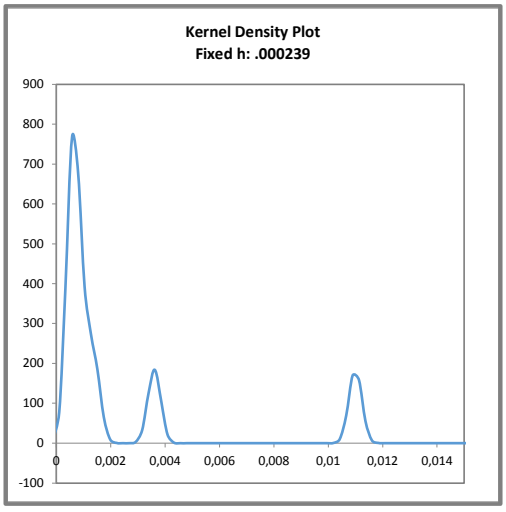

Abb. / Fig. 29:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results (with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows almost a symmetrical distribution of results with secondary peaks, due to three results above the target range (result at approx. 0,06 mg/L not shown).

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Blei / Lead [mg/L]	Abweichung [mg/L] Deviation [mg/L]	z'-Score (σ_{pt})	Hinweis Remark
1	< 0,0050			
2	0,00064	-0,0003	-0,93	
3	0,00100	0,0001	0,20	
4	0,0590			
5	0,0110			
6	0,00059	-0,0004	-1,1	
7	0,00060	-0,0003	-1,1	
8	0,00360	0,0027	8,4	
9	0,00063	-0,0003	-1,0	
10	< 0,0010			
11	0,00140	0,0005	1,5	

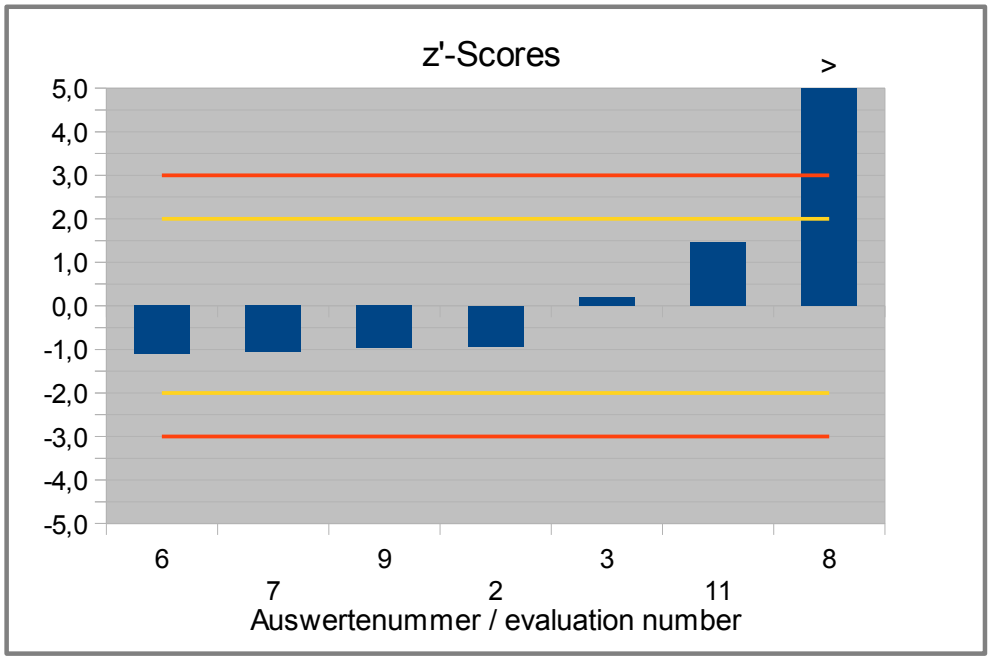

Abb. / Fig. 30: z'-Scores Lead

Summe der Eluate 1 + 2 + 3 / Sum of Eluates 1 + 2 + 3

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	6
Number of outliers	
Mean	0,0120
Median	0,00280
Robust Mean (X_{pt})	**0,00390**
Robust standard deviation (S*)	**0,00430**
Number with 3 replicates	
Repeatability SD (S_r)	
Repeatability (CV_r)	
Reproducibility SD (S_R)	
Reproducibility (CV_R)	
Target range:	
Target standard deviation σ_{pt}	
lower limit of target range	
upper limit of target range	
Quotient S/σ_{pt}*	
Standard uncertainty $U_{(Xpt)}$	
Results in the target range	
Percent in the target range	

Comment:

Due to the inconsistent distribution of results, no statistical evaluation was carried out.

