DLA
Dienstleistung
Lebensmittel
Analytik GbR

Evaluation-Report
proficiency test

DLA 31/2014

Elements in mineral food supplements

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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 validity of the particular testing method.
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.

2 Evaluation

2.1 Assigned value

Because the analysed material was no certified reference material the robust mean of the submitted results was used as assigned value X. The distribution of submitted results showed no hint for bimodal distribution or other reasons for a higher variability.

2.2 Standard deviation

For comparison to the target standard deviation a robust standard deviation (Sx) was calculated.

2.3 Outliers

Statistical outliers were determined by Mandel´s-H-Statistic (95% significance). Detected outliers were stated for information only, when z-score was < -2 or > 2.

2.4 Target standard deviation

The target standard deviation of the assigned value is determined according to the following methods.

2.4.1 General model (Horwitz / Thompson)

The relative target standard deviation in % of the assigned value was derived from following equation (Horwitz)

$$\sigma_{(\%)} = 2^{(1-0,5\log X)} .$$

From the result the target standard deviation was calculated

$$\sigma = X * \sigma_{(\%)} / 100.$$

For analytes with a content below 120 µg/kg after the evaluation of a lot of mycotoxin- proficiency testing schemes after 1997 it was suggested for the target standard deviation a steady value of 22 % (Thompson), analogical:

$$\sigma = 0,22\ C / mr;$$

with σ = Target standard deviation for contents < 120 µg/kg
C = assigned content, expressed as a dimensionless mass ratio
mr = dimensionless mass ratio.

2.4.2 Precision experiment

Using the reproducibility standard deviation σ_R and the repeatability standard deviation σ_r of a precision experiment the between-laboratories standard deviation (σ_L) can be calculated :

$$\sigma_L = \sqrt{(\sigma_R^2 - \sigma_r^2)} .$$

And then, using the number of replicate measurements n, each participant is to perform, the standard deviation for proficiency assessment is calculated :

$$\sigma = \sqrt{(\sigma_L^2 + (\sigma_r^2 / n))} .$$

If available, the precision data from official methods for each parameter are used to calculate the target standard deviation.

2.5 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) the result (x) of the participant is deviating from the assigned value (X).

Participants' z-scores are derived as:

$$z = (x - X) / \sigma \quad ;$$

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

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

2.6 z'-Score

The z'-Score can be used to assess the results of the participants in case the standard uncertainty must be considered (s. 2.8).

The calculation is carried out as follows (3)

For the following evaluation is defined as $\hat{\sigma}'$, the target standard deviation considering the standard uncertainty of the results.

The requirements for the analytical performance are considered as fulfilled then, if

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

2.7 Quotient S^x/σ

Following the Horrat-value the results of a proficiency-test (PT) can be considered convincing, if the quotient of robust standard deviation and target standard deviation 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.

2.8 Standard uncertainty

The assigned value X has a standard uncertainty u_X that depends on the analytical method, differences between the analytical methods used, the

test material, the number of participant laboratories and perhaps on other factors. The standard uncertainty u_X for this PT is calculated as follows

$$u_X = 1{,}25 * S^x / \sqrt{(p)} \quad .$$

If $u_X \leq 0{,}3 * \sigma$ the standard uncertainty of the assigned value needs not be included in the interpretation of the results of the PT. The quotient u_X / σ is given in the evaluation.

3 Realisation

3.1 Test material

The test material was a mineral food supplement with only one, ground ingredients which is on the market.
App. 1 kg of the material was intensively mixed and put in portions of app. 3 gram. The portions where numbered chronologically.

3.1.1 Homogeneity

The calculation of the repeatability standard deviation of the participants for Al, Ca, Fe, Co, Mn, and V was used as an indicator of homogeneity. The results are from 3,1 % (Fe) to 12,2 % (Co) and they are the same magnitude as specified in the relevant official German methods to determine elements. The repeatability standard deviation of the participants is given in the documentation.

3.2 Tests

Two test samples were sent to every participating laboratory in the 20th week of 2014. The test method was optional. The tests should be finished at 27.06.2014. Two participants got a third sample on request because the amound was not sufficient for several different methods.

3.3 Results and statistic evaluation

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

The statistical evaluation was carried out if at least 7 results were submitted.
According to 2.4.1 of this report were evaluated: Ba, Cr, Li, Mo, V.
According to 2.4.1 and 2.6 of this report were evaluated: Al, Co, Ni, U.
According 2.4.2 of this report were evaluated (ASU L 00.00-144): Ca, Fe, Mg, Mn.
According 2.4.2 and 2.6 of this report were evaluated (ASU L 00.00-144): Cu, Na, P, Zn.
According 2.4.2 of this report was evaluated (ASU L 00.00-93): I.
Results for Y are given in part 4 of this report.
Results for Nd are given in the documentation.
Queried and documented were further results and the testing method applied.
23 out of 25 participants submitted results in time.

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 upper table the characteristics are listed:

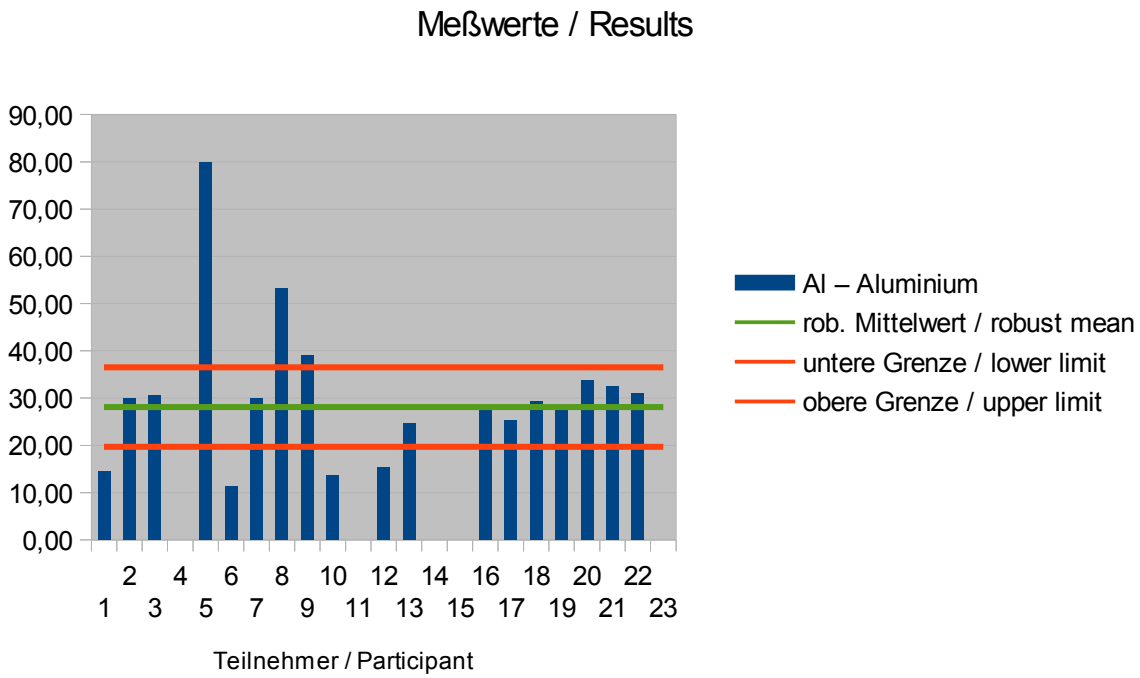
number of the results
number of outliers
mean
median
robust mean (X)
robust standard deviation(S^x)
target standard deviation (σ) or (σ´)
target standard deviation (for information)
lower limit of target range (X – 2σ) or (X – 2σ´)
upper limit of target range (X + 2σ) or (X + 2σ´)
quotient S^x/σ
standard uncertainty u_x
quotient u_X/σ
results in target range.

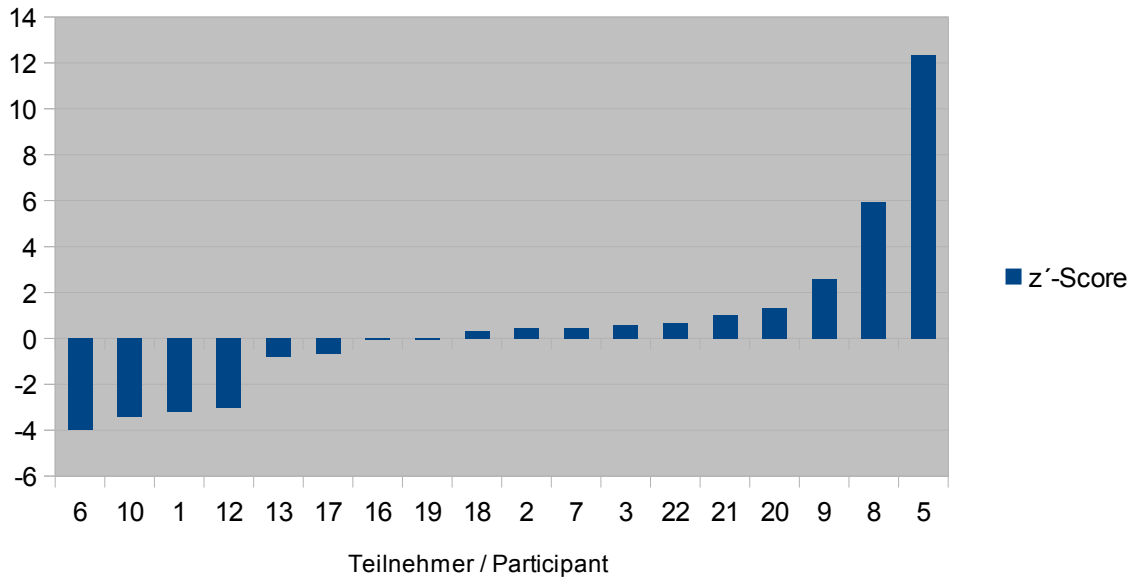
In the lower table -laboratories- the individual results of the participating laboratory are listed:

evaluation number	test result	deviation from assigned value	Z-Score (σ)	remarks

4.1 Aluminium in mg/kg

Statistic Data	
number of the results	18
number of outliers	1
mean	30,5
median	29,7
robust mean (X)	28,1
robust standard deviation (S^x)	10,9
target standard deviation (sigma')	4,2
target standard deviation for information (sigma)	2,72
lower limit of target range	19,7
upper limit of target range	36,5
quotient S^x/σ	4,0
standard uncertainty U^x	3,20
quotient U^x/σ	1,2
results in target range	11
percent in target range	61

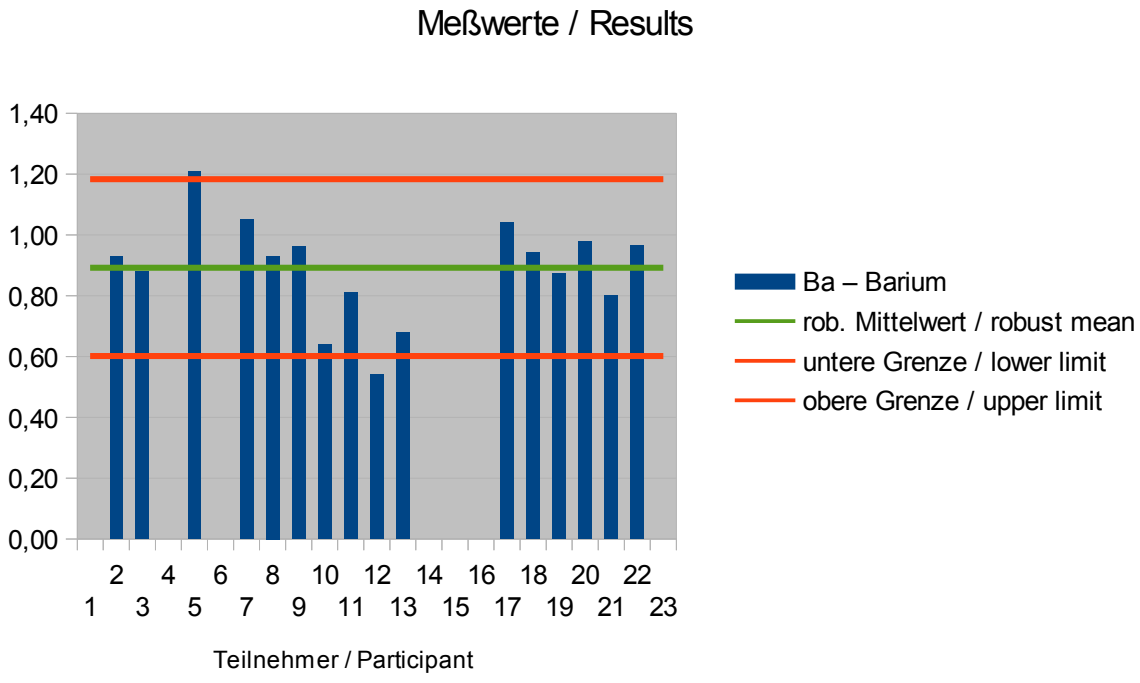


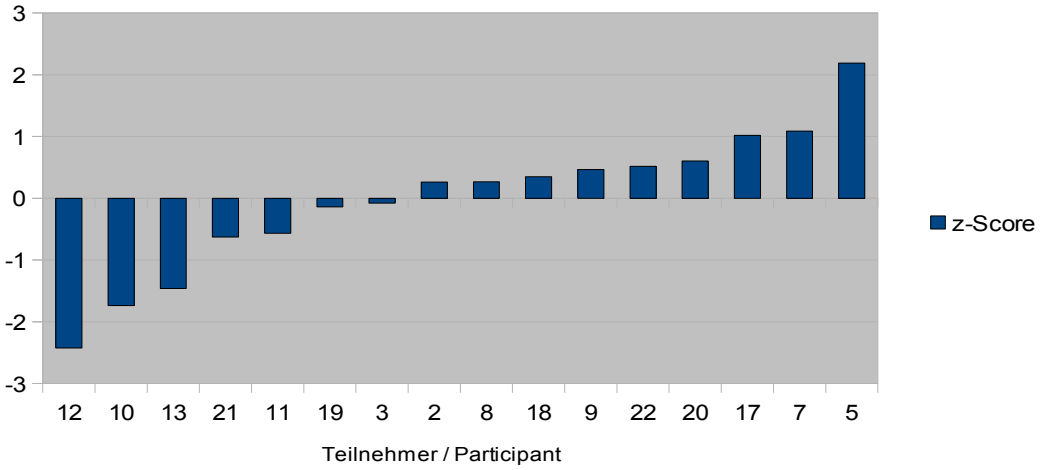


Auswerte nummer / Evaluation number	Al - Aluminium	Abweichung / Deviation	z´-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	14,58	-13,51	-3,2	-5,0	
2	30	1,91	0,5	0,7	
3	30,5	2,41	0,6	0,9	
4					
5	80	51,91	12,4	19,1	Ausreisser / Outlier
6	11,4	-16,69	-4,0	-6,1	
7	30	1,91	0,5	0,7	
8	53,13	25,05	6,0	9,2	
9	39	10,91	2,6	4,0	
10	13,7	-14,39	-3,4	-5,3	
11					
12	15,3	-12,79	-3,0	-4,7	
13	24,7	-3,39	-0,8	-1,2	
14					
15					
16	27,8	-0,29	-0,1	-0,1	
17	25,19	-2,9	-0,7	-1,1	
18	29,4	1,31	0,3	0,5	
19	27,8	-0,29	-0,1	-0,1	
20	33,7	5,61	1,3	2,1	
21	32,41	4,32	1,0	1,6	
22	30,9	2,81	0,7	1,0	
23					

4.2 Barium in mg/kg

Statistic Data	
number of the results	16
number of outliers	2
mean	0,89
median	0,93
robust mean (X)	0,89
robust standard deviation (S^x)	0,16
target standard deviation for information	0,15
lower limit of target range	0,60
upper limit of target range	1,18
quotient S^x/σ	1,1
standard uncertainty U^x	0,05
quotient U^x/σ	0,4
results in target range	14
percent in target range	88

