

DLA
Dienstleistung
Lebensmittel
Analytik GbR

Evaluation Report
proficiency test

DLA 22/2014

**Contaminated Food:
Heavy Metals (Pb, Cd, Hg, As)**

Dienstleistung Lebensmittel Analytik GbR
Pinnberg 5
22927 Großhansdorf

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

Coodinator: Dr. G. Wichmann

Contents

1.	Introduction.....	3
2.	Realisation.....	3
2.1	Test material.....	3
2.1.1	Homogeneity.....	3
2.2	Test.....	4
2.3	Results.....	4
3.	Evaluation.....	5
3.1	Assigned value.....	5
3.2	Standard deviation.....	5
3.3	Outliers.....	5
3.4	Target standard deviation.....	5
3.4.1	General model (Horwitz).....	5
3.4.2	Precision experiment.....	5
3.5	z-Score.....	6
3.6	Quotient	6
3.7	Standard uncertainty.....	6
4.	Results.....	7
4.1	Lead in mg/kg	8
4.2	Cadmium in mg/kg.....	10
4.3	Mercury in mg/kg.....	12
4.4	Arsenic in mg/kg.....	14
5.	Documentation.....	16
5.1	Primary data in mg/kg.....	16
5.1.1	Lead.....	16
5.1.2	Cadmium	17
5.1.3	Mercury.....	18
5.1.4	Arsenic.....	19
5.2	DLA-portion-numbers and homogeneity.....	20
5.2.1	Repeatability standard deviation of participants.....	20
5.2.2	Comparison of sample number/test result.....	20
5.3	Analytical methods.....	21
6.	Index of participant laboratories.....	25
6.	Index of literature.....	26

1. Introduction

The participation in proficiency testing schemes is an essential element of the quality-management-system of every laboratory testing food and feed. 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. Realisation

2.1 Test material

The test material was a plant-powder mixture with a natural content of cadmium (Cd), lead (Pb), mercury (Hg) and arsenic (As) and with an added glucose content of approx. 8% for the homogeneity test.

Approximately 0,6 kg of the material were homogenized and then packaged in portions to approximately 5 g. The portions were numbered chronologically.

The detectability of the heavy metals ((Cd, Pb, Hg, As) was assured.

2.1.1 Homogeneity

The calculation of the repeatability standard deviation of the participants for cadmium was used as an indicator of homogeneity. The result is similar to the repeatability standard deviation of the German official method ASU § 64 LFGB L00.00-135 (11). The repeatability standard deviation of the participants is given in the documentation.

To verify the homogeneity of the test material glucose was added before homogenisation additionally. The homogeneity was examined with glucose/HPLC.

Sample	Glucose		
1	8,10	g/100g	
2	8,53	g/100g	
3	8,31	g/100g	
4	8,43	g/100g	
5	8,29	g/100g	
mean	8,3	g/100g	
Standard deviation	0,16	1,9%	

The homogeneity is considered verified with a standard deviation of 1,9%.

Additionally in the documentation the portion numbers are assigned graphically to the results of cadmium. There is no laboratory-independent trend recognizable in the results which could suggest inhomogeneity.

2.2 Test

Two portions of test material were sent to every participating laboratory in the 21nd week of 2014. The testing method was optional. The tests should be finished at 7th July 2014 the latest.

2.3 Results

The participants submitted their results in standard forms, which have been handed out with the samples. The finally calculated concentrations of As, Cd, Hg and Pb as average of duplicate determinations of both numbered samples was used for the statistical evaluation. Queried and documented were single results, recovery and the testing method used. Two participants have not submitted any results. All other participants have submitted at least one result in time.

3. Evaluation

3.1 Assigned value

Because the analysed material was no certified reference material the robust mean of the submitted results was used as assigned value $\bar{X}$ (6). The distribution of submitted results showed no hint for bimodal distribution or other reasons for a higher variability.

3.2 Standard deviation

For comparison to the target standard deviation a robust standard deviation (S_*) was calculated (6).

3.3 Outliers

Statistical outliers were determined by Mandel's-h-Statistic (5). Detected outliers were stated for information only, when z-score was < -2 or > 2 .