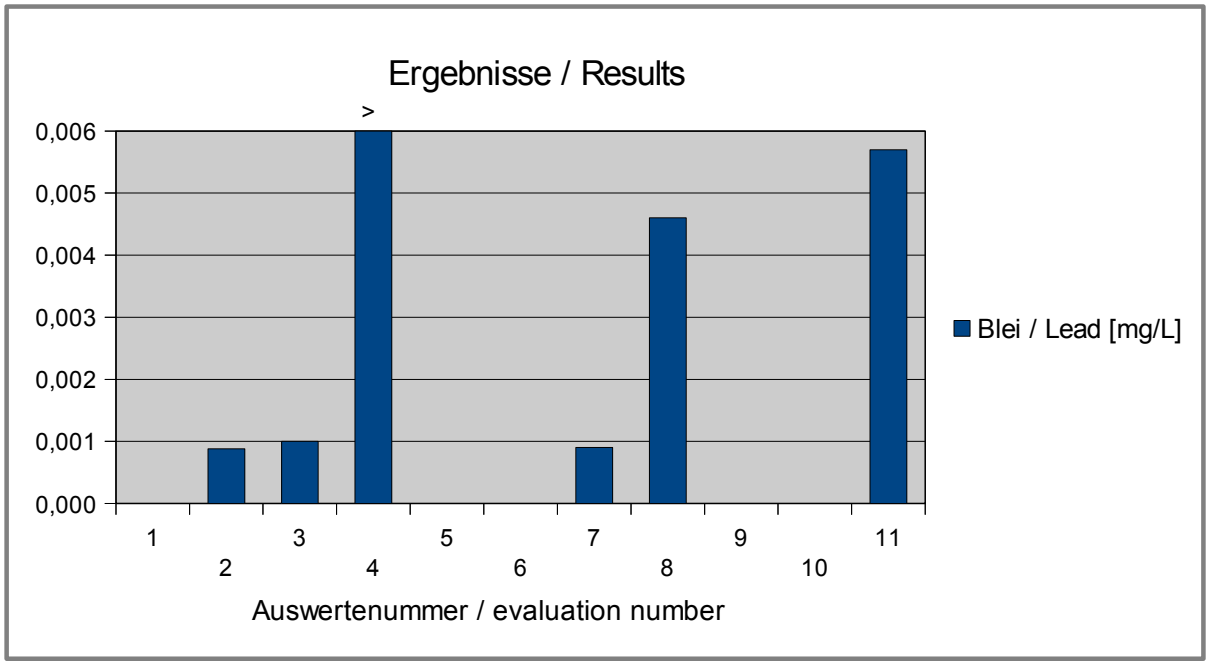

Abb. / Fig. 31: Results Lead (Sum of Eluates 1 + 2 + 3)

Comment:
A kernel density estimation was not made due to the number of < 8 results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Blei / Lead [mg/L]	Abweichung [mg/L] Deviation [mg/L]	z'-Score (σ_{pt})	Hinweis Remark
1				
2	0,00088 *			
3	0,00100 *			
4	0,0590 *			
5				
6				
7	0,0009 *			
8	0,0046 *			
9				
10				
11	0,0057 *			

* The sums of the eluates were calculated by DLA

4.7 z-Scores of the participants: overview table

Evalutaion number	Al	Al	Cr	Cr	Fe	Fe
	1st Eluate	Sum	1st Eluate	Sum	1st Eluate	Sum
1	-	-	-1,2	-	-1,2	-1,6
2	-0,86	-1,0	-0,85	-1,3	-1,6	-1,8
3	0,60	1,4	1,2	0,87	1,4	1,8
4	1,9	0,27	0,62	0,53	0,79	0,83
5	0,47	1,6	0,58	0,66	-0,67	-0,01
6	-2,0	-	-1,3	-	-1,7	-
7	-0,41	-1,1	0,30	-0,12	0,86	0,80
8	-0,76	-1,2	-0,32	-0,6	0,35	-0,02
9	-0,76	-	-0,35	-	-0,30	-
10	-0,15	-	-0,21	-	-0,03	-
11	1,9	-	2,0	-	2,8	-

Evaluation number	Mn	Mn	Ni	Ni	Pb	Pb
	1st Eluate	Sum	1st Eluate	Sum	1st Eluate	Sum
1	-0,70	-	-0,87	-1,8	-	-
2	-0,33	-0,74	-0,66	-0,81	-0,93	-
3	1,7	3,9	0,92	0,75	0,20	-
4	1,3	0,66	0,59	0,63	-	-
5	0,04	-0,05	0,32	0,88	-	-
6	-1,1	-	-0,86		-1,1	-
7	-0,02	-0,56	0,41	0,42	-1,1	-
8	-0,78	-1,2	-0,18	-0,31	8,4	-
9	-1,1	-	-0,48	-	-1,0	-
10	0,1	-	-0,45	-		-
11	1,0		1,2	-	1,5	-

5. Documentation

5.1 Details by the participants

Note: Information given in German were translated by DLA to the best of our knowledge (without guarantee of correctness).

5.1.1 Primary Data

Parameter	Auswerte-nummer	Einheit	Eluat	Abschließen-des Ergebnis	Ergebnis A	Ergebnis B	Ergebnis C	BG
Analyte	**Evaluation number**	**Unit**	**Eluate**	**Final Result**	**Result A**	**Result B**	**Result C**	**LOQ**
Al	1	mg/L	1	<	<	<	<	0,1
		mg/L	2	<	<	<	<	0,1
		mg/L	3	<	<	<	<	0,1
Al	2	mg/L	1	0,0666	0,0413	0,0585	0,1	<10µg/L
		mg/L	2	0,00727	0,0033	0,0092	0,0093	<10µg/L
		mg/L	3	0,00427	0,0027	0,0026	0,0075	<10µg/L
Al	3	mg/L	1	0,1	0,1	0,11	0,09	<0,05
		mg/L	2		0,08	0,07	0,07	<0,05
		mg/L	3		<0,05	<0,05	<0,05	<0,05
Al	4	mg/L	1	0,130	0,164	0,128	0,099	<0,03
		mg/L	2	<0,03	<0,03	<0,03	<0,03	<0,03
		mg/L	3	<0,03	<0,03	<0,03	-	<0,03
Al	5	mg/L	1	0,097	0,097	0,091	0,1	
		mg/L	2	0,045	0,05	0,044	0,042	
		mg/L	3	0,042	0,031	0,056	0,041	
Al	6	mg/L	1	0,0416866667	0,03624	0,03983	0,04899	0,1 mg/l
		mg/L	2					
		mg/L	3					
Al	7	mg/L	1	0,077	0,079	0,083	0,068	0,004
		mg/L	2	< 0,004	< 0,004	< 0,004	< 0,004	0,004
		mg/L	3	< 0,004	< 0,004	< 0,004	< 0,004	0,004
Al	8	mg/L	1	0,069	0,0778	0,055	0,0734	0,0463
		mg/L	2	0	0	0	0	0,0463
		mg/L	3	0,005	0,0033	0,0042	0,006	0,0463
Al	9	mg/L	1	0,069	0,0604	0,0793	0,0672	0,0022
		mg/L	2					
		mg/L	3					
Al	10	mg/L	1	0,083	0,09	0,103	0,055	0,05
		mg/L	2					
		mg/L	3					
Al	11	mg/L	1	0,13				0,01
		mg/L	2	0,14				0,01
		mg/L	3	0,11				0,01