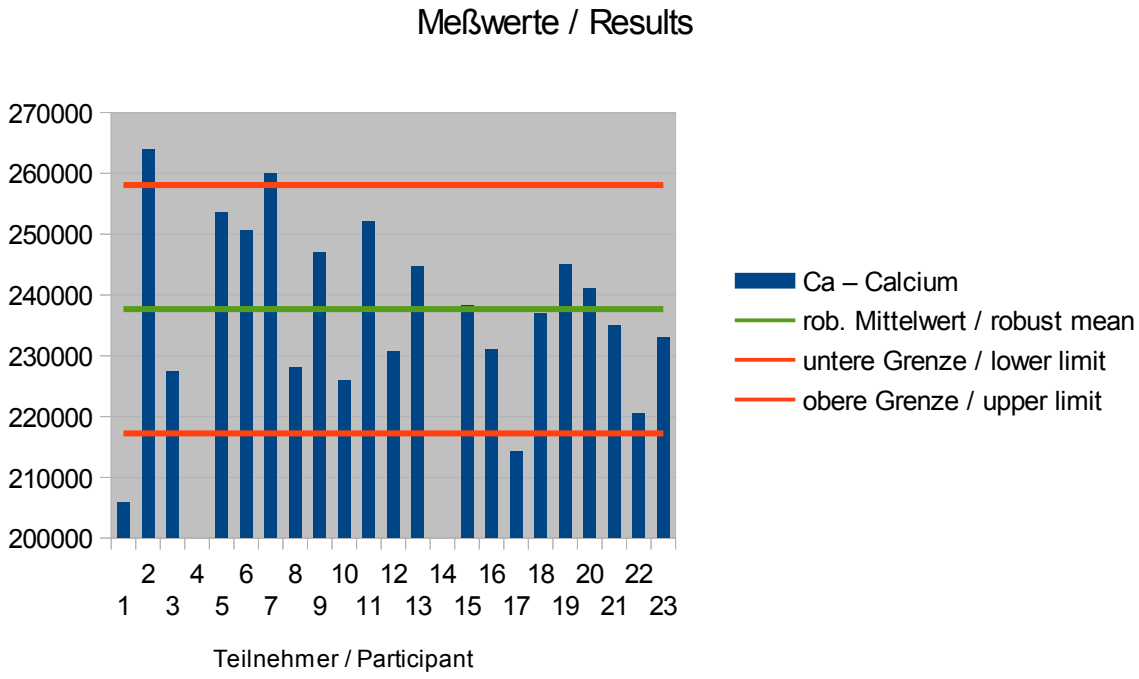


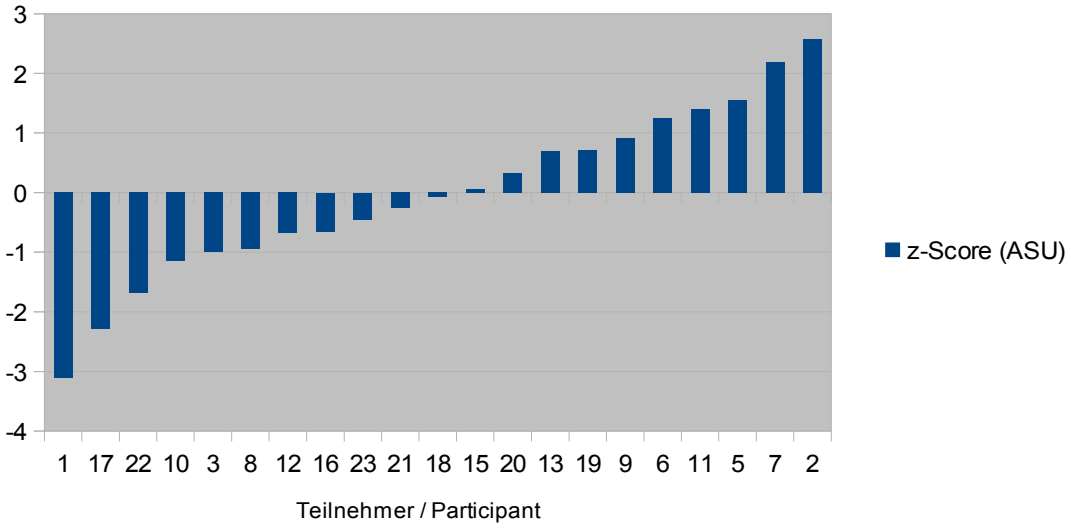


Auswerte nummer / Evaluation number	Ba – Barium	Abweichung / Deviation	z-Score	Hinweis / Remark
1				
2	0,93	0,04	0,3	
3	0,88	-0,01	-0,1	
4				
5	1,21	0,32	2,2	Ausreisser / Outlier
6				
7	1,05	0,16	1,1	
8	0,93	0,04	0,3	
9	0,96	0,07	0,5	
10	0,64	-0,25	-1,7	
11	0,81	-0,08	-0,6	
12	0,54	-0,35	-2,4	Ausreisser / Outlier
13	0,68	-0,21	-1,5	
14				
15				
16				
17	1,04	0,15	1,0	
18	0,94	0,05	0,3	
19	0,87	-0,02	-0,1	
20	0,98	0,09	0,6	
21	0,8	-0,09	-0,6	
22	0,97	0,07	0,5	
23				

4.3 Calcium in mg/kg

Statistic Data	
number of the results	21
number of outliers	2
mean	237378
median	237000
robust mean (X)	237656
robust standard deviation (S^x)	15340
target standard deviation (σ)	10212
target standard deviation (Horwitz) for information	5901
lower limit of target range	217233
upper limit of target range	258080
quotient S^x/σ	1,5
standard uncertainty U^x	4184
quotient U^x/σ	0,4
results in target range	17
percent in target range	81

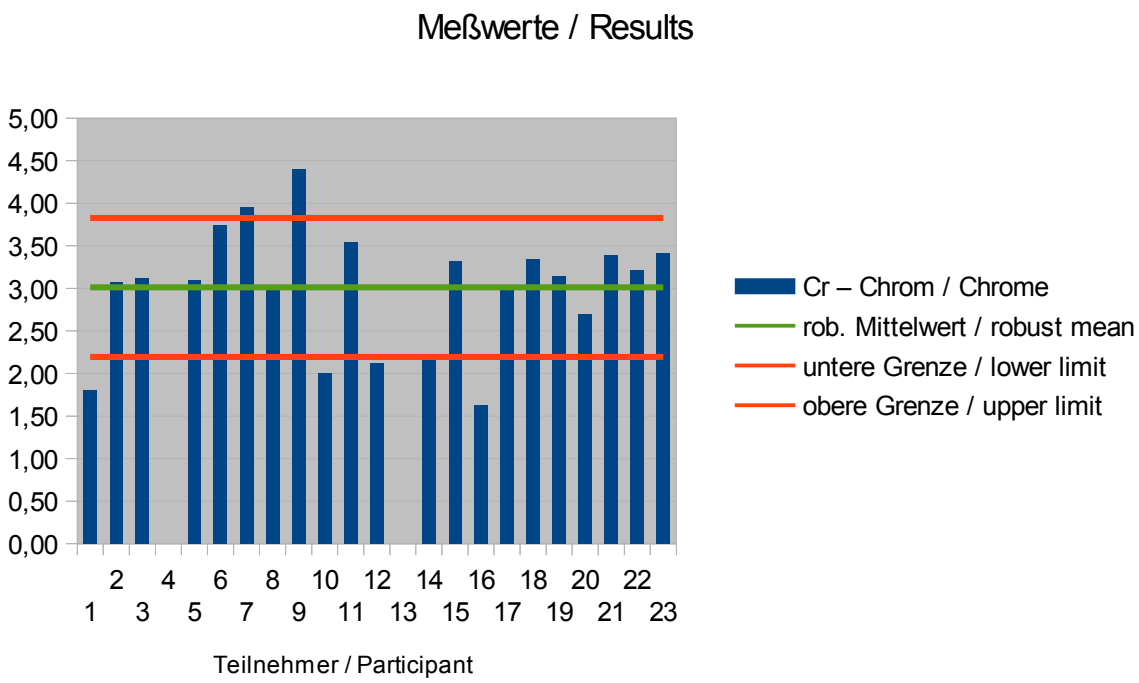


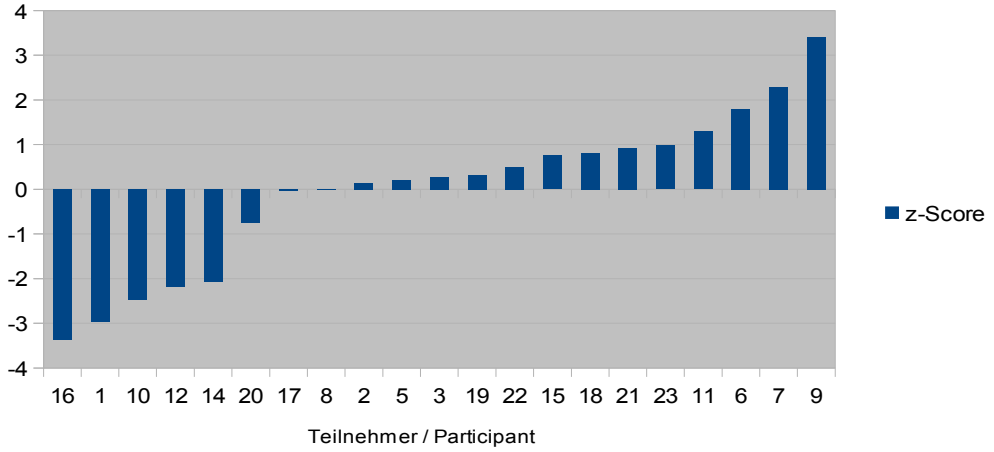


Auswerte nummer / Evaluation number	Ca - Calcium	Abweichung / Deviation	z-Score (ASU)	z-Score (Horwitz) zur info	Hinweis / Remark
1	205864,8	-31791,68	-3,1	-5,4	Ausreisser / Outlier
2	264000	26343,52	2,6	4,5	Ausreisser / Outlier
3	227500	-10156,48	-1,0	-1,7	
4					
5	253500	15843,52	1,6	2,7	
6	250500	12843,52	1,3	2,2	
7	260000	22343,52	2,2	3,8	
8	228000	-9656,48	-0,9	-1,6	
9	247000	9343,52	0,9	1,6	
10	226000	-11656,48	-1,1	-2,0	
11	252000	14343,52	1,4	2,4	
12	230778	-6878,48	-0,7	-1,2	
13	244726	7069,52	0,7	1,2	
14					
15	238288	631,52	0,1	0,1	
16	231000	-6656,48	-0,7	-1,1	
17	214250	-23406,48	-2,3	-4,0	
18	237000	-656,48	-0,1	-0,1	
19	245000	7343,52	0,7	1,2	
20	241000	3343,52	0,3	0,6	
21	235000	-2656,48	-0,3	-0,5	
22	220482	-17174,48	-1,7	-2,9	
23	233052	-4604,48	-0,5	-0,8	

4.4 Chrome in mg/kg

Statistic Data	
number of the results	21
number of outliers	2
mean	3,01
median	3,12
robust mean (X)	3,01
robust standard deviation (S^x)	0,75
target standard deviation (sigma')	0,41
lower limit of target range	2,19
upper limit of target range	3,83
quotient S^x/σ	1,8
standard uncertainty U^x	0,20
quotient U^x/σ	0,5
results in target range	14
percent in target range	67

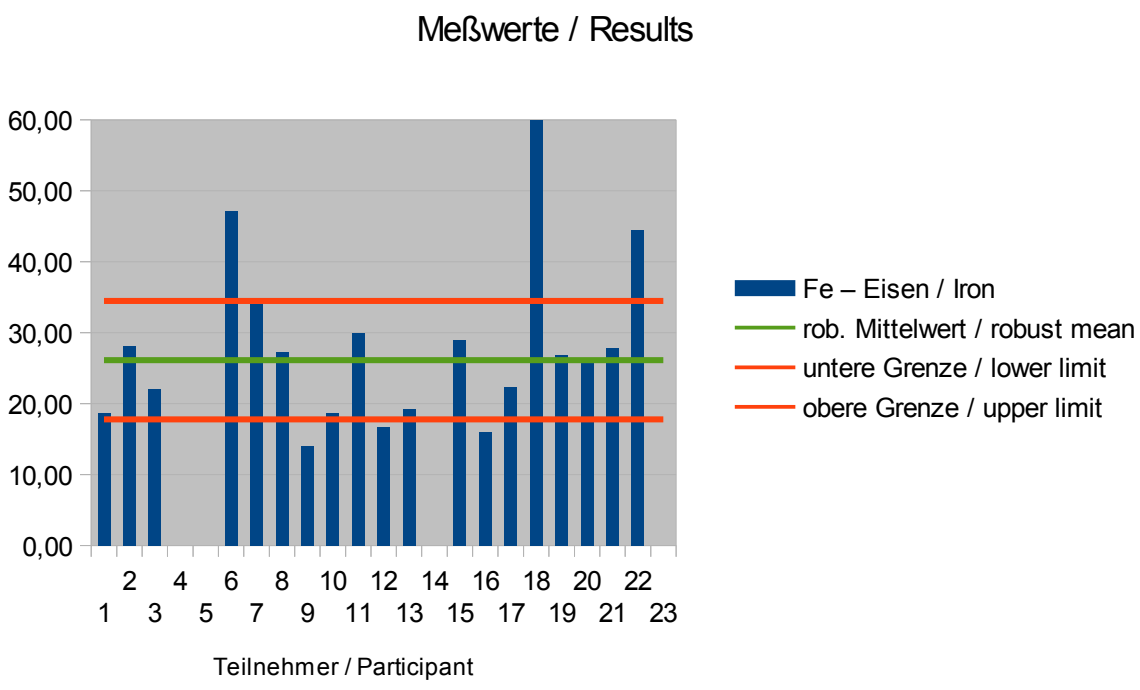


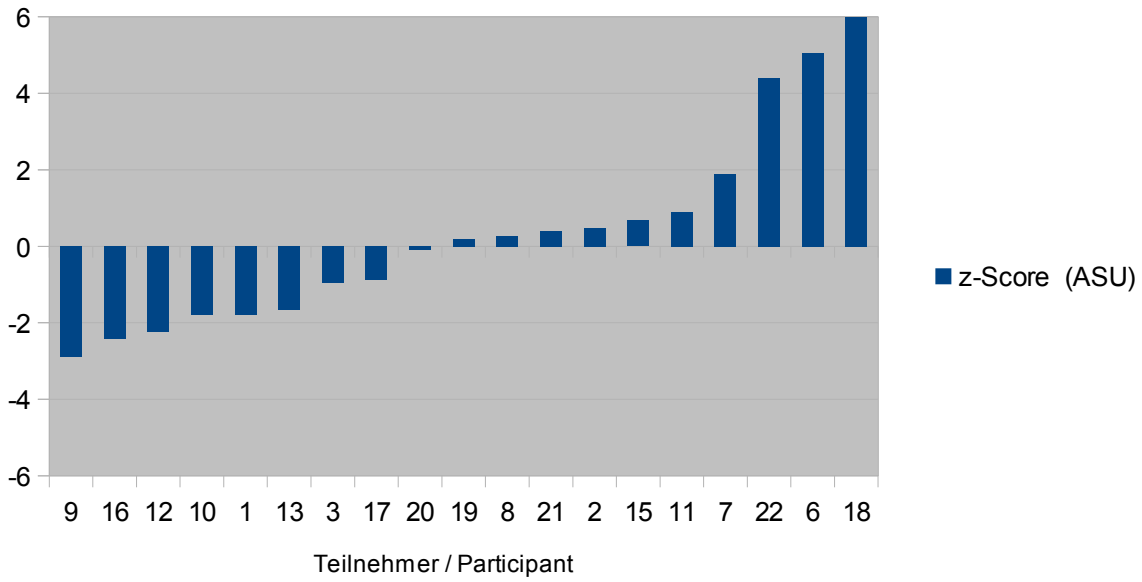


Auswerte nummer / Evaluation number	Cr - Chrom / Chrome	Abweichung / Deviation	z-Score	Hinweis / Remark
1	1,8	-1,21	-3,0	
2	3,07	0,06	0,1	
3	3,12	0,11	0,3	
4				
5	3,1	0,08	0,2	
6	3,75	0,73	1,8	
7	3,95	0,94	2,3	
8	3,01	-0,01	0,0	
9	4,4	1,39	3,4	Ausreisser / Outlier
10	2	-1,01	-2,5	
11	3,54	0,53	1,3	
12	2,12	-0,9	-2,2	
13				
14	2,17	-0,84	-2,1	
15	3,32	0,31	0,8	
16	1,63	-1,38	-3,4	Ausreisser / Outlier
17	3	-0,02	0,0	
18	3,34	0,33	0,8	
19	3,14	0,13	0,3	
20	2,7	-0,31	-0,8	
21	3,39	0,38	0,9	
22	3,22	0,2	0,5	
23	3,41	0,4	1,0	

4.5 Iron in mg/kg

Statistic Data	
number of the results	19
number of outliers	1
mean	44,4
median	26,9
robust mean (X)	26,1
robust standard deviation (S^x)	9,25
target standard deviation (sigma')	4,17
target standard deviation (Horwitz) for information	2,56
lower limit of target range	17,8
upper limit of target range	34,47
quotient S^x/σ	2,2
standard uncertainty U^x	2,65
quotient U^x/σ	0,6
results in target range	13
percent in target range	68