3.4 Target standard deviation

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

3.4.1 General model (Horwitz)

The relative target standard deviation in % of the assigned value is calculated according to the following equation.

$$\hat{\sigma} \text{ (%) } = 2^{(1-0,5 \log X)}$$

Out of this is calculated the target standard deviation in mg/kg

$$\hat{\sigma} = \bar{X} * \hat{\sigma} \text{ (%) } / 100.$$

3.4.2 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 :

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

The statistical evaluation was realised with the target standard deviation according to Horwitz.

The target standard deviation according to ASU § 64 LFGB L00.00-135 is given for information.

3.5 z-Score

To assess the results of the participants the z-score is used. It indicates about which multiple of the target standard deviation ($\hat{\sigma}$) the result (x) of the participant is deviating from the assigned value (X) (6).

Participants' z-scores were derived as:

$$z = (x - X) / \hat{\sigma} ;$$

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

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

3.6 Quotient $S^x/\hat{\sigma}$

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.

For this PT the results for lead, cadmium, mercury and arsenic showed a suitable comparability.

3.7 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 (6).

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

If $u_X \leq 0,3 * \hat{\sigma}$ the standard uncertainty of the assigned value needs not be included in the interpretation of the results of the PT (6). The Quotient $u_X/\hat{\sigma}$ is reported in the characteristics of the test.

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 - test - the characteristics are listed:

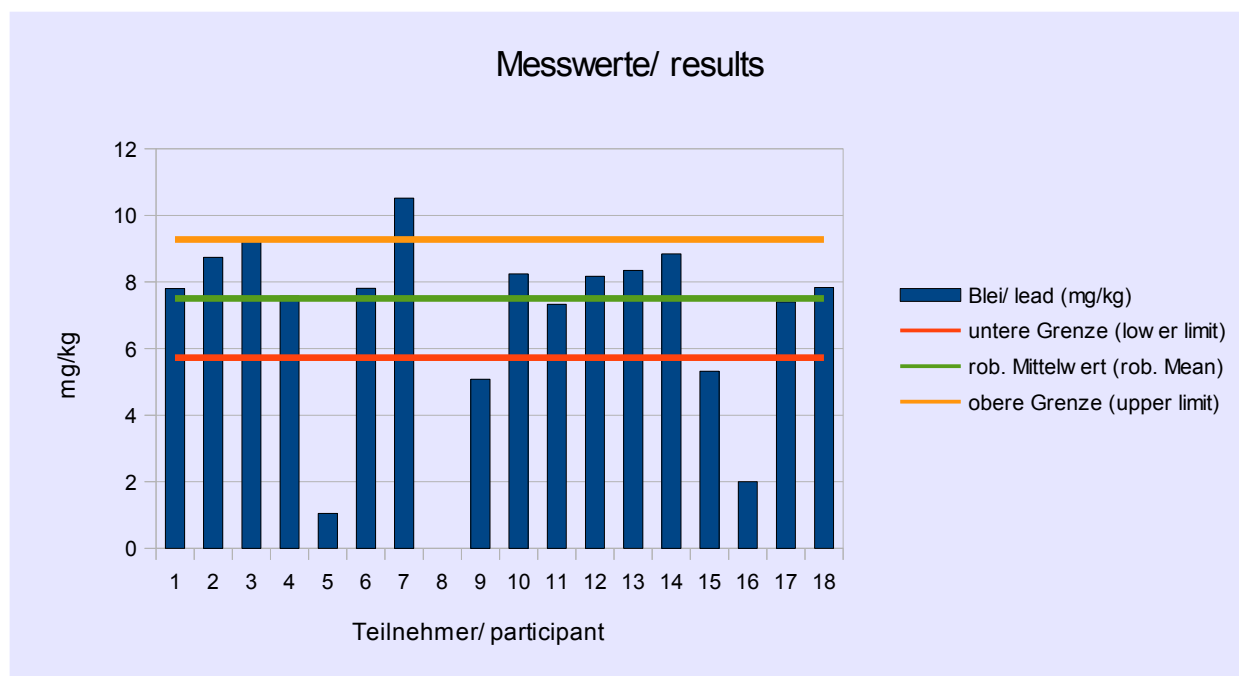
Number of results	
Number of outliers	
Mean	
Median	
Robust mean ($\bar{X}$)	
Robust standard deviation (S^*)	
Target standard deviation($\hat{\sigma}$) (Horwitz)	
Target standard deviation (ASU § 64 LFGB L00.00-135 for Information)	
Lower limit of target range	
Upper limit of target range	
Quotient $S^*/\hat{\sigma}$	
Standard uncertainty u_x	
Quotient $u_x/\hat{\sigma}$	
Number of results in the target range	