* The sums of the eluates were calculated by DLA

Parameter	Auswerte-nummer	Einheit	Eluat	Abschließen-des Ergebnis	Ergebnis A	Ergebnis B	Ergebnis C	BG
Analyte	Evaluation number	Unit	Eluate	Final Result	Result A	Result B	Result C	LOQ
Cr	1	mg/L	1	0,19	0,22	0,2	0,15	0,05
		mg/L	2	<	<	<	<	0,05
		mg/L	3	<	<	<	<	0,05
Cr	2	mg/L	1	0,20933	0,174	0,228	0,226	<10µg/L
		mg/L	2	0,06193	0,0523	0,0682	0,0653	<10µg/L
		mg/L	3	0,04783	0,0477	0,0477	0,0481	<10µg/L
Cr	3	mg/L	1	0,031	0,35	0,28	0,27	<0,02
		mg/L	2		0,22	0,1	0,1	<0,02
		mg/L	3		0,043	0,035	0,037	<0,02
Cr	4	mg/L	1	0,282	0,249	0,366	0,23	<0,005
		mg/L	2	0,114	0,08	0,152	0,109	<0,005
		mg/L	3	0,067	0,046	0,087	-	<0,005
Cr	5	mg/L	1	0,28	0,3	0,26	0,29	
		mg/L	2	0,11	0,13	0,1	0,11	
		mg/L	3	0,082	0,083	0,078	0,085	
Cr	6	mg/L	1	0,1847	0,1863	0,1967	0,1711	0,020 mg/l
		mg/L	2					
		mg/L	3					
Cr	7	mg/L	1	0,266	0,3	0,251	0,246	0,0001
		mg/L	2	0,088	0,097	0,083	0,084	0,0001
		mg/L	3	0,058	0,066	0,051	0,056	0,0001
Cr	8	mg/L	1	0,2355	0,2339	0,2164	0,2562	0,018
		mg/L	2	0,077	0,0744	0,0701	0,0865	0,018
		mg/L	3	0,063	0,0612	0,0605	0,067	0,018
Cr	9	mg/L	1	0,234	0,273	0,205	0,223	0,0022
		mg/L	2					
		mg/L	3					
Cr	10	mg/L	1	0,241	0,27	0,227	0,227	0,005
		mg/L	2					
		mg/L	3					
Cr	11	mg/L	1	0,35				0,01
		mg/L	2	0,29				0,01
		mg/L	3	0,34				0,01

* The sums of the eluates were calculated by DLA

Parameter	Auswerte-nummer	Einheit	Eluat	Abschließen-des Ergebnis	Ergebnis A	Ergebnis B	Ergebnis C	BG
Analyte	Evaluation number	Unit	Eluate	Final Result	Result A	Result B	Result C	LOQ
Fe	1	mg/L	1	5	5,2	5,1	4,7	0,1
		mg/L	2	0,57	0,69	0,5	0,51	0,1
		mg/L	3	0,29	0,42	0,26	0,18	0,1
Fe	2	mg/L	1	4,69233	4,17	5,002	4,905	<100µg/L
		mg/L	2	0,55	0,47	0,617	0,563	<100µg/L
		mg/L	3	0,393	0,396	0,396	0,387	<100µg/L
Fe	3	mg/L	1	6,9	7,5	6,5	6,6	<0,1
		mg/L	2		2,2	0,7	1,33	<0,1
		mg/L	3		0,44	0,34	0,44	<0,1
Fe	4	mg/L	1	6,453	6,06	7,9	5,4	<0,01
		mg/L	2	0,940	0,57	1,32	0,93	<0,01
		mg/L	3	0,515	0,38	0,65		<0,01
Fe	5	mg/L	1	5,4	5,6	4,8	5,6	
		mg/L	2	1	1,3	0,85	0,92	
		mg/L	3	0,81	0,86	0,72	0,86	
Fe	6	mg/L	1	4,6726666667	4,845	4,683	4,49	0,025 mg/l
		mg/L	2					
		mg/L	3					
Fe	7	mg/L	1	6,5	7,25	6,1	6,15	0,01
		mg/L	2	0,874	0,921	0,848	0,853	0,01
		mg/L	3	0,511	0,608	0,46	0,464	0,01
Fe	8	mg/L	1	6,136	6,1414	5,8342	6,4319	0,034
		mg/L	2	0,629	0,5912	0,5742	0,7201	0,034
		mg/L	3	0,417	0,3906	0,392	0,4671	0,034
Fe	9	mg/L	1	5,667	5,978	5,717	5,306	0,0022
		mg/L	2					
		mg/L	3					
Fe	10	mg/L	1	5,86	6,3	6	5,29	0,01
		mg/L	2					
		mg/L	3					
Fe	11	mg/L	1	7,9				0,01
		mg/L	2	7,8				0,01
		mg/L	3	8,7				0,01