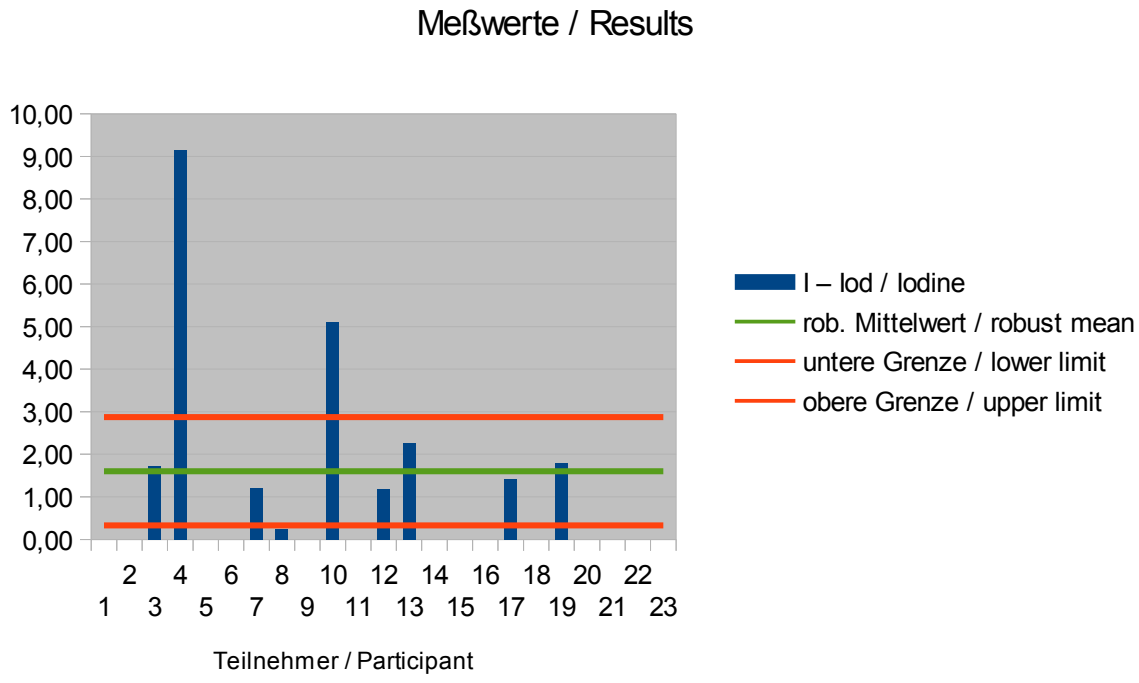


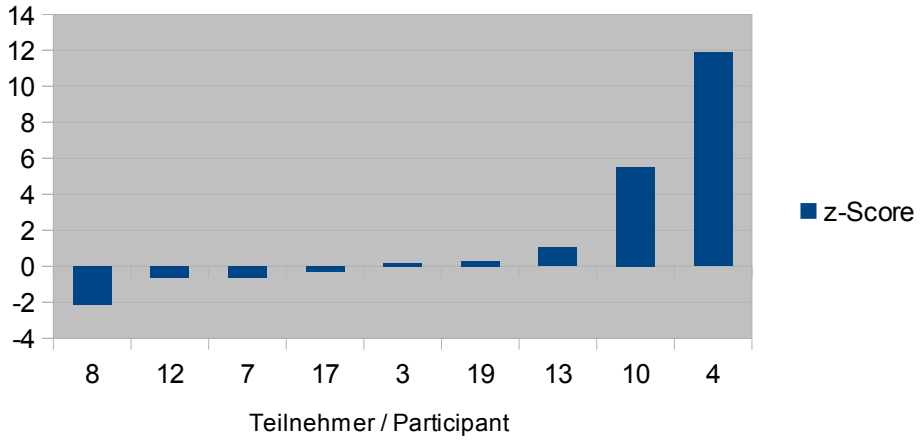


Auswerte nummer / Evaluation number	Fe - Eisen / Iron	Abweichung / Deviation	z-Score (ASU)	Z-Score (Horwitz) zur info	Hinweis / Remark
1	18,67	-7,45	-1,8	-2,9	
2	28,1	1,98	0,5	0,8	
3	22,1	-4,02	-1,0	-1,6	
4					
5					
6	47,2	21,08	5,1	8,2	
7	34	7,88	1,9	3,1	
8	27,28	1,16	0,3	0,5	
9	14	-12,12	-2,9	-4,7	
10	18,6	-7,52	-1,8	-2,9	
11	29,9	3,78	0,9	1,5	
12	16,75	-9,37	-2,2	-3,7	
13	19,2	-6,92	-1,7	-2,7	
14					
15	28,92	2,8	0,7	1,1	
16	16	-10,12	-2,4	-4,0	
17	22,4	-3,72	-0,9	-1,5	
18	375	348,88	83,6	136,4	Ausreisser / Outlier
19	26,9	0,78	0,2	0,3	
20	25,7	-0,42	-0,1	-0,2	
21	27,82	1,7	0,4	0,7	
22	44,46	18,34	4,4	7,2	
23					

4.6 Iodine in mg/kg

Statistic Data	
number of the results	9
number of outliers	2
mean	2,67
median	1,72
robust mean (X)	1,6
robust standard deviation (S^x)	0,92
target standard deviation (sigma')	0,64
lower limit of target range	0,33
upper limit of target range	2,87
quotient S^x/σ	1,5
standard uncertainty U^x	0,38
quotient U^x/σ	0,6
results in target range	6
percent in target range	67

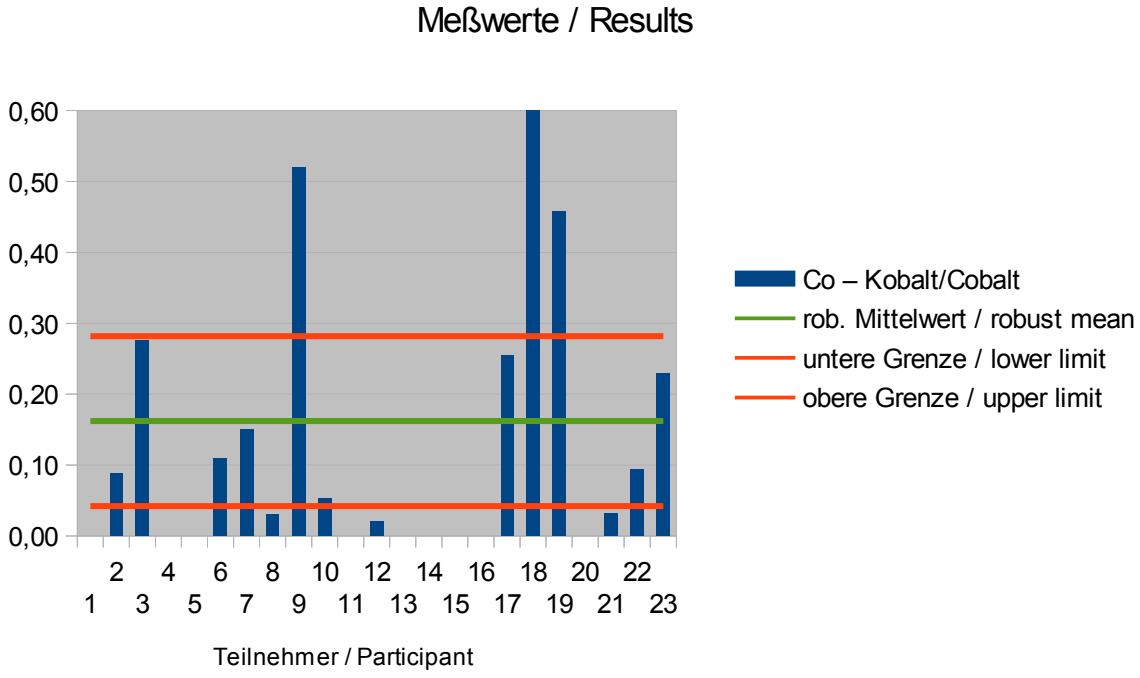


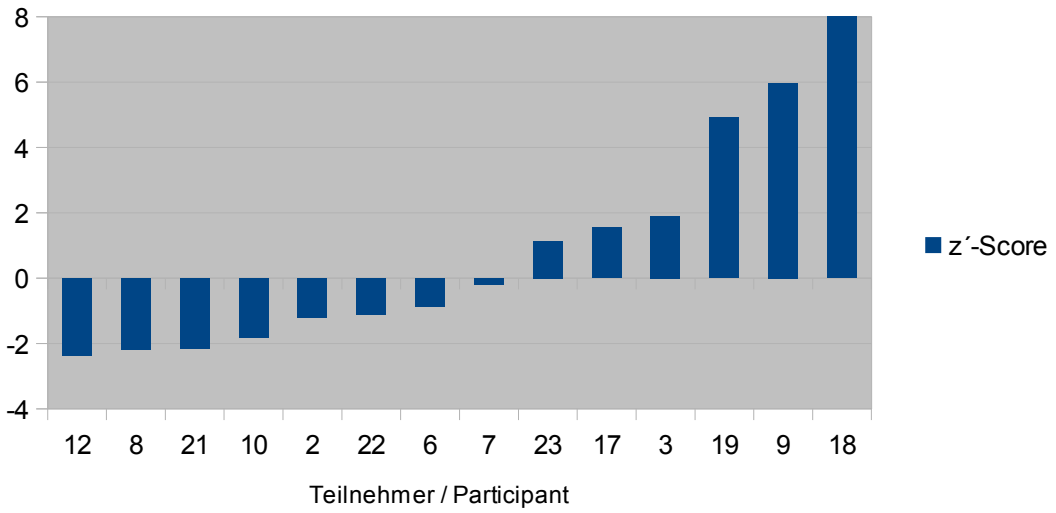


Auswerte nummer / Evaluation number	I - Iod / Iodine	Abweichung / Deviation	z-Score	Hinweis / Remark
1				
2				
3	1,72	0,12	0,2	
4	9,15	7,55	11,9	Ausreisser eliminiert/ Outlier eliminated
5				
6				
7	1,2	-0,4	-0,6	
8	0,24	-1,36	-2,1	
9				
10	5,1	3,5	5,5	Ausreisser / Outlier
11				
12	1,19	-0,41	-0,7	
13	2,27	0,67	1,1	
14				
15				
16				
17	1,41	-0,19	-0,3	
18				
19	1,79	0,19	0,3	
20				
21				
22				
23				

4.7 Cobalt in mg/kg

Statistic Data	
number of the results	14
number of outliers	1
mean	0,27
median	0,13
robust mean (X)	0,16
robust standard deviation (S^x)	0,15
target standard deviation (sigma')	0,06
target standard deviation (Horwitz) for information	0,03
lower limit of target range	0,04
upper limit of target range	0,28
quotient S^x/σ	4,3
standard uncertainty U^x	0,05
quotient U^x/σ	1,4
results in target range	8
percent in target range	57

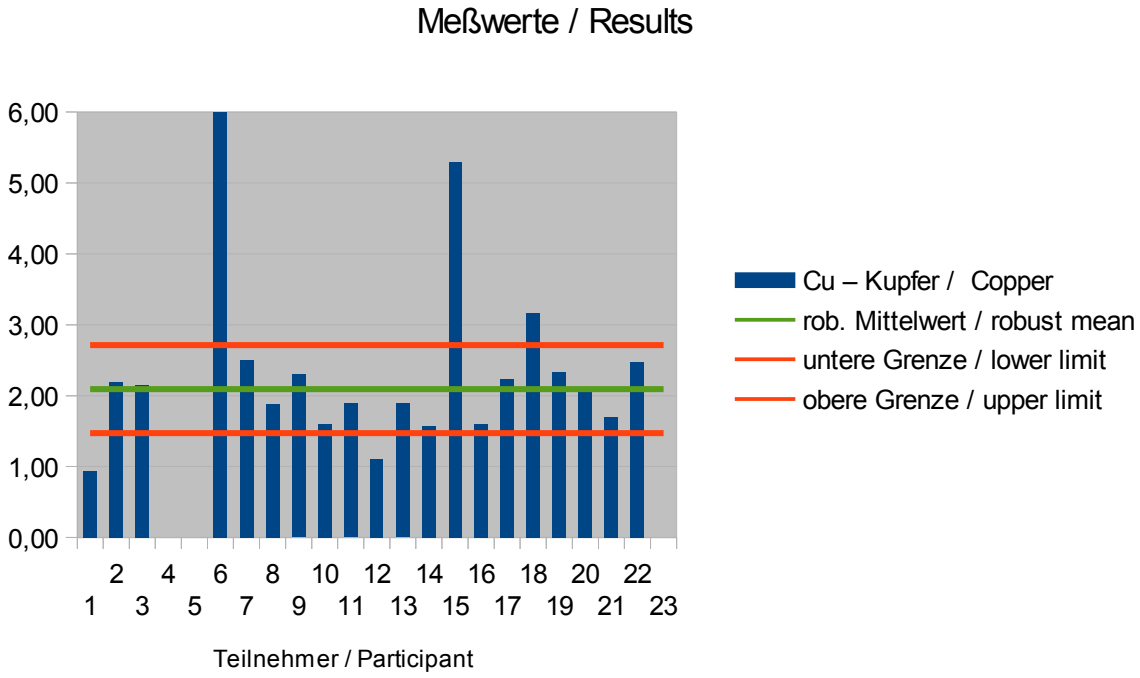


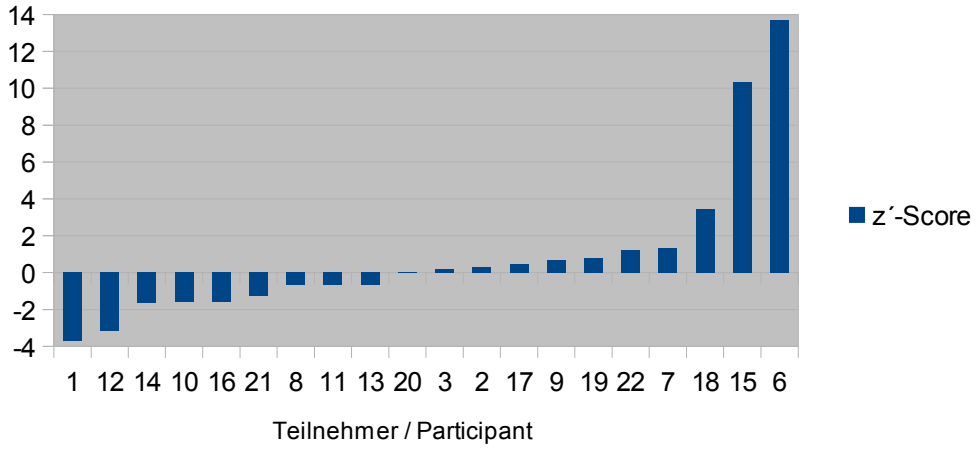


Auswerte nummer / Evaluation number	Co - Kobalt/Cobalt	Abweichung / Deviation	z´-Score	Z-Score (Horwitz) zur info	Hinweis / Remark
1					
2	0,09	-0,07	-1,2	-2,1	
3	0,28	0,11	1,9	3,3	
4					
5					
6	0,11	-0,05	-0,9	-1,5	
7	0,15	-0,01	-0,2	-0,4	
8	0,03	-0,13	-2,2	-3,9	
9	0,52	0,36	6,0	10,5	
10	0,05	-0,11	-1,8	-3,2	
11					
12	0,02	-0,14	-2,4	-4,2	
13					
14					
15					
16					
17	0,26	0,09	1,6	2,7	
18	1,53	1,37	22,8	40,1	Ausreisser eliminiert / Outlier eliminated
19	0,46	0,29	4,9	8,7	
20					
21	0,03	-0,13	-2,2	-3,8	
22	0,09	-0,07	-1,1	-2,0	
23	0,23	0,07	1,1	2,0	

4.8 Copper in mg/kg

Statistic Data	
number of the results	20
number of outliers	2
mean	2,36
median	2,13
robust mean (X)	2,09
robust standard deviation (S^x)	0,65
target standard deviation (sigma')	0,31
target standard deviation (ASU) for information	0,25
lower limit of target range	1,47
upper limit of target range	2,71
quotient S^x/σ	2,6
standard uncertainty U^x	0,18
quotient U^x/σ	0,7
results in target range	15
percent in target range	75