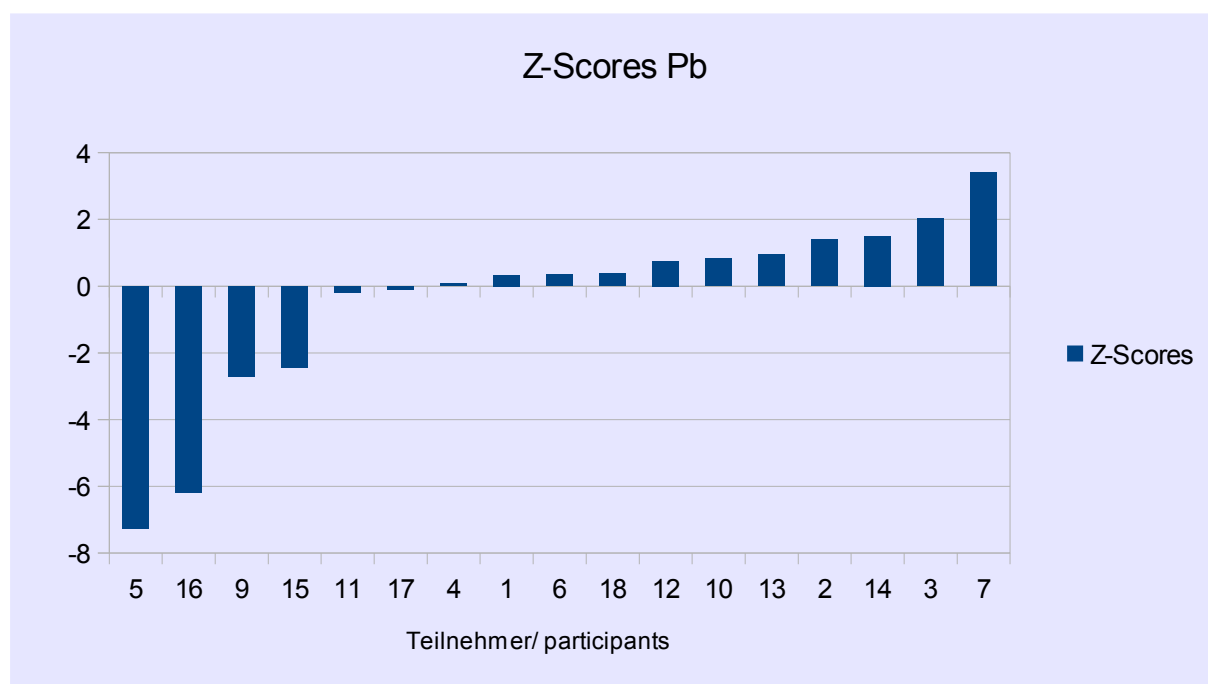
In the lower table - Laboratories - the individual results of the participating laboratories are listed:

Evaluation number	Result	Deviation	z-Score	Remarks
-------------------	--------	-----------	---------	---------

4.1 Lead in mg/kg

Characteristics	
Number of results	17
Number of outliers	2
Mean	7,14
Median	7,81
Robust mean ($\bar{x}$)	7,50
Robust standard deviation (S^*)	1,83
Target standard deviation ($\hat{\sigma}$) (Horwitz)	0,89
Target standard deviation (ASU § 64 LFGB L00.00-135 for Information)	0,70
Lower limit of target range ($\bar{x} - 2 \hat{\sigma}$)	5,73
Upper limit of target range ($\bar{x} + 2 \hat{\sigma}$)	9,27
Quotient $S^*/\hat{\sigma}$	2,07
standard uncertainty u_x	0,55
Quotient $u_x/\hat{\sigma}$	0,63
Number of results in the target range	13 (78%)