* The sums of the eluates were calculated by DLA

Parameter	Auswerte-nummer	Einheit	Eluat	Abschließen-des Ergebnis	Ergebnis A	Ergebnis B	Ergebnis C	BG
Analyte	Evaluation number	Unit	Eluate	Final Result	Result A	Result B	Result C	LOQ
Mn	1	mg/L	1	0,068	0,062	0,084	0,059	0,01
		mg/L	2	<	<	<	<	0,01
		mg/L	3	<	<	<	<	0,01
Mn	2	mg/L	1	0,07447	0,0806	0,0565	0,0863	<10µg/L
		mg/L	2	0,00843	0,0091	0,0068	0,0094	<10µg/L
		mg/L	3	0,00697	0,0071	0,0071	0,0067	<10µg/L
Mn	3	mg/L	1	0,11	0,13	0,11	0,07	<0,02
		mg/L	2		0,05	0,02	0,02	<0,02
		mg/L	3		<0,02	<0,02	<0,02	<0,02
Mn	4	mg/L	1	0,103	0,091	0,139	0,079	<0,005
		mg/L	2	0,014	0,007	0,02	0,014	<0,005
		mg/L	3	0,006	0,004	0,008	-	<0,005
Mn	5	mg/L	1	0,081	0,094	0,088	0,063	
		mg/L	2	0,014	0,018	0,014	0,0098	
		mg/L	3	0,011	0,011	0,012	0,0089	
Mn	6	mg/L	1	0,0613866667	0,04852	0,07146	0,06418	0,010 mg/l
		mg/L	2					
		mg/L	3					
Mn	7	mg/L	1	0,08	0,091	0,077	0,071	0,001
		mg/L	2	0,009	0,01	0,01	0,008	0,001
		mg/L	3	0,005	0,006	0,005	0,005	0,001
Mn	8	mg/L	1	0,0665	0,0558	0,0682	0,0754	0,017
		mg/L	2	0,0068	0,0056	0,007	0,0077	0,017
		mg/L	3	0,0047	0,0037	0,0051	0,0054	0,017
Mn	9	mg/L	1	0,061	0,0566	0,0507	0,0757	0,0022
		mg/L	2					
		mg/L	3					
Mn	10	mg/L	1	0,082	0,098	0,091	0,058	0,01
		mg/L	2					
		mg/L	3					
Mn	11	mg/L	1	0,098				0,01
		mg/L	2	0,13				0,01
		mg/L	3	0,14				0,01

* The sums of the eluates were calculated by DLA

Parameter	Auswerte-nummer	Einheit	Eluat	Abschließen-des Ergebnis	Ergebnis A	Ergebnis B	Ergebnis C	BG
Analyte	Evaluation number	Unit	Eluate	Final Result	Result A	Result B	Result C	LOQ
Ni	1	mg/L	1	0,13	0,16	0,13	0,1	0,01
		mg/L	2	<0,011	<	0,012	<	0,01
		mg/L	3	<	<	<	<	0,01
Ni	2	mg/L	1	0,13697	0,0959	0,16	0,155	<10µg/L
		mg/L	2	0,02163	0,0152	0,025	0,0247	<10µg/L
		mg/L	3	0,01307	0,0114	0,0114	0,0164	<10µg/L
Ni	3	mg/L	1	0,19	0,2	0,16	0,19	<0,02
		mg/L	2		0,1	0,03	0,03	<0,02
		mg/L	3		<0,02	<0,02	<0,02	<0,02
Ni	4	mg/L	1	0,179	0,148	0,24	0,148	<0,005
		mg/L	2	0,033	0,018	0,045	0,037	<0,005
		mg/L	3	0,020	0,011	0,028	-	<0,005
Ni	5	mg/L	1	0,17	0,2	0,14	0,19	
		mg/L	2	0,038	0,048	0,031	0,036	
		mg/L	3	0,034	0,028	0,043	0,031	
Ni	6	mg/L	1	0,242	0,1409	0,1312	0,1187	0,010 mg/l
		mg/L	2					
		mg/L	3					
Ni	7	mg/L	1	0,173	0,195	0,161	0,164	0,001
		mg/L	2	0,031	0,033	0,029	0,03	0,001
		mg/L	3	0,019	0,022	0,016	0,018	0,001
Ni	8	mg/L	1	0,1532	0,1401	0,1494	0,1702	0,0084
		mg/L	2	0,0237	0,0216	0,0215	0,0278	0,0084
		mg/L	3	0,0156	0,0137	0,0156	0,0176	0,0084
Ni	9	mg/L	1	0,143	0,176	0,114	0,138	0,0022
		mg/L	2					
		mg/L	3					
Ni	10	mg/L	1	0,144	0,155	0,142	0,134	0,002
		mg/L	2					
		mg/L	3					
Ni	11	mg/L	1	0,201				0,001
		mg/L	2	0,19				0,001
		mg/L	3	0,201				0,001