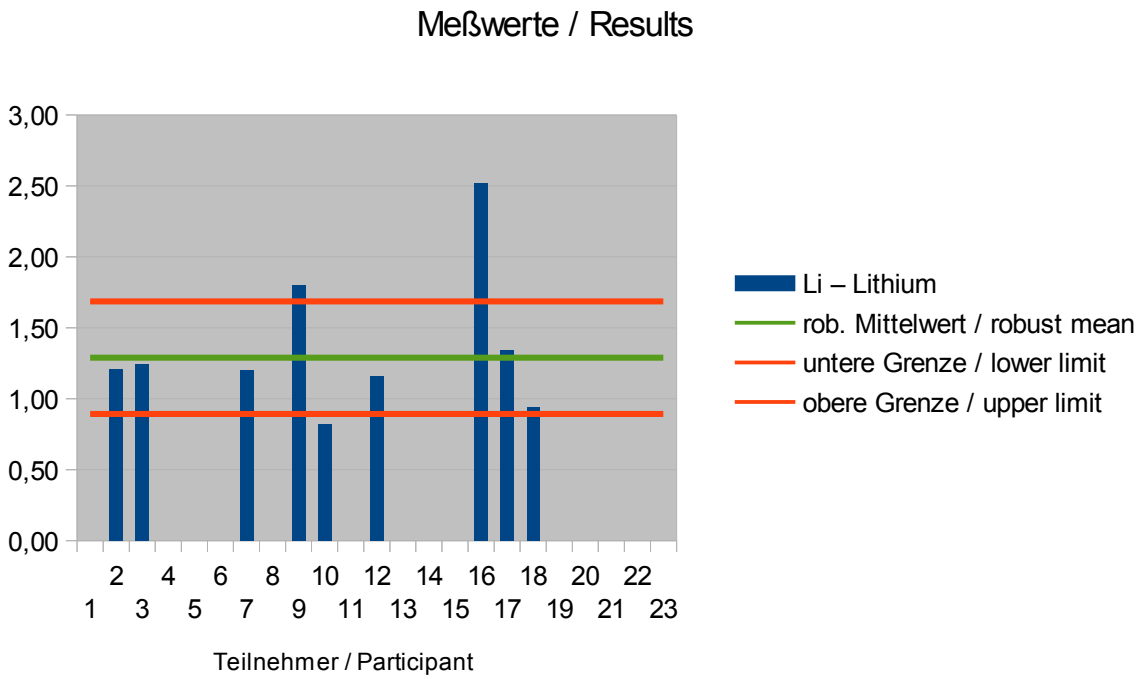


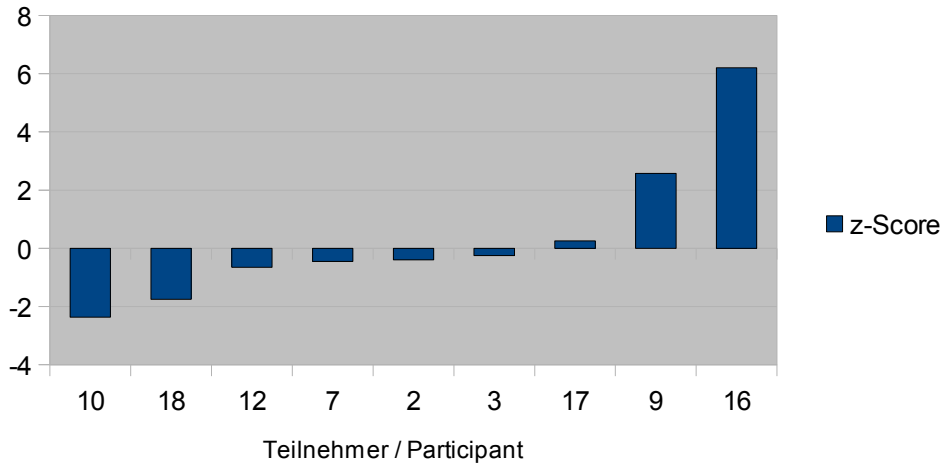


Auswerte nummer / Evaluation number	Cu - Kupfer / Copper	Abweichung / Deviation	z´-Score	Z-Score (ASU) zur info	Hinweis / Remark
1	0,94	-1,15	-3,7	-4,5	
2	2,19	0,1	0,3	0,4	
3	2,15	0,06	0,2	0,2	
4					
5					
6	6,35	4,26	13,7	16,8	Ausreisser / Outlier
7	2,5	0,41	1,3	1,6	
8	1,88	-0,21	-0,7	-0,8	
9	2,3	0,21	0,7	0,8	
10	1,6	-0,49	-1,6	-1,9	
11	1,89	-0,2	-0,6	-0,8	
12	1,11	-0,98	-3,2	-3,9	
13	1,89	-0,2	-0,6	-0,8	
14	1,57	-0,52	-1,7	-2,1	
15	5,3	3,21	10,3	12,7	Ausreisser / Outlier
16	1,6	-0,49	-1,6	-1,9	
17	2,23	0,14	0,5	0,6	
18	3,16	1,07	3,4	4,2	
19	2,33	0,24	0,8	0,9	
20	2,1	0,01	0,0	0,0	
21	1,7	-0,39	-1,3	-1,5	
22	2,47	0,38	1,2	1,5	
23					

4.9 Lithium in mg/kg

Statistic Data	
number of the results	9
number of outliers	1
mean	1,36
median	1,21
robust mean (X)	1,29
robust standard deviation (S^x)	0,40
target standard deviation (sigma')	0,20
lower limit of target range	0,89
upper limit of target range	1,69
quotient S^x/σ	2,0
standard uncertainty U^x	0,17
quotient U^x/σ	0,8
results in target range	6
percent in target range	67

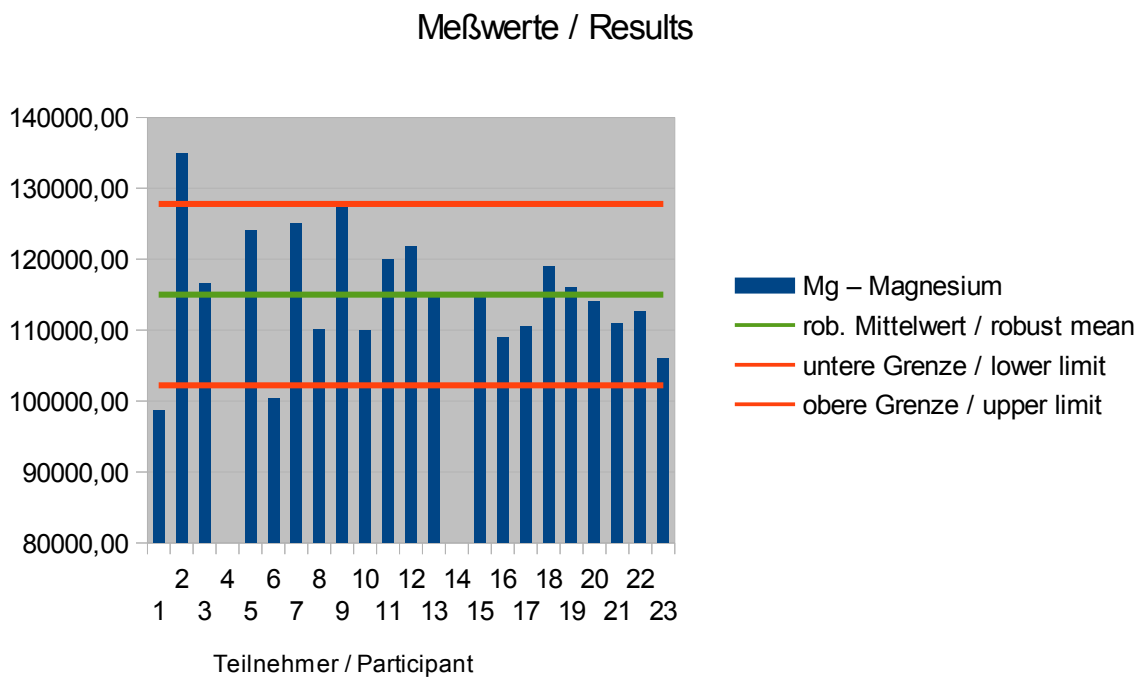


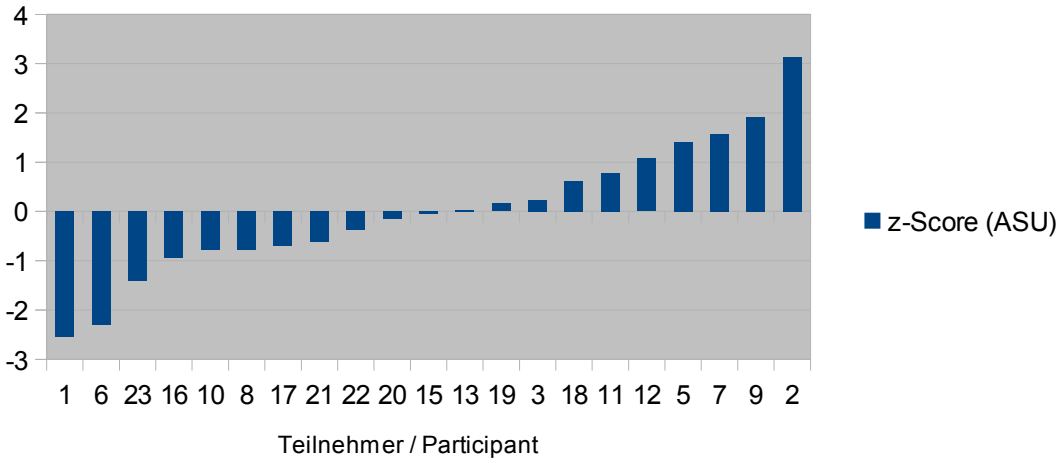


Auswerte nummer / Evaluation number	Li - Lithium	Abweichung / Deviation	z-Score	Hinweis / Remark
1				
2	1,21	-0,08	-0,4	
3	1,24	-0,05	-0,2	
4				
5				
6				
7	1,2	-0,09	-0,4	
8				
9	1,8	0,51	2,6	
10	0,82	-0,47	-2,4	
11				
12	1,16	-0,13	-0,7	
13				
14				
15				
16	2,52	1,23	6,2	Ausreisser / Outlier
17	1,34	0,05	0,3	
18	0,94	-0,35	-1,7	
19				
20				
21				
22				
23				

4.10 Magnesium in mg/kg

Statistic Data	
number of the results	21
number of outliers	2
mean	115080
median	114706
robust mean (X)	115000
robust standard deviation (S^x)	8471
target standard deviation (sigma')	6390
target standard deviation (Horwitz) for information	3185
lower limit of target range	102220
upper limit of target range	127779
quotient S^x/σ	1,3
standard uncertainty U^x	2311
quotient U^x/σ	0,4
results in target range	18
percent in target range	86

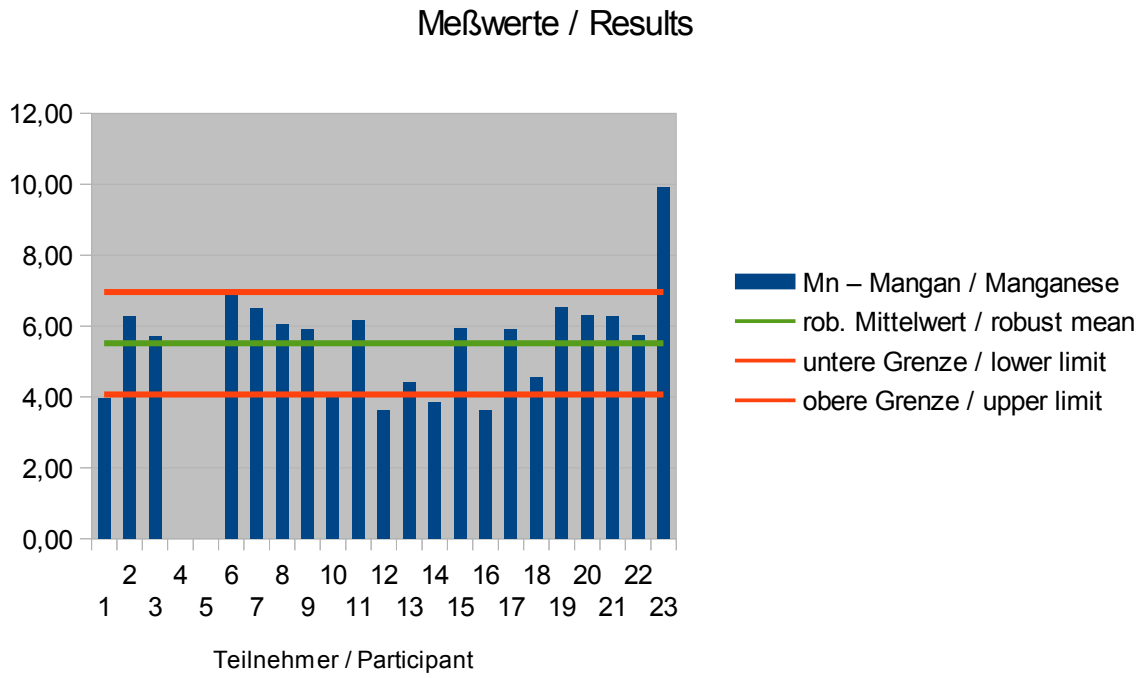


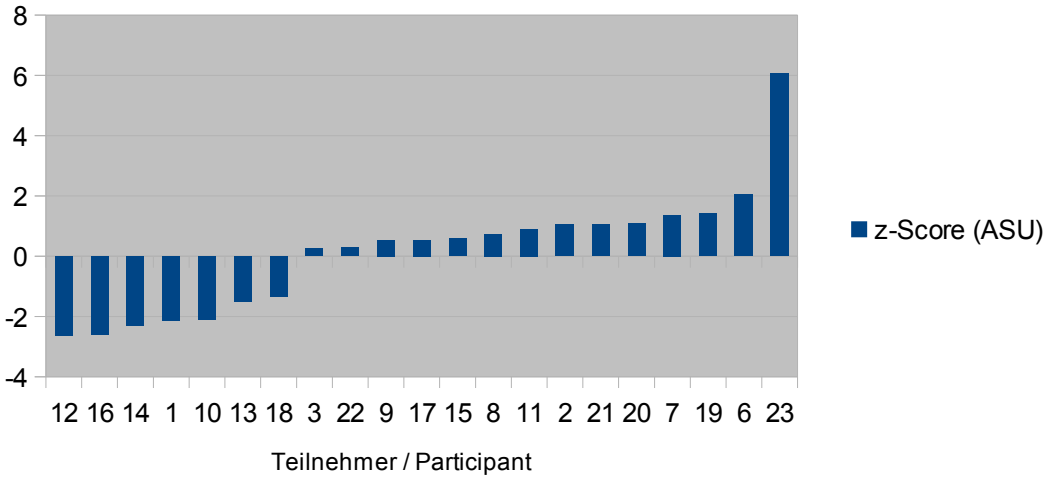


Auswerte nummer / Evaluation number	Mg - Magnesium	Abweichung / Deviation	z-Score (ASU)	z-Score (Horwitz) zur info	Hinweis / Remark
1	98683,3	-16316,22	-2,6	-5,1	Ausreisser / Outlier
2	135000	20000,48	3,1	6,3	Ausreisser / Outlier
3	116500	1500,48	0,2	0,5	
4					
5	124000	9000,48	1,4	2,8	
6	100300	-14699,52	-2,3	-4,6	
7	125000	10000,48	1,6	3,1	
8	110070	-4929,52	-0,8	-1,5	
9	127250	12250,48	1,9	3,8	
10	110000	-4999,52	-0,8	-1,6	
11	120000	5000,48	0,8	1,6	
12	121860	6860,48	1,1	2,2	
13	115149	149,48	0,0	0,0	
14					
15	114706	-293,52	0,0	-0,1	
16	109000	-5999,52	-0,9	-1,9	
17	110500	-4499,52	-0,7	-1,4	
18	119000	4000,48	0,6	1,3	
19	116000	1000,48	0,2	0,3	
20	114000	-999,52	-0,2	-0,3	
21	111000	-3999,52	-0,6	-1,3	
22	112675	-2324,52	-0,4	-0,7	
23	105988	-9011,52	-1,4	-2,8	

4.11 Manganese in mg/kg

Statistic Data	
number of the results	21
number of outliers	1
mean	5,63
median	5,91
robust mean (X)	5,52
robust standard deviation (S^x)	1,34
target standard deviation (sigma')	0,72
target standard deviation (Horwitz) for information	0,68
lower limit of target range	4,07
upper limit of target range	6,96
quotient S^x/σ	1,9
standard uncertainty U^x	0,37
quotient U^x/σ	0,5
results in target range	14
percent in target range	67