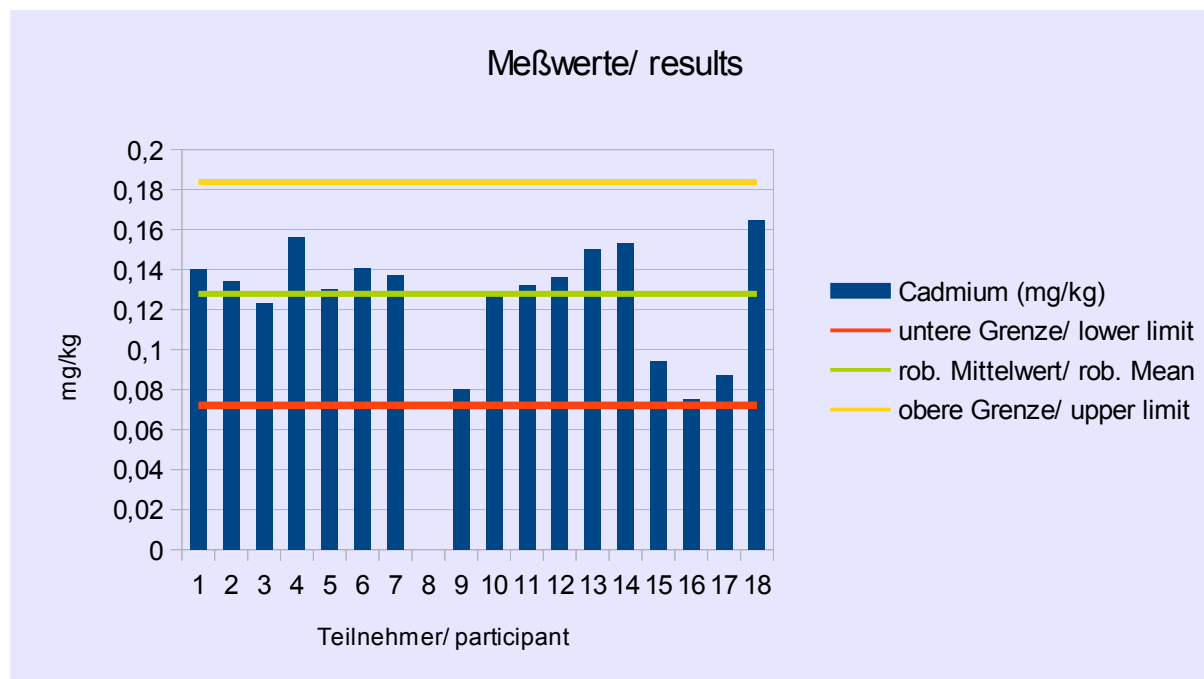


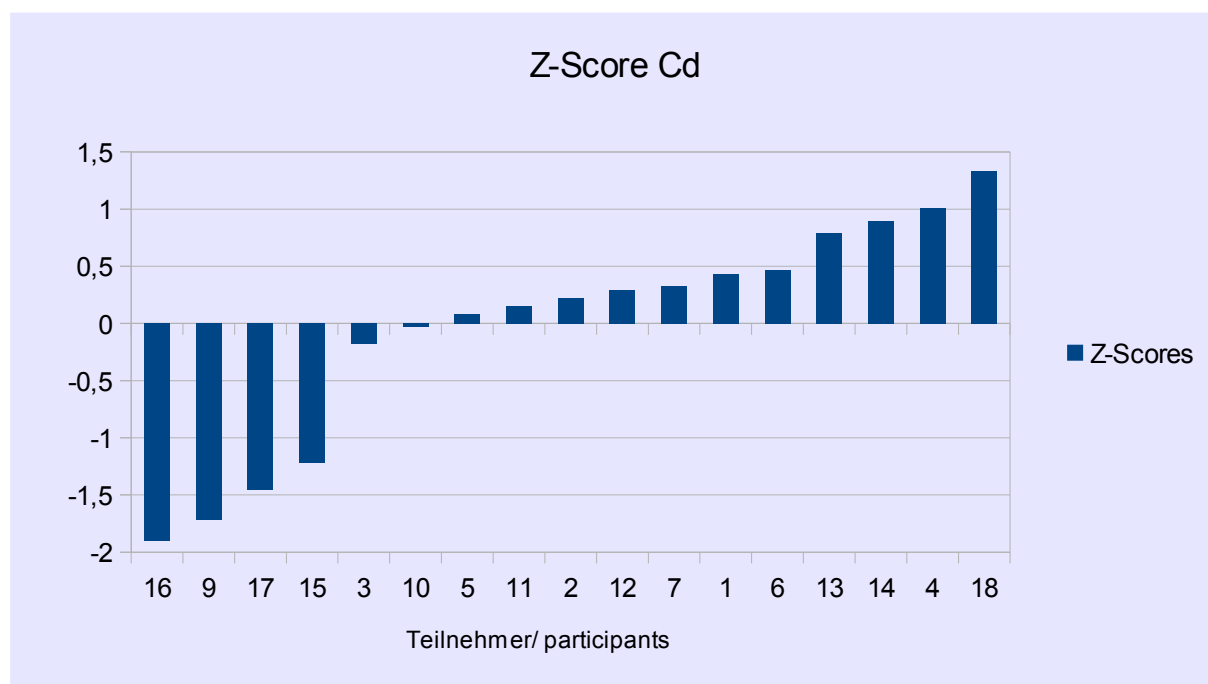
Laboratories

Teilnehmer/ participant	Blei/ lead (mg/kg)	Abweichung/ deviation	z-Score	Bemerkung/ remark
1	7,8	0,30	0,3	
2	8,74	1,24	1,4	
3	9,3	1,80	2,0	
4	7,58	0,07	0,1	
5	1,05	-6,45	-7,3	Ausreißer/ outlier
6	7,81	0,31	0,3	
7	10,52	3,02	3,4	
8				
9	5,08	-2,42	-2,7	
10	8,24	0,74	0,8	
11	7,33	-0,17	-0,2	
12	8,17	0,67	0,8	
13	8,35	0,84	1,0	
14	8,84	1,34	1,5	
15	5,32	-2,18	-2,5	
16	2,0	-5,50	-6,2	Ausreißer/ outlier
17	7,399	-0,10	-0,1	
18	7,84	0,33	0,4	

4.2 Cadmium in mg/kg

Characteristics	
Number of results	17
Number of outliers	0
Mean	0,127
Median	0,134
Robust mean ($\bar{x}$)	0,128
Robust standard deviation (S^*)	0,029
Target standard deviation ($\hat{\sigma}$) (Horwitz)	0,028
Target standard deviation (ASU § 64 LFGB L00.00-135 for Information)	0,011
Lower limit of target range ($\bar{x} - 2 \hat{\sigma}$)	0,072
Upper limit of target range ($\bar{x} + 2 \hat{\sigma}$)	0,184
Quotient $S^*/\hat{\sigma}$	1,0
standard uncertainty u_x	0,0088
Quotient $u_x/\hat{\sigma}$	0,31
Number of results in the target range	17 (100%)