* The sums of the eluates were calculated by DLA

Parameter	Auswerte-nummer	Einheit	Eluat	Abschließen-des Ergebnis	Ergebnis A	Ergebnis B	Ergebnis C	BG
Analyte	Evaluation number	UPbt	Eluate	Final Result	Result A	Result B	Result C	LOQ
Pb	1	mg/L	1	<	<	<	<	0,005
		mg/L	2	<	<	<	<	0,005
		mg/L	3	<	<	<	<	0,005
Pb	2	mg/L	1	0,00064	0,00078	0,00072	0,00041	<2µg/L
		mg/L	2	0,00016	0,00011	0,00006	0,00032	<2µg/L
		mg/L	3	0,00008	0,00003	0,00003	0,00018	<2µg/L
Pb	3	mg/L	1	0,001	0,001	0,001	0,001	<0,001
		mg/L	2		<0,001	<0,001	<0,001	<0,001
		mg/L	3		<0,001	<0,001	<0,001	<0,001
Pb	4	mg/L	1	0,059	0,051	0,062	0,064	<0,03
		mg/L	2	<0,03	<0,03	<0,03	<0,03	<0,03
		mg/L	3	<0,03	<0,03	<0,03	-	<0,03
Pb	5	mg/L	1	0,011	0,01	0,011	0,012	
		mg/L	2					
		mg/L	3					
Pb	6	mg/L	1	0,0005866667	0,00051	0,00073	0,00052	0,0005 mg/l
		mg/L	2					
		mg/L	3					
Pb	7	mg/L	1	0,0006	0,0008	0,0006	0,0004	0,0001
		mg/L	2	0,0002	0,0002	0,0001	< 0,0001	0,0001
		mg/L	3	0,0001	0,0001	0,0001	0,0001	0,0001
Pb	8	mg/L	1	0,0036	0,0019	0,0007	0,0019	0,00388
		mg/L	2	0,0006	0,001	0,0007	0,0002	0,00388
		mg/L	3	0,0004	0,0005	0,0002	0,0004	0,00388
Pb	9	mg/L	1	0,00063	0,00048	0,00035	0,00105	0,0002
		mg/L	2					
		mg/L	3					
Pb	10	mg/L	1	<0,001	<0,001	<0,002	<0,003	0,001
		mg/L	2					
		mg/L	3					
Pb	11	mg/L	1	0,0014				0,001
		mg/L	2	0,0014				0,001
		mg/L	3	0,0029				0,001

* The sums of the eluates were calculated by DLA

5.1.2 Analytical Methods

Parameter	Auswerte-nummer	Eluat	Methodenbeschreibung, wie in einem regulären Prüfbericht angegeben	Vorbehandlung des Gegenstands	Simulanzmittel Citronensäure 5 g/L	Füllvolumen 20 mL	Zeit und Temperatur: 2 h bei 70°C + 24 h bei 40°C	Hinweise zur Analytik	Methode ist akkreditiert	Sonstige Hinweise
Analyte	Evaluation number	Eluate	DesAliption of the methods like in a report analysis	Pre conditioning of material	Simulant citric acid 5 g/L	Filling volume 20 mL	Time and temperature: 2 h at 70°C + 24 h at 40°C	Remarks to analysis	Method acAledited	Further Remarks
Al	1	1	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		2	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		3	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
Al	2	1			yes	yes	yes		no	
		2			yes	yes	yes		no	
		3			yes	yes	yes		no	
Al	3	1	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		2	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		3	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
Al	4	1	The test was based on the Council of Europe Resolution on metals and alloys used in food contact materials (CM / Res (2013) 9).	no	yes	yes	yes	ICP-OES	no	
		2		no	yes	yes	yes	ICP-OES	no	
		3		no	yes	yes	yes	ICP-OES	no	
Al	5	1	CM/Res (2013) 9		yes	yes	yes		no	
		2	CM/Res (2013) 9		yes	yes	yes		no	
		3	CM/Res (2013) 9		yes	yes	yes		no	
Al	6	1	Migration metals and alloys - MET 232	cleaning with RBS soap	yes	yes	yes	ICP-OES	yes	result A < sample 1, result B < sample 2; result C < sample 3; all measured in the simulant after (2h-70 °C + 24h-40°C)
		2								
		3								
Al	7	1	Council of Europe Resolution CM/Res (2013) 9 on metals and alloys used in food contact materials (Fassung vom 11. Juni 2013)	no	yes	yes	yes	Analysis: 23.10.2019	yes	22 - 23.10.2019
		2	"	no	yes	yes	yes	Analysis: 24.10.2019	yes	23 - 24.10.2019
		3	"	no	yes	yes	yes	Analysis: 25.10.2019	yes	24 - 25.10.2019
Al	8	1		normal household cleaning	yes	yes	yes		no	
		2		normal household cleaning	yes	yes	yes		no	
		3		normal household cleaning	yes	yes	yes		no	
Al	9	1	DIN EN ISO 17294-2	rinsing	yes	yes	yes	ICP-MS	yes	
		2	2005-02		-	-	-		-	
		3	Water quality - Application of inductively coupled plasma mass spectrometry (ICP-MS) - Part 2: Determination of 62 elements Deviation: other matrices: digestion and migration of commodities, additional analytes: mercury and iron		-	-	-		-	
Al	10	1	CM/Res 2013/9	pre-rinsed according to CM/Res	yes	yes	yes	ICP-MS	yes	
		2			-	-	-		-	
		3			-	-	-		-	
Al	11	1			yes	yes	yes		no	
		2			yes	yes	yes		no	
		3			yes	yes	yes		no	