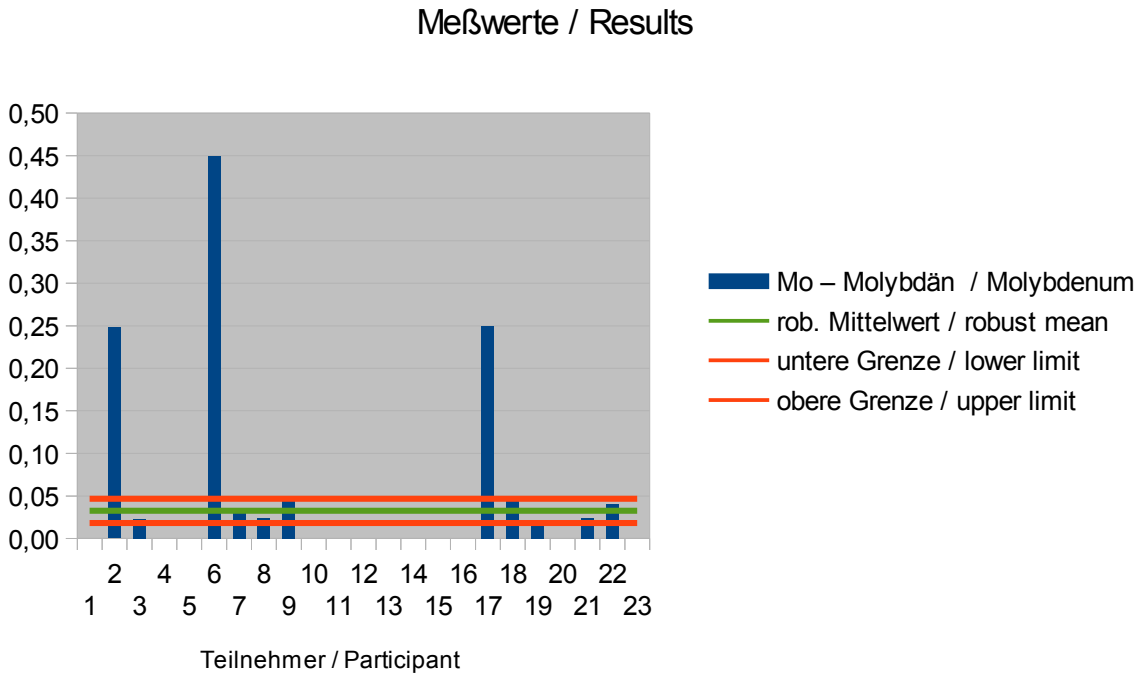


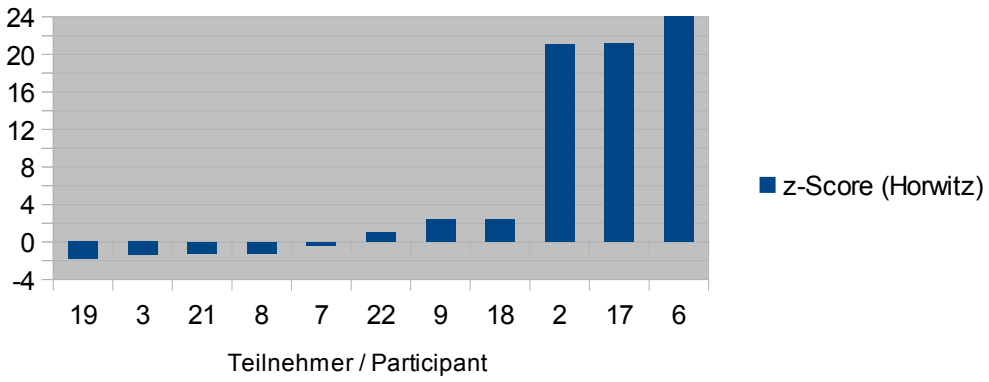


Auswerte nummer / Evaluation number	Mn - Mangan / Manganese	Abweichung / Deviation	z-Score (ASU)	z-Score (Horwitz) zur info	Hinweis / Remark
1	3,96	-1,56	-2,2	-2,3	
2	6,27	0,75	1,0	1,1	
3	5,7	0,18	0,3	0,3	
4					
5					
6	7	1,48	2,1	2,2	
7	6,5	0,98	1,4	1,4	
8	6,06	0,54	0,7	0,8	
9	5,9	0,38	0,5	0,6	
10	4	-1,52	-2,1	-2,2	
11	6,17	0,65	0,9	1,0	
12	3,62	-1,9	-2,6	-2,8	
13	4,42	-1,1	-1,5	-1,6	
14	3,85	-1,67	-2,3	-2,4	
15	5,94	0,42	0,6	0,6	
16	3,63	-1,89	-2,6	-2,8	
17	5,91	0,39	0,5	0,6	
18	4,56	-0,96	-1,3	-1,4	
19	6,54	1,02	1,4	1,5	
20	6,3	0,78	1,1	1,1	
21	6,28	0,76	1,1	1,1	
22	5,73	0,21	0,3	0,3	
23	9,9	4,38	6,1	6,4	Ausreisser / Outlier

4.12 Molybdenum in mg/kg

Statistic Data	
number of the results	11
number of outliers	3
mean	0,110
median	0,040
robust mean (X)	0,033
robust standard deviation (S^x)	0,014
target standard deviation (sigma')Horwitz-Thompson	0,007
lower limit of target range	0,018
upper limit of target range	0,047
quotient S^x/σ	2,0
standard uncertainty U^x	0,005
quotient U^x/σ	0,7
results in target range	6
percent in target range	55

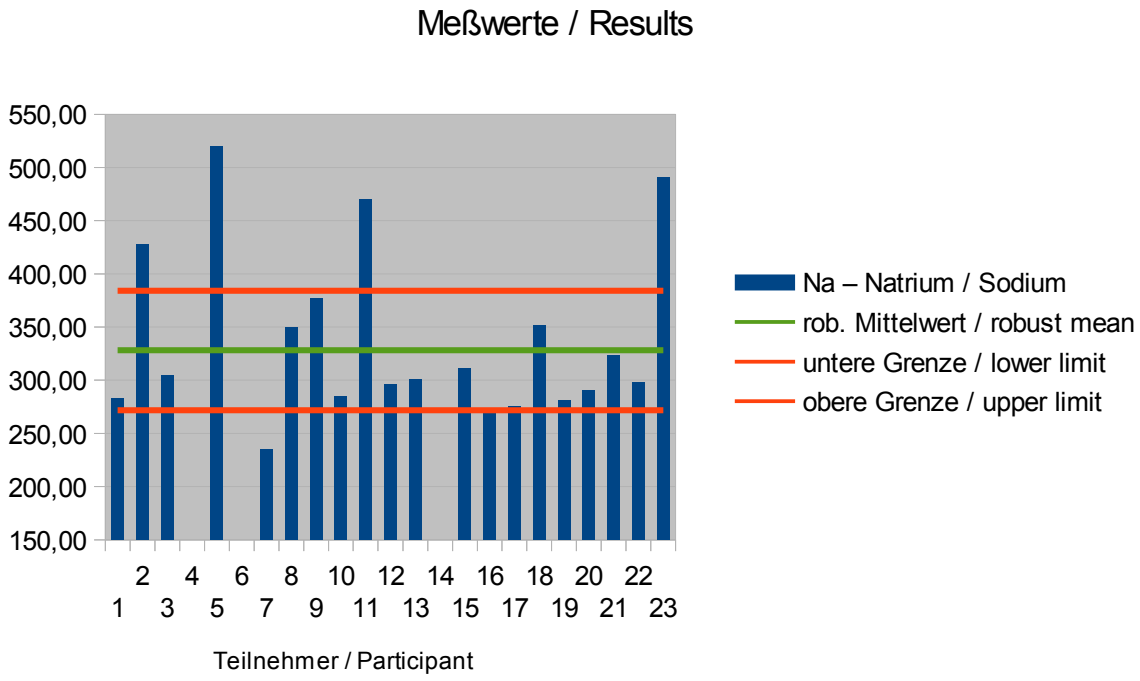


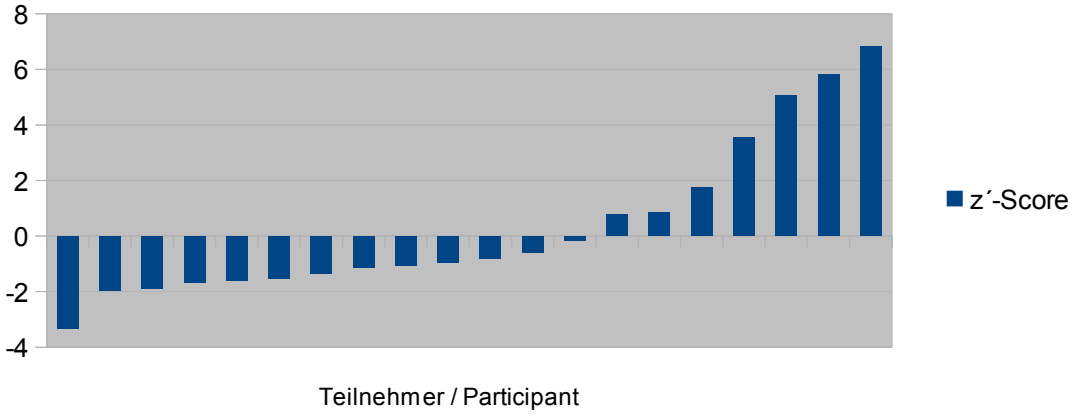


Auswerte nummer / Evaluation number	Mo - Molybdän / Molybdenum	Abweichung / Deviation	z-Score (Horwitz)	Hinweis / Remark
1				
2	0,25	0,22	21,1	Eliminiert / eliminated
3	0,02	-0,01	-1,4	
4				
5				
6	0,45	0,42	40,8	Eliminiert / eliminated
7	0,03	0	-0,4	
8	0,02	-0,01	-1,2	
9	0,05	0,02	2,4	
10				
11				
12				
13				
14				
15				
16				
17	0,25	0,22	21,2	Eliminiert / eliminated
18	0,05	0,02	2,4	
19	0,02	-0,01	-1,8	
20				
21	0,02	-0,01	-1,2	
22	0,04	0,01	1,0	
23				

4.13 Sodium in mg/kg

Statistic Data	
number of the results	20
number of outliers	2
mean	337
median	303
robust mean (X)	328
robust standard deviation (S^x)	69,7
target standard deviation (sigma')	28,1
target standard deviation (ASU) for information	20,2
lower limit of target range	272
upper limit of target range	384
quotient S^x/σ	3,4
standard uncertainty U^x	19,5
quotient U^x/σ	1,0
results in target range	15
percent in target range	75

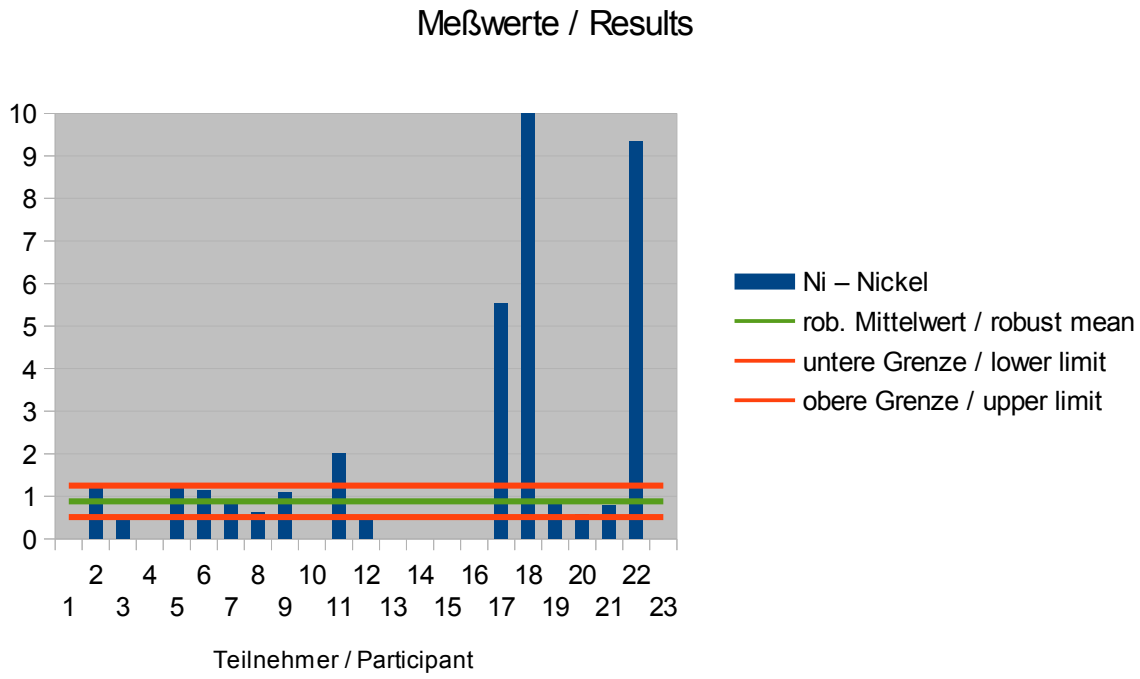


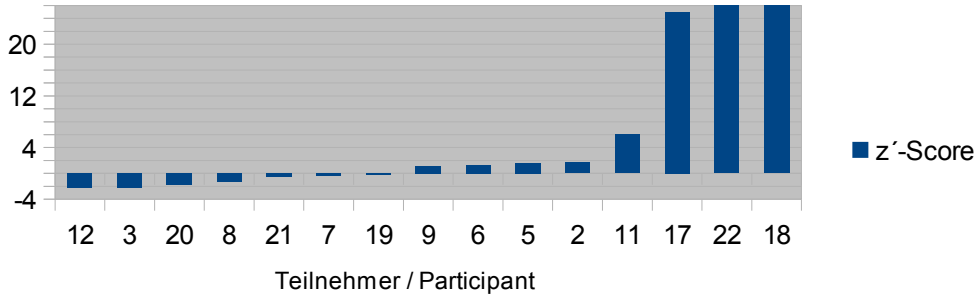


Auswerte nummer / Evaluation number	Na - Natrium / Sodium	Abweichung / Deviation	z-Score	z-Score (ASU) zur info	Hinweis / Remark
1	282,8	-45,26	´	-2,2	
2	428	99,94	3,6	4,9	
3	305	-23,06	-0,8	-1,1	
4					
5	520	191,94	6,8	9,5	Ausreisser / Outlier
6					
7	235	-93,06	-3,3	-4,6	
8	349,86	21,8	0,8	1,1	
9	377	48,94	1,7	2,4	
10	285	-43,06	-1,5	-2,1	
11	470	141,94	5,1	7,0	
12	296	-32,06	-1,1	-1,6	
13	301	-27,06	-1,0	-1,3	
14					
15	311,2	-16,86	-0,6	-0,8	
16	273	-55,06	-2,0	-2,7	
17	275,25	-52,81	-1,9	-2,6	
18	352	23,94	0,9	1,2	
19	281	-47,06	-1,7	-2,3	
20	290	-38,06	-1,4	-1,9	
21	323,33	-4,73	-0,2	-0,2	
22	298	-30,06	-1,1	-1,5	
23	491	162,94	5,8	8,1	Ausreisser / Outlier

4.14 Nickel in mg/kg

Statistic Data	
number of the results	15
number of outliers	1
mean	2,8
median	1,1
robust mean (X)	0,88
robust standard deviation (S^x)	0,36
target standard deviation (sigma')	0,186
target standard deviation for information (Horwitz)	0,144
lower limit of target range	0,511
upper limit of target range	1,25
quotient S^x/σ	2,5
standard uncertainty U^x	0,12
quotient U^x/σ	0,8
results in target range	9
percent in target range	60

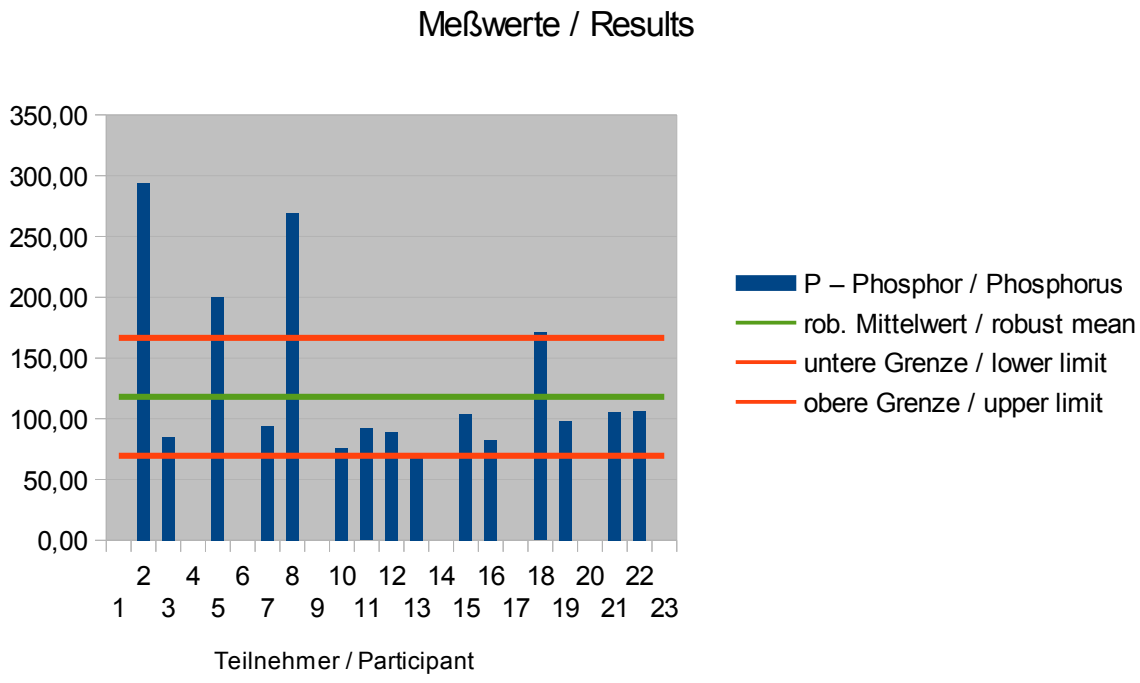


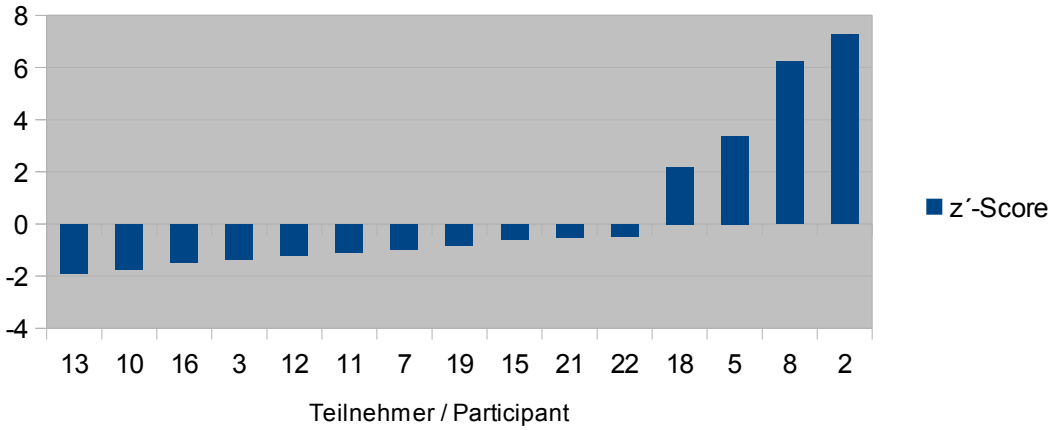


Auswerte nummer / Evaluation number	Ni – Nickel	Abweichung / Deviation	z´-Score	Z-Score (Horwitz) zur info	Hinweis / Remark
1					
2	1,2	0,32	1,7	2,2	
3	0,47	-0,41	-2,2	-2,9	
4					
5	1,19	0,3	1,6	2,1	
6	1,13	0,25	1,3	1,7	
7	0,8	-0,08	-0,4	-0,6	
8	0,63	-0,25	-1,4	-1,7	
9	1,1	0,22	1,2	1,5	
10					
11	2	1,12	6,0	7,8	
12	0,46	-0,42	-2,3	-2,9	
13					
14					
15					
16					
17	5,53	4,64	25,0	32,3	Eliminiert / eliminated
18	16	15,12	81,4	105,1	Eliminiert / eliminated
19	0,84	-0,04	-0,2	-0,3	
20	0,56	-0,32	-1,7	-2,2	
21	0,78	-0,1	-0,5	-0,7	
22	9,34	8,46	45,6	58,8	Eliminiert / eliminated
23					

4.15 Phosphorus in mg/kg

Statistic Data	
number of the results	15
number of outliers	2
mean	129
median	98,0
robust mean (X)	118
robust standard deviation (S^x)	54,1
target standard deviation (sigma')	24,3
target standard deviation (ASU) for information	16,9
lower limit of target range	69,5
upper limit of target range	167
quotient S^x/σ	3,2
standard uncertainty U^x	17,5
quotient U^x/σ	1,0
results in target range	11
percent in target range	73

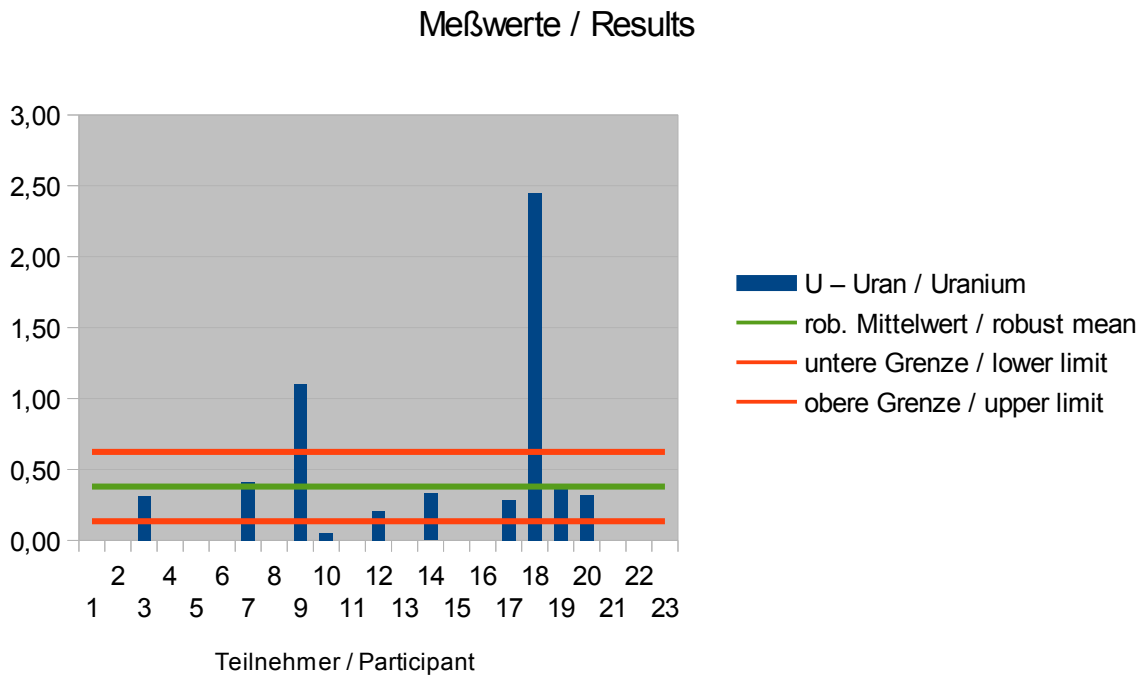


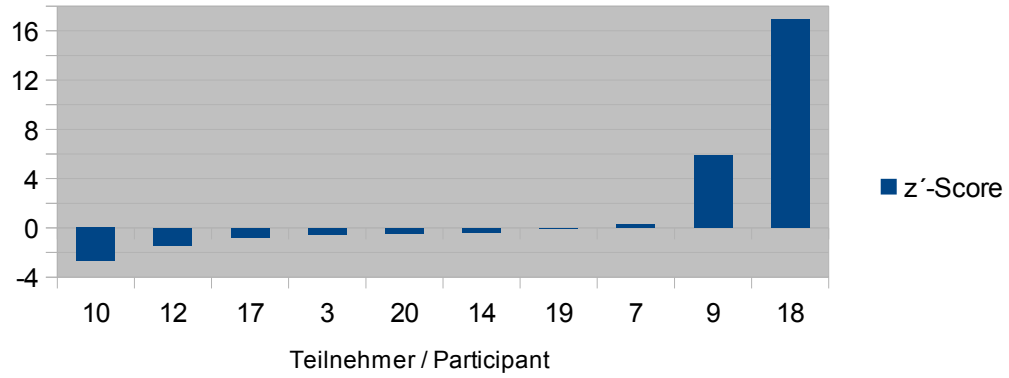


Auswerte nummer / Evaluation number	P - Phosphor / Phosphorus	Abweichung / Deviation	z´-Score	Z-Score (ASU) zur info	Hinweis / Remark
1					
2	294	175,92	7,2	10,4	Ausreisser / Outlier
3	85	-33,08	-1,4	-2,0	
4					
5	200	81,92	3,4	4,9	
6					
7	94	-24,08	-1,0	-1,4	
8	269,33	151,24	6,2	9,0	Ausreisser / Outlier
9					
10	76,1	-41,98	-1,7	-2,5	
11	92	-26,08	-1,1	-1,5	
12	88,9	-29,18	-1,2	-1,7	
13	72	-46,08	-1,9	-2,7	
14					
15	103,8	-14,28	-0,6	-0,8	
16	82,5	-35,58	-1,5	-2,1	
17					
18	171	52,92	2,2	3,1	
19	98	-20,08	-0,8	-1,2	
20					
21	105,34	-12,74	-0,5	-0,8	
22	106,2	-11,88	-0,5	-0,7	
23					

4.16 Uranium in mg/kg

Statistic Data	
number of the results	10
number of outliers	1
mean	0,583
median	0,325
robust mean (X)	0,379
robust standard deviation (S^x)	0,253
target standard deviation (sigma')	0,122
target standard deviation (Horwitz) for information	0,070
lower limit of target range	0,135
upper limit of target range	0,624
quotient S^x/σ	3,6
standard uncertainty U^x	0,100
quotient U^x/σ	1,4
results in target range	7
percent in target range	70

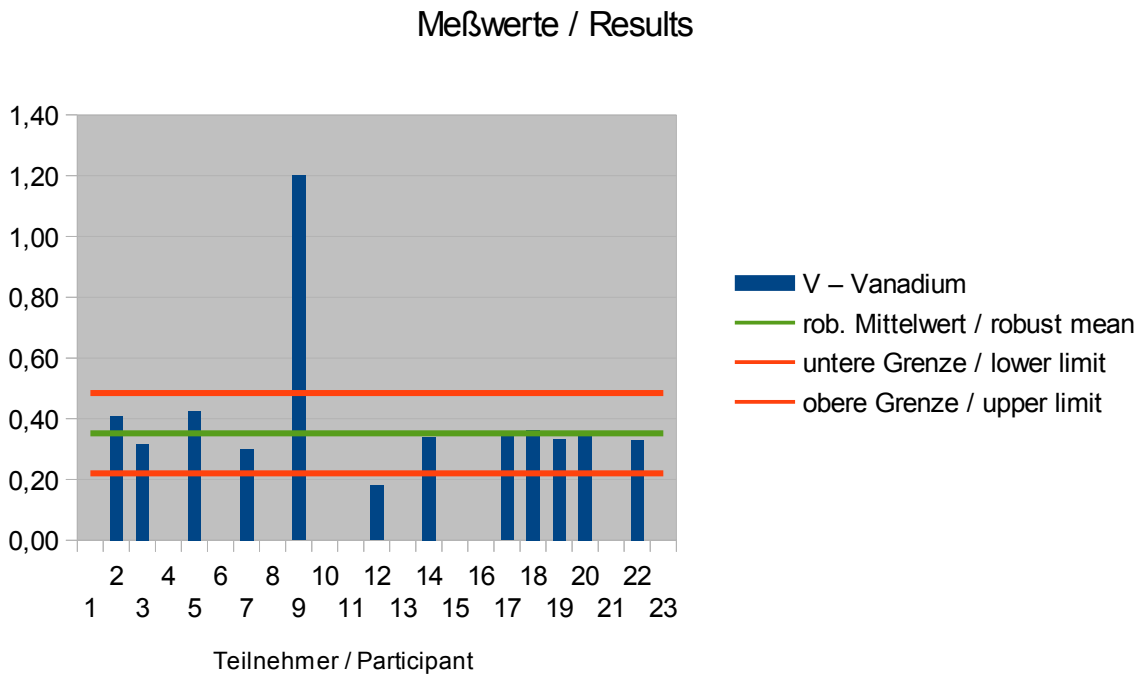


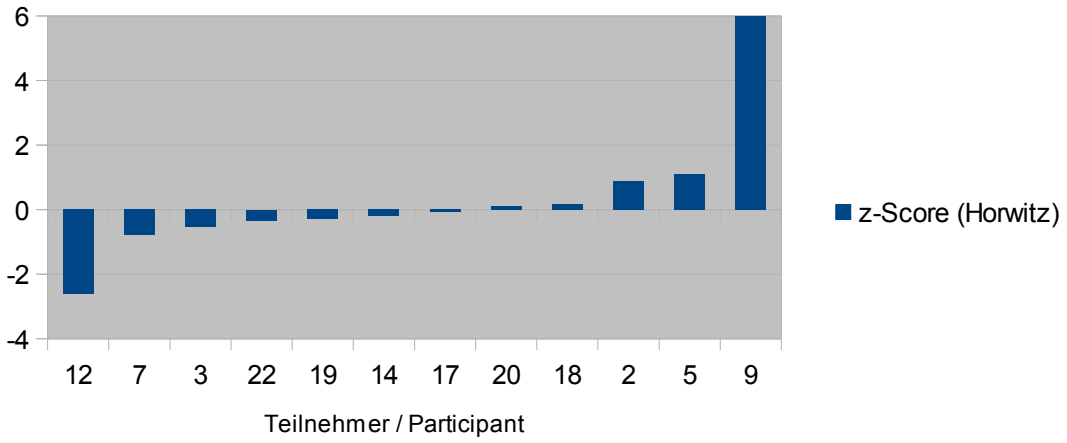


Auswerte nummer / Evaluation number	U - Uran / Uranium	Abweichung / Deviation	z´-Score	Z-Score (Horwitz) zur info	Hinweis / Remark
1					
2					
3	0,31	-0,07	-0,6	-1,0	
4					
5					
6					
7	0,41	0,03	0,3	0,4	
8					
9	1,1	0,72	5,9	10,3	
10	0,05	-0,33	-2,7	-4,7	
11					
12	0,21	-0,17	-1,4	-2,5	
13					
14	0,33	-0,05	-0,4	-0,7	
15					
16					
17	0,28	-0,1	-0,8	-1,4	
18	2,45	2,07	16,9	29,5	Ausreisser / Outlier
19	0,37	-0,01	0,0	-0,1	
20	0,32	-0,06	-0,5	-0,8	
21					
22					
23					

4.17 Vanadium in mg/kg

Statistic Data	
number of the results	12
number of outliers	1
mean	0,409
median	0,344
robust mean (X)	0,353
robust standard deviation (S^x)	0,058
target standard deviation (sigma')	0,066
lower limit of target range	0,221
upper limit of target range	0,485
quotient S^x/σ	0,9
standard uncertainty U^x	0,021
quotient U^x/σ	0,3
results in target range	10
percent in target range	83

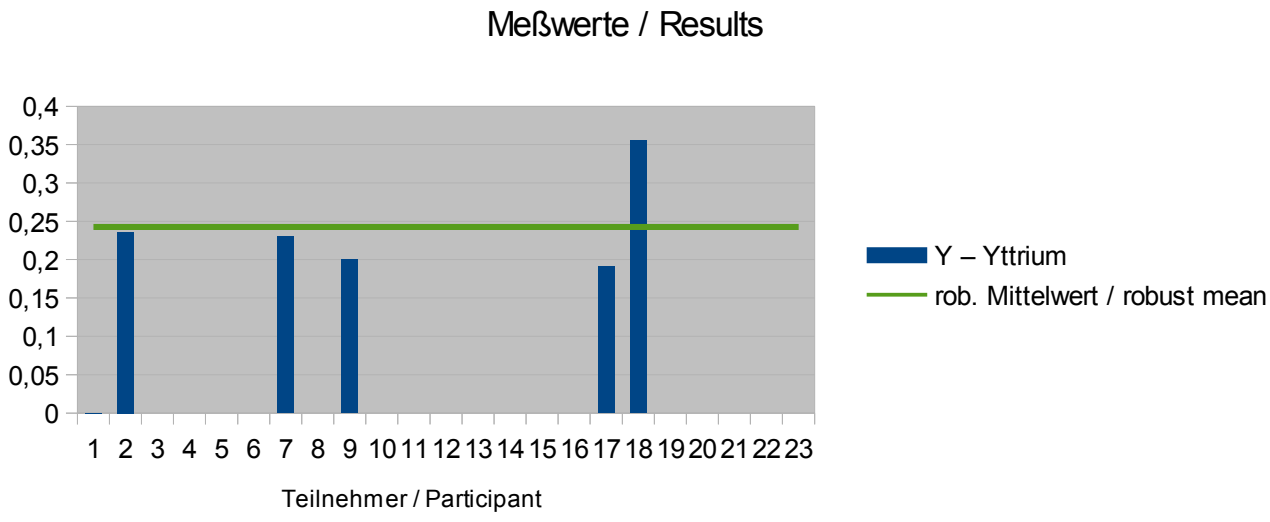




Auswerte nummer / Evaluation number	V - Vanadium	Abweichung / Deviation	z-Score (Horwitz)	Hinweis / Remark
1				
2	0,41	0,06	0,9	
3	0,32	-0,04	-0,5	
4				
5	0,43	0,07	1,1	
6				
7	0,3	-0,05	-0,8	
8				
9	1,2	0,85	12,8	Ausreisser / Outlier
10				
11				
12	0,18	-0,17	-2,6	
13				
14	0,34	-0,01	-0,2	
15				
16				
17	0,35	-0,01	-0,1	
18	0,36	0,01	0,2	
19	0,33	-0,02	-0,3	
20	0,36	0,01	0,1	
21				
22	0,33	-0,02	-0,3	
23				