Parameter	Auswerten ummer	Eluat	Methodenbeschreibung, wie in einem regulären Prüfbericht angegeben	Vorbehandlung des Gegenstands	Simulanzmittel Citronensäure 5 g/L	Füllvolumen 20 mL	Zeit und Temperatur: 2 h bei 70°C + 24 h bei 40°C	Hinweise zur Analytik	Methode ist akkreditiert	Sonstige Hinweise
Analyte	Evaluation number	Eluate	Description of the methods like in a report analysis	Pre conditioning of material	Simulant citric acid 5 g/L	Filling volume 20 mL	Time and temperature: 2 h at 70°C + 24 h at 40°C	Remarks to analysis	Method accredited	Further Remarks
Cr	1	1	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		2	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		3	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
Cr	2	1			yes	yes	yes		no	
		2			yes	yes	yes		no	
		3			yes	yes	yes		no	
Cr	3	1	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		2	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		3	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
Cr	4	1		no	yes	yes	yes	ICP-OES	no	
		2		no	yes	yes	yes	ICP-OES	no	
		3		no	yes	yes	yes	ICP-OES	no	
Cr	5	1	CM/Res (2013) 9		yes	yes	yes		no	
		2	CM/Res (2013) 9		yes	yes	yes		no	
		3	CM/Res (2013) 9		yes	yes	yes		no	
Cr	6	1	Migration metals and alloys - MET 232	cleaning with RBS soap	yes	yes	yes	ICP-OES	yes	
		2								
		3								
Cr	7	1	"	no	yes	yes	yes	Analysis: 23.10.2019	yes	22 - 23.10.2019
		2	"	no	yes	yes	yes	Analysis: 24.10.2019	yes	23 - 24.10.2019
		3	"	no	yes	yes	yes	Analysis: 25.10.2019	yes	24 - 25.10.2019
Cr	8	1		normal household cleaning	yes	yes	yes		no	
		2		normal household cleaning	yes	yes	yes		no	
		3		normal household cleaning	yes	yes	yes		no	
Cr	9	1			-	-	-		-	
		2			-	-	-		-	
		3			-	-	-		-	
Cr	10	1	CM/Res 2013/9	pre-rinsed according to CM/Res	yes	yes	yes	ICP-MS	yes	
		2			-	-	-		-	
		3			-	-	-		-	
Cr	11	1			yes	yes	yes		yes	
		2			yes	yes	yes		yes	
		3			yes	yes	yes		yes	

Parameter	Auswerten ummer	Eluat	Methodenbeschreibung, wie in einem regulären Prüfbericht angegeben	Vorbehandlung des Gegenstands	Simulanzmittel Citronensäure 5 g/L	Füllvolumen 20 mL	Zeit und Temperatur: 2 h bei 70°C + 24 h bei 40°C	Hinweise zur Analytik	Methode ist akkreditiert	Sonstige Hinweise
Analyte	Evaluation number	Eluate	DesFeiption of the methods like in a report analysis	Pre conditioning of material	Simulant citric acid 5 g/L	Filling volume 20 mL	Time and temperature: 2 h at 70°C + 24 h at 40°C	Remarks to analysis	Method acFeedited	Further Remarks
Fe	1	1	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		2	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		3	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
Fe	2	1			yes	yes	yes		no	
		2			yes	yes	yes		no	
		3			yes	yes	yes		no	
Fe	3	1	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		2	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		3	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
Fe	4	1		no	yes	yes	yes	ICP-OES	no	
		2		no	yes	yes	yes	ICP-OES	no	
		3		no	yes	yes	yes	ICP-OES	no	
Fe	5	1	CM/Res (2013) 9		yes	yes	yes		no	
		2	CM/Res (2013) 9		yes	yes	yes		no	
		3	CM/Res (2013) 9		yes	yes	yes		no	
Fe	6	1	Migration metals and alloys - MET 232	cleaning with RBS soap	yes	yes	yes	ICP-OES	no	
		2								
		3								
Fe	7	1	"	no	yes	yes	yes	Analysis: 23.10.2019	yes	22 - 23.10.2019
		2	"	no	yes	yes	yes	Analysis: 24.10.2019	yes	23 - 24.10.2019
		3	"	no	yes	yes	yes	Analysis: 25.10.2019	yes	24 - 25.10.2019
Fe	8	1		normal household cleaning	yes	yes	yes		no	
		2		normal household cleaning	yes	yes	yes		no	
		3		normal household cleaning	yes	yes	yes		no	
Fe	9	1			-	-	-		-	
		2			-	-	-		-	
		3			-	-	-		-	
Fe	10	1	CM/Res 2013/9	pre-rinsed according to CM/Res	yes	yes	yes	ICP-MS	yes	
		2			-	-	-		-	
		3			-	-	-		-	
Fe	11	1			yes	yes	yes		no	
		2			yes	yes	yes		no	
		3			yes	yes	yes		no	

Parameter	Auswerte-nummer	Eluat	Methodenbeschreibung, wie in einem regulären Prüfbericht angegeben	Vorbehandlung des Gegenstands	Simulanzmittel Citronensäure 5 g/L	Füllvolumen 20 mL	Zeit und Temperatur: 2 h bei 70°C + 24 h bei 40°C	Hinweise zur Analytik	Methode ist akkreditiert	Sonstige Hinweise
Analyte	Evaluation number	Eluate	DesMniption of the methods like in a report analysis	Pre conditioning of material	Simulant citric acid 5 g/L	Filling volume 20 mL	Time and temperature: 2 h at 70°C + 24 h at 40°C	Remarks to analysis	Method acMnedited	Further Remarks
Mn	1	1	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		2	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		3	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
Mn	2	1			yes	yes	yes		no	
		2			yes	yes	yes		no	
		3			yes	yes	yes		no	
Mn	3	1	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		2	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		3	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
Mn	4	1		no	yes	yes	yes	ICP-OES	no	
		2		no	yes	yes	yes	ICP-OES	no	
		3		no	yes	yes	yes	ICP-OES	no	
Mn	5	1	CM/Res (2013) 9		yes	yes	yes		no	
		2	CM/Res (2013) 9		yes	yes	yes		no	
		3	CM/Res (2013) 9		yes	yes	yes		no	
Mn	6	1	Migration metals and alloys - MET 232	cleaning with RBS soap	yes	yes	yes	ICP-OES	no	
		2								
		3								
Mn	7	1	"	no	yes	yes	yes	Analysis: 23.10.2019	yes	22 - 23.10.2019
		2	"	no	yes	yes	yes	Analysis: 24.10.2019	yes	23 - 24.10.2019
		3	"	no	yes	yes	yes	Analysis: 25.10.2019	yes	24 - 25.10.2019
Mn	8	1		normal household cleaning	yes	yes	yes		no	
		2		normal household cleaning	yes	yes	yes		no	
		3		normal household cleaning	yes	yes	yes		no	
Mn	9	1			-	-	-		-	
		2			-	-	-		-	
		3			-	-	-		-	
Mn	10	1	CM/Res 2013/9	pre-rinsed according to CM/Res	yes	yes	yes	ICP-MS	yes	
		2			-	-	-		-	
		3			-	-	-		-	
Mn	11	1			yes	yes	yes		yes	
		2			yes	yes	yes		yes	
		3			yes	yes	yes		yes	