4.18 Yttrium in mg/kg

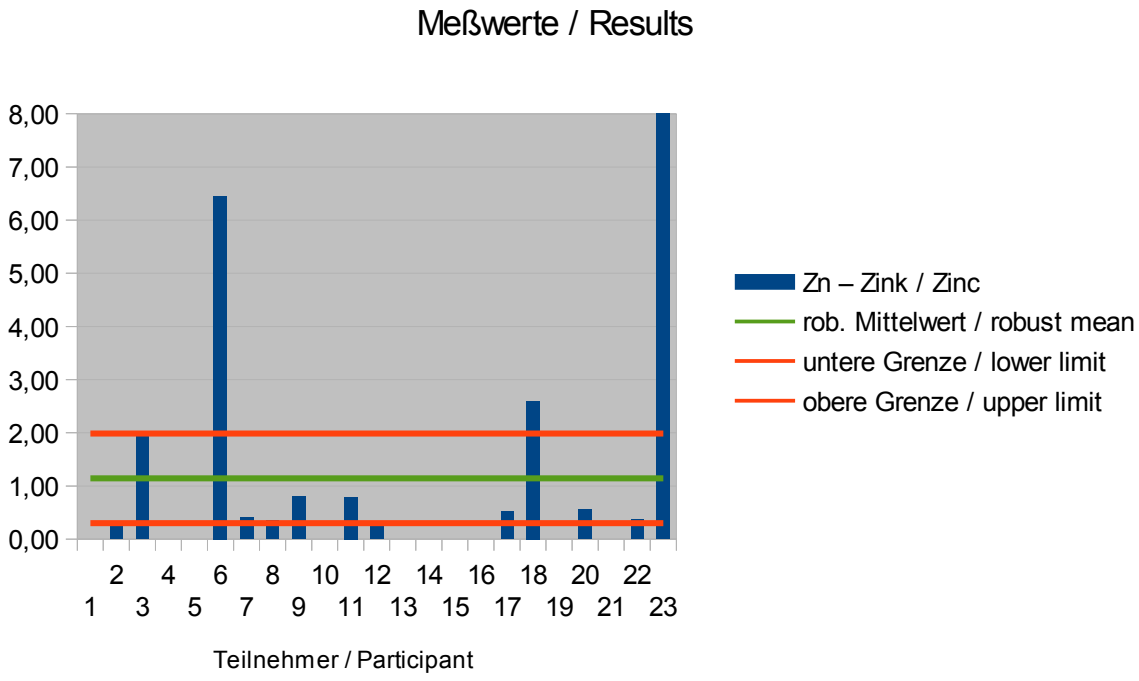
Statistic Data	
number of the results	5
number of outliers	0
mean	0,243
median	0,230
robust mean (X)	0,238
robust standard deviation (S^x)	0,069
target standard deviation (sigma')	nicht berechnet / not calculated
lower limit of target range	nicht berechnet / not calculated
upper limit of target range	nicht berechnet / not calculated
quotient S^x/σ	nicht berechnet / not calculated
standard uncertainty U^x	nicht berechnet / not calculated
quotient U^x/σ	nicht berechnet / not calculated
results in target range	nicht berechnet / not calculated
percent in target range	nicht berechnet / not calculated

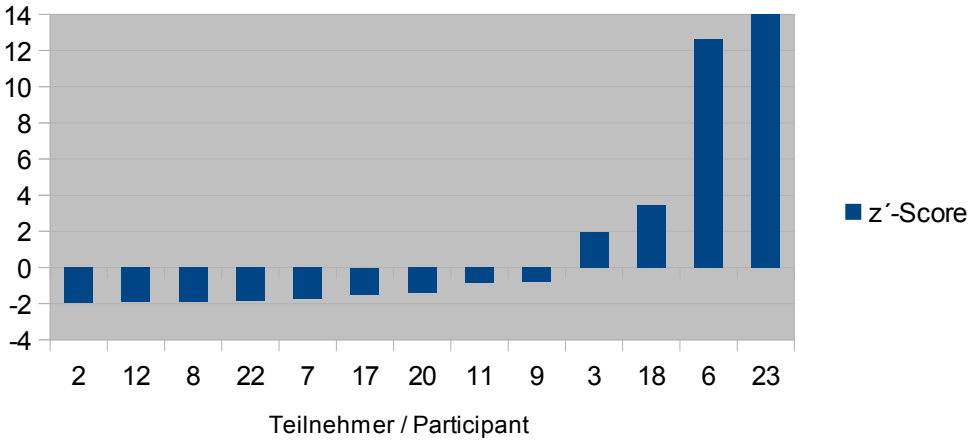


Auswerte nummer / Evaluation number	Y - Yttrium	Abweichung / Deviation	Hinweis / Remark
1			
2	0,24	0	
3			
4			
5			
6			
7	0,23	-0,01	
8			
9	0,2	-0,04	
10			
11			
12			
13			
14			
15			
16			
17	0,19	-0,05	
18	0,36	0,12	
19			
20			
21			
22			
23			

4.19 Zinc in mg/kg

Statistic Data	
number of the results	13
number of outliers	1
mean	2,84
median	0,550
robust mean (X)	1,14
robust standard deviation (S^x)	1,19
target standard deviation (sigma')	0,421
target standard deviation (ASU) for information	0,074
lower limit of target range	0,301
upper limit of target range	1,98
quotient S^x/σ	16
standard uncertainty U^x	0,414
quotient U^x/σ	5,6
results in target range	10
percent in target range	77





Auswerte nummer / Evaluation number	Zn - Zink / Zinc	Abweichung / Deviation	z´-Score	z-Score (ASU) zur info	Hinweis / Remark
1					
2	0,32	-0,82	-1,9	-11,0	
3	1,97	0,83	2,0	11,1	
4					
5					
6	6,45	5,31	12,6	71,3	
7	0,4	-0,74	-1,8	-10,0	
8	0,35	-0,8	-1,9	-10,7	
9	0,8	-0,34	-0,8	-4,6	
10					
11	0,79	-0,35	-0,8	-4,7	
12	0,34	-0,81	-1,9	-10,8	
13					
14					
15					
16					
17	0,52	-0,63	-1,5	-8,4	
18	2,6	1,46	3,5	19,6	
19					
20	0,55	-0,59	-1,4	-8,0	
21					
22	0,36	-0,78	-1,9	-10,5	
23	21,5	20,36	48,4	273,3	Ausreisser / Outlier

5 Documentation

5.1 Primary data

Part.	Unit	Al – Aluminium	Sample A	Sample B	Unit	Ba – Barium	Sample A	Sample B	Unit	Ca – Calcium	Sample A	Sample B
1	mg/kg	14,58	14,34	14,82	-	-	-	-	mg/100g	20586,48	20798,29	20374,68
2	ppm	30,0	29,5	30,6	ppm	0,930	0,95	0,91	%	26,4	26,4	26,3
3	mg/kg	30,5	30,6	30,3	mg/kg	0,88	0,9	0,86	mg/kg	227500	228000	227000
4												
5	mg/kg		84	76	mg/kg		1,45	0,97	g/kg		254	253
6	mg/kg	11,40	11,11	11,68					%	25,05	24,6	25,5
7	mg/kg	30	29	31	mg/kg	1,05	1,02	1,07	mg/kg	260000	257000	265000
8	mg/kg	53,13	49,75	55,2	mg/kg	0,93	0,92	0,94	%	22,8	22,2	23,16
9	ppm	39	38,6	40,5	ppm	0,96	0,98	0,94	g/kg	247	247,9	246
10	mg/kg	13,7	13,4	13,9	mg/kg	0,64	0,64	0,64	mg/kg	226000	231000	221000
11					mg/kg	0,81	0,82	0,80	g/kg	252	251	253
12	mg/kg		15	15,6	mg/kg		0,54	0,54	mg/kg		250370	211186
13	mg/kg	24,7	24,5	24,8	mg/kg	0,68	0,68	0,68	mg/kg	244726	243671	245781
14												
15									mg/kg	238288	238204	238371
16	mg/kg	27,8	27,4	28,2	mg/kg	< 1	< 1	< 1	mg/kg	231000	228000	234000
17	mg/kg	25,19	26,03	24,35	mg/kg	1,04	1,01	1,07	mg/kg	214250	213000	215500
18	mg/kg	29,4	30,7	28	mg/kg	0,94	0,93	0,95	g/kg	237	241	233
19	mg/100g	2,78	2,75	2,81	µg/100g	87,2	87,1	87,2	g/100 g	24,5	24,8	24,2
20	mg/kg	33,7	32,7	34,7	mg/kg	0,98	0,96	1	g/kg	241	240	241
21	mg/kg	32,4	31,6	33,2	mg/kg	0,80	0,80	0,80	g/100g	23,5	23,4	23,5
22	mg/kg	30,9	30,33	31,47	mg/kg	0,97	0,95	0,99	mg/kg	220482	221566	219399
23									ppm	233.052	233.057	233.046

Part.	Unit	Co - Cobalt	Sample A	Sample B	Unit	Cr - Chrome	Sample A	Sample B	Unit	Cu - Copper	Sample A	Sample B
1	-	-	-	-	mg/kg	1,8	1,73	1,88	mg/kg	0,94	0,94	0,94
2	ppm	0,0890	0,09	0,09	ppm	3,07	3,06	3,07	ppm	2,19	2,15	2,23
3	mg/kg	0,28	0,25	0,3	mg/kg	3,12	3,09	3,15	mg/kg	2,15	2,18	2,12
4												
5	mg/kg		<0,1	<0,1	mg/kg		2,94	3,25	mg/kg		<5	<5
6	mg/kg	0,11	0,13	0,09	mg/kg	3,75	3,74	3,75	mg/kg	6,35	6,2	6,5
7	mg/kg	0,15	0,15	0,15	mg/kg	3,95	3,3	4,1	mg/kg	2,5	2,48	2,76
8	mg/kg	0,03	0,03	0,03	mg/kg	3,01	3,04	2,99	mg/kg	1,88	2,22	1,67
9	ppm	0,52	0,52	0,51	ppm	4,4	4,23	4,55	ppm	2,3	2,2	2,4
10	mg/kg	0,05	0,05	0,06	mg/kg	2,0	2,0	2,0	mg/kg	1,6	1,5	1,6
11					mg/kg	3,54	3,42	3,66	mg/kg	1,89	1,83	1,95
12	mg/kg		0,02	0,02	mg/kg		2,1	2,13	mg/kg	1,89	1,83	1,95
13	mg/kg				mg/kg				mg/kg		1,07	1,15
14					mg/kg	2,17	2,24	2,1	mg/kg	1,89	1,87	1,90
15					mg/kg	3,32	3,32	3,33	mg/kg	1,57	1,58	1,55
16	mg/kg	< 1	< 1	< 1	mg/kg	1,63	1,66	1,60	mg/kg	5,3	5,29	5,32
17	mg/kg	0,26	0,25	0,26	mg/kg	3	3,02	2,97	mg/kg	1,60	1,42	1,77
18	mg/kg	1,53	1,63	1,42	mg/kg	3,34	3,36	3,32	mg/kg	2,23	2,11	2,36
19	µg/100g	45,7	44,4	47	µg/100g	314	320	308	mg/kg	3,16	3,55	2,76
20	mg/kg	<0,1	<0,1	<0,1	mg/kg	2,7	2,72	2,64	µg/100g	233	260	206
21	mg/kg	0,032	0,034	0,031	mg/kg	3,39	3,41	3,37	mg/kg	2,1	2,24	1,96
22	mg/kg	**0,09**	0,09	0,1	mg/kg	**3,22**	3,13	3,3	mg/kg	1,70	1,65	1,75
23	ppm	0,23	0,24	0,22	ppm	3,41	3,43	3,39	mg/kg	2,47	2,43	2,52

Part.	Unit	Fe – Iron	Sample A	Sample B	Unit	I – Iodine	Sample A	Sample B	Unit	Li – Lithium	Sample A	Sample B
1	mg/kg	18,67	18,58	18,76	-	-	-	-				
2	ppm	28,1	28,1	28,2	ppm	< 100	< 100	< 100	-	-	-	-
3	mg/kg	22,1	22,1	22,1	mg/kg	1,72	1,7	1,73	ppm	1,21	1,22	1,21
4					mg/kg	9,15	9,4	8,9	mg/kg	1,24	1,32	1,17
5	mg/kg		<5	<5								
6	mg/kg	47,20	46,7	47,7								
7	mg/kg	34	33	35	mg/kg	1,2	1,1	1,2				
8	mg/kg	27,28	26,84	27,55	mg/kg	0,24	0,25	0,24	mg/kg	1,2	1,3	1,1
9	ppm	14	14,04	13,85	ppm	-						
10	mg/kg	18,6	18,7	18,5	mg/kg	5,1	5,0	5,1	ppm	1,8	1,63	1,91
11	mg/kg	29,9	29,8	30					mg/kg	0,82	0,83	0,80
12	mg/kg		16,5	17	mg/kg		1,25	1,12				
13	mg/kg	19,2	19,0	19,3	mg/kg	2,27	2,26	2,28	mg/kg		1,16	1,16
14									mg/kg			
15	mg/kg	28,92	28,92	28,91								
16	mg/kg	16,0	16,5	15,5	---	---	---	---				
17	mg/kg	22,4	22,35	22,45	mg/kg	1,41	1,41	1,41	mg/kg	2,52	2,49	2,55
18	mg/kg	375	385	365	mg/kg				mg/kg	1,34	1,33	1,35
19	mg/100g	2,69	2,74	2,65	µg/100g	179	178	180	mg/kg	0,94	0,95	0,93
20	mg/kg	25,7	26,7	24,6	mg/kg							
21	mg/kg	27,8	27,7	27,9		not calculated			mg/kg			
22	mg/kg	44,46	44,63	44,3	mg/kg					not calculated		
23									mg/kg			

Part.	Unit	Mg - Magnesium	Sample A	Sample B	Unit	Mn - Manganese	Sample A	Sample B	Unit	Mo - Molybdenum	Sample A	Sample B
1	mg/100g	9868,33	9863,16	9873,5	mg/kg	3,96	3,95	3,97	-	-	-	-
2	%	13,5	13,5	13,5	ppm	6,27	6,26	6,28	ppm	0,248	0,244	0,252
3	mg/kg	116500	117000	116000	mg/kg	5,7	5,76	5,64	mg/kg	0,02	0,02	0,02
4												
5	g/kg		124	124	mg/kg		<5	5,3	mg/kg		0,16	<0,100
6	%	10,03	9,96	10,1	mg/kg	7,00	7	7	mg/kg	0,45	0,45	0,44
7	mg/kg	125000	124000	128000	mg/kg	6,5	6,8	6,4	mg/kg	0,03	0,03	0,03
8	%	11,01	10,82	11,12	mg/kg	6,06	5,98	6,1	mg/kg	0,02	0,03	0,02
9	g/kg	127	123,7	130,8	ppm	5,9	5,57	6,15	ppm	0,05	0,04	0,06
10	mg/kg	110000	112000	108000	mg/kg	4,0	4,1	3,9	mg/kg	<0,05	<0,05	<0,05
11	g/kg	120	119	120	mg/kg	6,17	6,09	6,25	mg/kg		<0,060	<0,060
12	mg/kg		123371	120349	mg/kg		3,57	3,66	mg/kg			
13	mg/kg	115149	114947	115352	mg/kg	4,42	4,37	4,47				
14					mg/kg	3,85	3,9	3,8				
15	mg/kg	114706	115050	114361	mg/kg	5,94	5,95	5,93				
16	mg/kg	109000	108000	110000	mg/kg	3,63	3,68	3,57	mg/kg	< 1	< 1	< 1
17	mg/kg	110500	109500	111500	mg/kg	5,91	6,04	5,78	mg/kg	0,25	0,32	0,18
18	g/kg	119	118	120	mg/kg	4,56	4,73	4,39	mg/kg	0,05	0,04	0,06
19	g/100 g	11,6	11,6	11,5	µg/100g	654	667	641	µg/100 g	1,96	2,11	1,82
20	g/kg	114	114	114	mg/kg	6,3	6,37	6,22	mg/kg			
21	g/100g	11,1	11,0	11,1	mg/kg	6,28	6,28	6,28	mg/kg	0,024	0,021	0,027
22	mg/kg	112675	112888	112463	mg/kg	5,73	5,86	5,6	mg/kg	0,04	0,04	0,04
23	ppm	105.988	106.168	105.807	ppm	9,9	10,1	9,7				

Part.	Unit	Na - Natrium	Sample A	Sample B	Unit	Nd - Neodym	Sample A	Sample B	Unit	Ni - Nickel	Sample A	Sample B
1	mg/100g	28,28	28,43	28,12	-	-	-	-	-	-	-	-
2	ppm	428	426	430	ppm	< 0,25	< 0,25	< 0,25	ppm	1,20	1,17	1,22
3	mg/kg	305	307	304	mg/kg				mg/kg	0,47	0,48	0,46
4												
5	g/kg		0,51	0,53					mg/kg		1,65	0,72
6	%	<0,05	<0,05	<0,05					mg/kg	1,13	1,17	1,09
7	mg/kg	235	221	239	mg/kg	0,07	0,07	0,07	mg/kg	0,8	0,75	0,8
8	mg/kg	349,86	341,75	354,73					mg/kg	0,63	0,65	0,62
9	ppm	377	377,9	376,6	ppm	-			ppm	1,1	1,06	1,21
10	mg/kg	285	288	282	mg/kg	0,50	0,50	0,50	mg/kg	<0,5	<0,5	<0,5
11	mg/kg	470	470	470	mg/kg				mg/kg	2,00	1,89	2,10
12	mg/kg		292	300					mg/kg		0,47	0,46
13	mg/kg	301	297	305	mg/kg				mg/kg			
14												
15	mg/kg	311,2	309	313,5								
16	mg/kg	273	273	273	---	---	---	---	mg/kg	< 1	< 1	< 1
17	mg/kg	275,25	278	272,5	mg/kg	n.b.	n.b.	n.b.	mg/kg	5,53	5,5	5,55
18	mg/kg	352	345	359	mg/kg	0,83	0,84	0,84	mg/kg	16	17,1	14,8
19	mg/100g	28,1	28,7	27,5					µg/100g	84	96,7	71,4
20	mg/kg	290	297	283	mg/kg				mg/kg	0,56	0,57	0,54
21	mg/kg	323	320	327		not calculated			mg/kg	0,78	0,76	0,80
22	mg/kg	298	301,2	294,8	mg/kg				mg/kg	9,34	9,41	9,28
23	ppm	491	490	491								

Part.	Unit	P - Phosphorus	Sample A	Sample B	Unit	U - Uranium	Sample A	Sample B	Unit	Va - Vanadium	Sample A	Sample B
1	mg/100g	<10	<10	<10	-	-	-	-	-	-	-	-
2	ppm	294	293	295	ppm	< 1,00	< 1,00	< 1,00	ppm	0,410	0,4	0,42
3	mg/kg	85	85,8	84,3	mg/kg	0,31	0,32	0,3	mg/kg	0,32	0,34	0,29
4												
5	g/kg		0,2	0,2					mg/kg		0,41	0,44
6	%	<0,05	<0,05	<0,05								
7	mg/kg	94	95	92	mg/kg	0,41	0,39	0,43	mg/kg	0,3	0,3	0,3
8	mg/kg	269,33	202,32	281,47								
9	ppm	-			ppm	1,1	1,03	1,10	ppm	1,2	0,96	1,48
10	mg/kg	76,1	77,4	74,8	mg/kg	0,05	0,05	0,050	mg/kg	<0,5	<0,5	<0,5
11	mg/kg	92	92	91								
12	mg/kg		89	88,8	mg/kg		0,2	0,21	mg/kg		0,18	0,18
13	mg/kg	72,0	70,2	73,8	mg/kg				mg/kg			
14					mg/kg	0,33	0,33	0,32	mg/kg	0,34	0,34	0,33
15	mg/kg	103,8	103,8	103,8								
16	mg/kg	82,5	80	85	---	---	---	---	mg/kg	< 1	< 1	< 1
17					mg/kg	0,28	0,28	0,28	mg/kg	0,35	0,35	0,35
18	mg/kg	171	183	158	mg/kg	2,45	2,55	2,35	mg/kg	0,36	0,37	0,36
19	mg/100g	9,8	9,91	9,69	µg/100g	37,4	37,7	37,1	µg/100g	33,3	33,4	33,3
20	mg/kg				mg/kg	0,32	0,3	0,34	mg/kg	0,36	0,37	0,35
21	mg/kg	105	106	105		not calculated				not calculated		
22	mg/kg	106,2	102,8	109,7	mg/kg				mg/kg	0,33	0,33	0,33
23												

Part.	Unit	Y – Yttrium	Sample A	Sample B	Unit	Zn – Zinc	Sample A	Sample B
1	-	-	-	-	mg/kg	<1	<1	<1
2	ppm	0,236	0,24	0,23	ppm	0,324	0,33	0,32
3	mg/kg				mg/kg	1,97	1,99	1,95
4								
5					mg/kg		<5	<5
6					mg/kg	6,45	6,4	6,5
7	mg/kg	0,23	0,22	0,24	mg/kg	0,4	0,4	0,4
8					mg/kg	0,35	0,36	0,33
9	ppm	0,2	0,21	0,19	ppm	0,8	0,70	0,94
10	mg/kg	<0,5	<0,5	<0,5	mg/kg	<0,5	<0,5	<0,5
11					mg/kg	0,79	0,76	0,81
12					mg/kg		0,32	0,35
13	mg/kg				mg/kg	<0,50	<0,50	<0,50
14								
15								
16	mg/kg	< 1	< 1	< 1	mg/kg	< 1	< 1	< 1
17	mg/kg	0,19	0,19	0,19	mg/kg	0,52	0,53	0,5
18	mg/kg	0,36	0,38	0,33	mg/kg	2,6	2,94	2,25
19					µg/100g	<100	<100	<100
20	mg/kg				mg/kg	0,55	0,53	0,56
21		not calculated			mg/kg	<1	<1	<1
22	mg/kg				mg/kg	0,36	0,36	0,37
23					ppm	21,5	22,1	20,9

5.2 Homogeneity

5.2.1 *Repeatability standard deviation of results*

The Repeatability standard deviation of duplicate tests of the participants was calculated from the data documented in 5.1.

It is for Al (adjusted for two outlier) = 4,6% of X.

It is for Ca = 4,6 % of X (lt. ASU 00.00-144-Cheese: 2,0%).

It is for Fe (adjusted for one outlier) = 3,1 % of X (lt. ASU 00.00-144-Cheese: 6,1%).

It is for Co (adjusted for two outlier) = 12,2 % of X.

It is for Mn = 4,4 % of X (lt. ASU 00.00-144-Infant food soya: 4,3%).

And it is for V (adjusted for two outlier) = 6,2 % of X .

5.3 Analytical methods

Details from the participants

Teilnehmer / Participant	Method	Homogenisation	Digestion	Weight	Reference material	Calibration methode	Method is accredited	Remark
							yes/no	
1	AOAC 999.10	-	-	1.5g	-	-	yes	
2	ASU L-00.00-144 mod.	yes	microwave	0,3 - 1,0 g	---	ext. Std.	no	---
3	DIN EN ISO 11885	spatula	Ultraclave HNO3/H2O2	0,5 g	Plant	5-point-calibr.	yes	Al, Ca, Fe, Mg, Na, P,
3	DIN EN ISO 17294-2	spatula	Ultraclave HNO3/H2O2	0,5 g	Plant	2-point-calibr.	yes	Ba, Co, Cu, Li, Mn, Mo, U, V, Zn
3	§ 64 LFGB L 00.00-19/3	spatula	Ultraclave HNO3/H2O2	0,5 g	Plant	6-point-calibr.	yes	Cr, Ni
3	§ 64 LFGB L 00.00-93	spatula	TMAH	0,3 g	powdered milk	5-point-calibr.	yes	I
4	EN 15111			0,3 g	yes	yes	no	
5								
6	VDLUFA, MB III, 10.8.2	Milling Retsch ZM 200	microwave	1 g	Enquete samples	2 point-calibr.	yes	Al
6	DIN EN 15510:2007	Milling Retsch ZM 200	HCl / HNO_3	1 g	Enquete samples	2 point-calibr.	yes	Ca, Cu, Fe, Mg, Mn, Na, P, Zn
6	DIN EN 15621:2010	Milling Retsch ZM 200	microwave	0,5 g	Enquete samples	2 point-calibr.	yes	Co, Mo
6	DIN EN 14083:2003	Milling Retsch ZM 200	microwave	0,5 g	Enquete samples	2 point-calibr.	yes	Cr, Ni
7	DIN EN ISO 17294-2	no	EN 15763	400 mg	INCT-MPH-2	simple linear	yes	
8	PMDE01_018 ICP-MS	shake	acidic microwave digestion	0,5-1,0 g	Ultra Scientific	external Standard series	yes	
8	VDLUFA III 11.7.15 ICP-MS	shake	basic extraction	1,0-5,0 g	Ultra Scientific	see above	yes	
9	M761 1st ed (ICP-MS)			200 mg			no	Al, Ba, Co, Cr, Cu, Li, Mn, Mo, Ni, U, V, Y, Zn
9	M761 1st ed (ICP-OES)			200 mg			no	Ca, Fe, Mg, Na
10	ICP-OES	yes	microwave	0,5 - 0,6 g	no	external	yes	Al, Ba, Ca, Cr, Cu, Fe, Li, Mg, Mn, Na, Ni, P, V, Y, Zn
10	ICP-MS	yes	microwave	0,5 - 0,6 g	no	external	yes	Co, Mo, Nd, U
10	Cerimetric after Sandell-Kolthoff	yes	H2SO4/HNO3/HClO4	10 - 15 mg	no	external	yes	I

Teilnehmer / Participant	Method	Homogenisation	Digestion	Weight	Reference material	Calibration methode	Method is accredited	Remark
							yes/no	
11	ICP-MS	shake	microwave HNO3/H2O2	0.5 g	NIST8418 Wheat Gluten, ERM-CE278k Mussel tissue	external calibriation	no	137Ba, 53Cr, 63Cu, 55Mn, 60Ni, 66Zn; reference material certificate NIST8418 is expired but OK.
11	ICP-OES	shake	microwave HNO3/H2O2	0.5 g	NIST8418 Wheat Gluten, ERM-CE278k Mussel tissue	external calibriation	no	Ca 317 nm, Fe 259 nm, Mg 280 nm, Na 588 nm, P 177 nm; reference material certificate NIST8418 is expired but OK.
12	DIN EN ISO 11885	shake	microwave HNO3	0,3 g	watery	aqueous 6 point	yes	others
12	DIN EN 15111	shake	TMAH-solution	0,3 g	powdered milk	Aqueous 3 point	no	I
12	DIN EN ISO 17294/ 1, 2	shake	microwave HNO3	0,3 g	watery	aqueous 6 point	yes	U
13	DIN EN ISO 11885		VDLUFA Methods VII, 2.1.3	0,8g/25ml		external		others
13	DIN EN ISO 17294-2		VDLUFA Methods VII, 2.1.3	0,3g/25ml		external		I
14	ICP-OES		microwave	0.5g		external	yes	Cr, Cu, Mn
14	ICP-MS		microwave	0.5g		external	yes	U, V
15	ICP-OES		microwave HNO3	0,2 - 0,9 g		external	yes	
16	ASU L00.00-144 (ICP-OES)	---	ASU L00.00-144 *	0,2 g - 0,5g				
17	ICP- MS	shake	microwave	ca 500 mg		external	yes	others
17	AAS Flame	shake	microwave	ca 500 mg		external	yes	Ca, Fe, Mg, Na
17	ICP- MS	shake	Extaction TMAH	ca 500 mg		external	yes	I
18	DIN EN ISO 17294-2	shake	$HNO_3/H2O_2$	0,4	none	6-point calib.	yes	others
18	EN ISO 11885	shake	$HNO_3/H2O_2$	0,4	none	6-point calib.	yes	Ca, Mg, Na, P
19	Determination after micowave digestion with ICP-MS	spatula	Microwave digestion with HNO3/H2O2	0.5 g	Al standard solution 1000mg/L, Merck	external calibriation	yes	
19	Determination after extraction with mmungwithxtraktion mit TMAH per ICP-MS	spatula	Extraktion mit TMAH (Iod)	0.5 g	I standard solution prepared with KI	external calibriation	yes	Varying results at different sample preparations

Teilnehmer / Participant	Method	Homogenisation	Digestion	Weight	Reference material	Calibration methode	Method is accredited	Remark
							yes/no	
20	DIN EN ISO 17294-2:2005-02	spatula	MW/HNO_3	0,20 g	CertiPUR (Merck)	Standard	yes	others
20	MEBAK III, 2.Ed. 1996, Kap. 4.2	spatula	MW/HNO_3	0,20 g	CertiPUR (Merck)	Standard	yes	Na
21	ICP-MS after Microwave digestion	mix	microwave, HNO3	0,2 g	tea leaves (TL), bush branches and leaves (NCS), Pine Needles (PN)	ext. With IS In	yes	
22	ICP/MS	shake		0.7-0.9 g	none	external	yes	
23	ICP-MS							

6 Index of participant laboratories

Teilnehmer / Participant	Ort / Town	Land / Country
		Germany
		Germany
		Germany
		Germany
		Denmark
		Germany
		Germany
		Belgium
		Germany
		Germany
		Germany
		Germany
		Belgium
		Switzerland
		Germany
		Germany
		Germany
		Germany
		Netherlands
		Germany
		THAILAND
		Germany
		Germany
		Netherlands
		Germany

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

7 Index of literature

1. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
2. Verordnung / Regulation 882/2004/EU; Verordnung über amtliche Kontrollen / Regulation on official controls
3. 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
4. Richtlinie / Directive 1993/99/EU; über zusätzliche Maßnahmen im Bereich der amtlichen Lebensmittelüberwachung / on additional measures concerning the official control of foodstuffs
5. ASU §64 LFGB : Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung
6. DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche
7. The International Harmonised Protocol for the Proficiency Testing of Ananlytical Laboratories ; J.AOAC Int., 76(4), 926 – 940 (1993)
8. The International Harmonised Protocol for the Proficiency Testing of Ananlytical Chemistry Laboratories ; Pure Appl Chem, 78, 145 – 196 (2006)
9. Evaluation of analytical methods used for regulation of food and drugs;W. Horwitz; Analytical Chemistry, 54, 67-76 (1982)
10. A Horwitz-like funktion describes precision in proficiency test; M. Thompson, P.J. Lowthian; Analyst, 120, 271-272 (1995)
11. Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing; M. Thompson; Analyst, 125, 385-386 (2000)
12. Protocol for the design, conduct and interpretation of method performance studies; W. Horwitz; Pure & Applied Chemistry, 67, 331-343 (1995)
13. ASU §64 L 00.00-19/2 : Bestimmung von Eisen, Kupfer, Mangan und Zink mit der Atomabsorptionsspektrometrie (AAS) in der Flamme
14. ASU §64 L 00.00-19/3 : Bestimmung von Blei, Cadmium, Chrom und Molybdän mit Graphitofen-Atomabsorptionsspektrometrie (GFAAS) nach Druckaufschluß
15. ASU §64 L 00.00-19/5 : Bestimmung von Selen mit der Atomabsorptionsspektrometrie (AAS) -Hydridtechnik
16. ASU §64 L 00.00-93 : Bestimmung von Iod in Lebensmitteln - ICP-MS-Verfahren
17. ASU §64 L 00.00-127: Bestimmung von Zinn in Lebensmitteln mit der Flammen- und Graphitrohr-Atomabsorptionsspektrometrie (GFAAS) nach Druckaufschluß

18. ASU §64 L 00.00-128 :Bestimmung Zinn in Lebensmitteln mit der Massensprektrometrie mit induktiv gekoppeltem Plasma (ICP-MS) nach Druckaufschluß

19. ASU §64 L 31.00-10; Bestimmung der Gehalte an Natrium, Kalium, Calcium und Magnesium in Frucht- und Gemüsesäften – Atomabsorptionsspektrometrisches Verfahren (AAS)

20. RICHTLINIE 98/83/EG DES RATES vom 3. November 1998 über die Qualität von Wasser für den menschlichen Gebrauch

21. DIN EN 15765:2010; Bestimmung von Zinn in Lebensmitteln mittels ICP-MS nach Druckaufschluß
DIN EN 15763:2010; Bestimmung von Arsen, Cadmium, Quecksilber und Blei in Lebensmitteln mittels ICP-MS nach Druckaufschluß.

22. ASU §64 L 00.00-144 : Bestimmung der Mineralstoffe Ca, K, Mg, Na, P und S sowie der Spurenelemente Fe, Cu, Mn und Zn in Lebensmitteln mit ICP-OES.