Parameter	Auswerte-nummer	Eluat	Methodenbeschreibung, wie in einem regulären Prüfbericht angegeben	Vorbehandlung des Gegenstands	Simulanzmittel Citronensäure 5 g/L	Füllvolumen 20 mL	Zeit und Temperatur: 2 h bei 70°C + 24 h bei 40°C	Hinweise zur Analytik	Methode ist akkreditiert	Sonstige Hinweise
Analyte	Evaluation number	Eluate	DesNiiption of the methods like in a report analysis	Pre conditioning of material	Simulant citric acid 5 g/L	Filling volume 20 mL	Time and temperature: 2 h at 70°C + 24 h at 40°C	Remarks to analysis	Method acNiedited	Further Remarks
Ni	1	1	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		2	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		3	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
Ni	2	1			yes	yes	yes		no	
		2			yes	yes	yes		no	
		3			yes	yes	yes		no	
Ni	3	1	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		2	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		3	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
Ni	4	1		no	yes	yes	yes	ICP-OES	no	
		2		no	yes	yes	yes	ICP-OES	no	
		3		no	yes	yes	yes	ICP-OES	no	
Ni	5	1	CM/Res (2013) 9		yes	yes	yes		no	
		2	CM/Res (2013) 9		yes	yes	yes		no	
		3	CM/Res (2013) 9		yes	yes	yes		no	
Ni	6	1	Migration metals and alloys - MET 232	cleaning with RBS soap	yes	yes	yes	ICP-OES	yes	
		2								
		3								
Ni	7	1	"	no	yes	yes	yes	Analysis: 23.10.2019	yes	22 - 23.10.2019
		2	"	no	yes	yes	yes	Analysis: 24.10.2019	yes	23 - 24.10.2019
		3	"	no	yes	yes	yes	Analysis: 25.10.2019	yes	24 - 25.10.2019
Ni	8	1		normal household cleaning	yes	yes	yes		no	
		2		normal household cleaning	yes	yes	yes		no	
		3		normal household cleaning	yes	yes	yes		no	
Ni	9	1			-	-	-		-	
		2			-	-	-		-	
		3			-	-	-		-	
Ni	10	1	CM/Res 2013/9	pre-rinsed according to CM/Res	yes	yes	yes	ICP-MS	yes	
		2			-	-	-		-	
		3			-	-	-		-	
Ni	11	1			yes	yes	yes		yes	
		2			yes	yes	yes		yes	
		3			yes	yes	yes		yes	

Parameter	Auswerte-nummer	Eluat	Methodenbeschreibung, wie in einem regulären Prüfbericht angegeben	Vorbehandlung des Gegenstands	Simulanzmittel Citronensäure 5 g/L	Füllvolumen 20 mL	Zeit und Temperatur: 2 h bei 70°C + 24 h bei 40°C	Hinweise zur Analytik	Methode ist akkreditiert	Sonstige Hinweise
Analyte	Evaluation number	Eluate	DesPbiption of the methods like in a report analysis	Pre conditioPbng of material	Simulant citric acid 5 g/L	Filling volume 20 mL	Time and temperature: 2 h at 70°C + 24 h at 40°C	Remarks to analysis	Method acPbedited	Further Remarks
Pb	1	1	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		2	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
		3	DIN EN 13130-1: 2004-08 /ICP-MS: DIN EN ISO 17294-2: 2017-01*	The sample was pre-cleaned with warm water and detergent and between each migration cycle with dist. H2O rinsed and dried	yes	yes	yes		no	
Pb	2	1			yes	yes	yes		no	
		2			yes	yes	yes		no	
		3			yes	yes	yes		no	
Pb	3	1	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		2	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
		3	according to CM/RES((2013)9)	Pre-rinsed	yes	yes	yes		yes	
Pb	4	1		no	yes	yes	yes	ICP-OES	no	
		2		no	yes	yes	yes	ICP-OES	no	
		3		no	yes	yes	yes	ICP-OES	no	
Pb	5	1	CM/Res (2013) 9		yes	yes	yes		no	
		2	CM/Res (2013) 9		yes	yes	yes		no	
		3	CM/Res (2013) 9		yes	yes	yes		no	
Pb	6	1	Migration metals and alloys - MET 232	cleaning with RBS soap	yes	yes	yes	ICP-MS	yes	
		2								
		3								
Pb	7	1	"	no	yes	yes	yes	Analysis: 23.10.2019	yes	22 - 23.10.2019
		2	"	no	yes	yes	yes	Analysis: 24.10.2019	yes	23 - 24.10.2019
		3	"	no	yes	yes	yes	Analysis: 25.10.2019	yes	24 - 25.10.2019
Pb	8	1		normal household cleaning	yes	yes	yes		no	
		2		normal household cleaning	yes	yes	yes		no	
		3		normal household cleaning	yes	yes	yes		no	
Pb	9	1			-	-	-		-	
		2			-	-	-		-	
		3			-	-	-		-	
Pb	10	1	CM/Res 2013/9	pre-rinsed according to CM/Res	yes	yes	yes	ICP-MS	yes	
		2			-	-	-		-	
		3			-	-	-		-	
Pb	11	1			yes	yes	yes		no	
		2			yes	yes	yes		no	
		3			yes	yes	yes		no	

5.2 Homogeneity

5.2.1 Homogeneity examination of sample material

The homogeneity of the sample materials were examined for chromium and nickel by ICP-MS:

Chromium

Repeated Measurements	mg/L
1	0,31
2	0,33
3	0,28
4	0,27
5	0,35

General Mean	0,308	
Repeatability standard deviation	0,0335	10,9%

Nickel

Repeated Measurements	mg/L
1	0,19
2	0,20
3	0,20
4	0,19
5	0,16

General Mean	0,188	
Repeatability standard deviation	0,0164	8,7%

5.3 Information on the Proficiency Test (PT)

Before the PT the participants received the following information in the sample cover letter:

PT number	***DLA 55-2019***
PT name	***Contact Material I: Release of Elements from Food Contact Material***
*Sample matrix**	*Samples A, B + C: stainless steel jugs*
Number of samples and sample amount	*3 identical samples A, B + C: 0.08 liters*
Storage	*Samples A, B + C: room temperature*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative: Aluminium, Lead, Chromium, Iron, Manganese and Nickel*
Methods of analysis	*The **below conditions** according to the Resolution of the Council of Europe on metals and alloys used in food contact materials (CM/Res(2013)9) for "hot fills" **should be followed**:* *1. filling volume: 60 mL* *2. simulant to be used: citric acid 5 g/L (in water),* *3. time and temperature: 2 h at 70°C, afterwards 24 h at 40°C,* *4. results given in mg/L.*
Notes to analysis	*The analysis of PT samples should be performed like a routine laboratory analysis.*
Result sheet	*The results for the eluates of sample A, B and C as well as the final results calculated as means should be filled in the result submission file. The results of the first eluate will be evaluated. If there are enough results of the second and third eluate, the sum of eluates will be evaluated.*
Units	*mg/L*
Number of significant digits	*at least 2*
Further information	*For information please specify:* – *Date of analysis* – *Limit of detection* – *Method is accredited*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Deadline	***the latest 15th November 2019***
Evaluation report	*The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.*
Coordinator and contact person of PT	*Matthias Besler-Scharf PhD*

* Control of mixture homogeneity and qualitative testings are carried out by DLA. Any testing of the content, homogeneity and stability of PT parameters is subcontracted by DLA.

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		Germany
		BELGIUM
		Germany
		Germany
		Germany
		Germany
		Germany
		ITALY
		ITALY
		Germany
		Germany

[Die Adressdaten der Teilnehmer wurden für die allgemeine Veröffentlichung des Auswerte-Berichts nicht angegeben.]

[The address data of the participants were deleted for publication of the evaluation report.]

7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by inter-laboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methoden-validierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and preci-sion) of measurement methods and results
5. Verordnung / Regulation 882/2004/EU; Verordnung über über amtliche Kontrollen zur Überprüfung der Einhaltung des Lebensmittel- und Futtermittelrechts sowie der Bestimmungen über Tiergesundheit und Tierschutz / Regulation on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
6. Evaluation of analytical methods used for regulation of food and drugs; W. Hor-witz; Analytical Chemistry, 54, 67-76 (1982)
7. The International Harmonised Protocol for the Proficiency Testing of Ananlytical Laboratories ; J.AOAC Int., 76(4), 926 – 940 (1993)
8. A Horwitz-like funktion describes precision in proficiency test; M. Thompson, P.J. Lowthian; Analyst, 120, 271-272 (1995)
9. Protocol for the design, conduct and interpretation of method performance studies; W. Horwitz; Pure & Applied Chemistry, 67, 331-343 (1995)
10. Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing; M. Thompson; Ana-lyst, 125, 385-386 (2000)
11. The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories; Pure Appl Chem, 78, 145 – 196 (2006)
12. AMC Kernel Density - Representing data distributions with kernel density estim-ates, amc technical brief, Editor M Thompson, Analytical Methods Committee, AMCTB No 4, Revised March 2006 and Excel Add-in Kernel.xla 1.0e by Royal Society of Chemistry
13. EURACHEM/CITAC Leitfaden, Ermittlung der Messunsicherheit bei analytischen Messun-gen (2003); Quantifying Uncertainty in Analytical Measurement (1999)
14. GMP+ Feed Certification scheme, Module: Feed Safety Assurance, chapter 5.7 Check-ing procedure for the process accuracy of compound feed with micro tracers in GMP+ BA2 Control of residues, Version: 1st of January 2015 GMP+ International B.V.
15. MTSE SOP No. 010.01 (2014): Quantitative measurement of mixing uniformity and carry-over in powder mixtures with the rotary detector technique, MTSE Micro Tracers Services Europe GmbH
16. Homogeneity and stability of reference materials; Linsinger et al.; Accred Qual Assur, 6, 20-25 (2001)
17. AOAC Official Methods of Analysis: Guidelines for Standard Method Performance Re-quirements, Appendix F, p. 2, AOAC Int (2016)
18. Council of Europe Resolution CM/Res(2013)9: Metals and alloys used in food contact materials and articles, 1st Edition, Committee of Experts on Packaging Materials for Food and Pharmaceutical Products (P-SC-EMB), published by Directorate for the Quality of Medicines & HealthCare, Council of Europe (2013)
19. ASU §64 L 59.11-28: Bestimmung von Bor, Barium, Calcium, Eisen, Kalium, Magnesium, Mangan, Natrium und Strontium in Mineralwasser mittels optischer Emissionsspektro-metrie mit induktiv gekoppeltem Plasma (ICP-OES)
20. DIN EN ISO 11885:2009-09: Wasserbeschaffenheit - Bestimmung von ausgewählten Ele-menten durch induktiv gekoppelte Plasma-Atom-Emissionsspektrometrie (ICP-OES) / Water quality -Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)