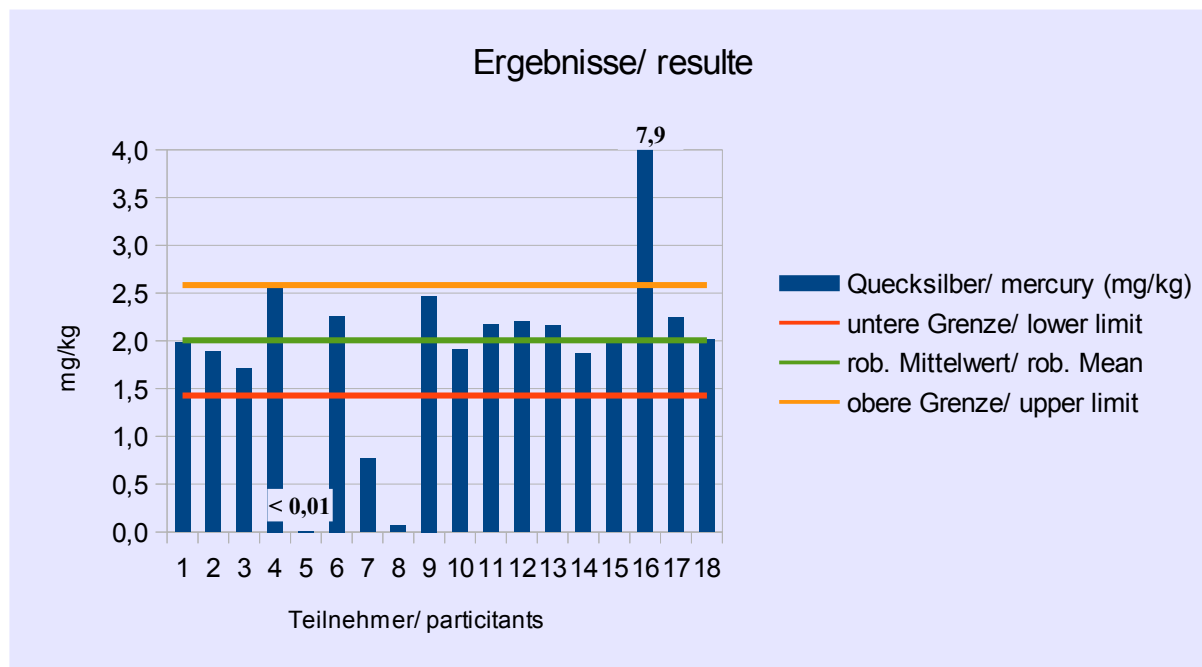


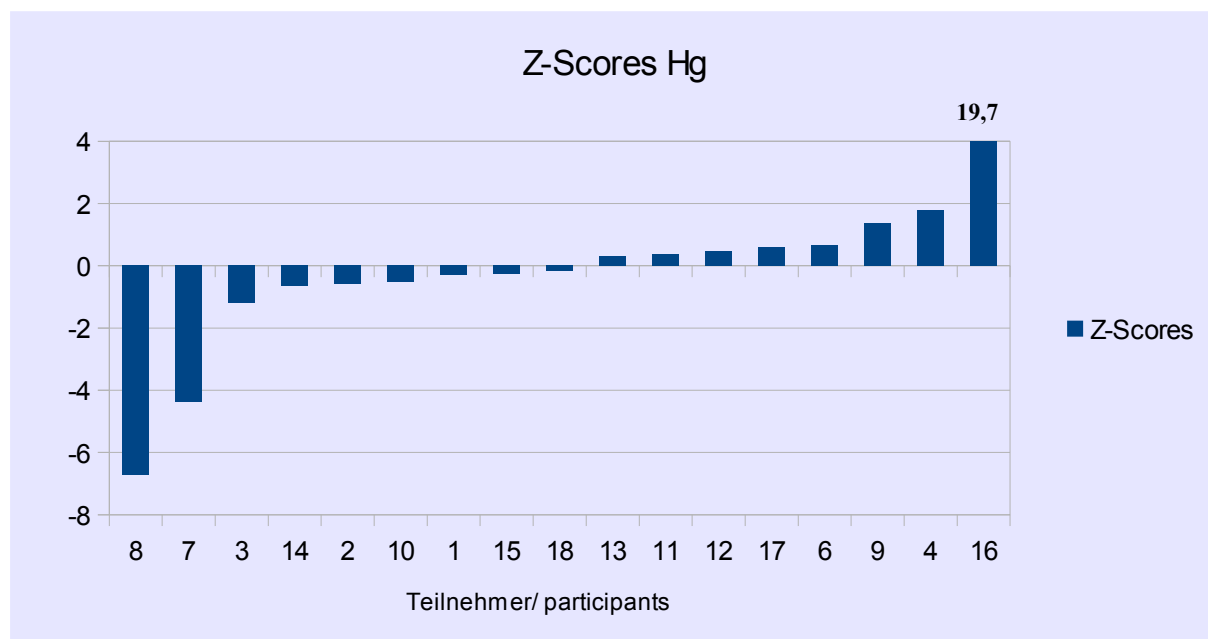
Laboratories

Teilnehmer/ participant	Cadmium (mg/kg)	Abweichung/ deviation	Z-Score	Bemerkung/ remark
1	0,14	0,01	0,4	
2	0,13	0,01	0,2	
3	0,12	0,00	-0,2	
4	0,16	0,03	1,0	
5	0,13	0,00	0,1	
6	0,14	0,01	0,5	
7	0,14	0,01	0,3	
8				
9	0,08	-0,05	-1,7	
10	0,13	0,00	0,0	
11	0,13	0,00	0,1	
12	0,14	0,01	0,3	
13	0,15	0,02	0,8	
14	0,15	0,03	0,9	
15	0,09	-0,03	-1,2	
16	0,08	-0,05	-1,9	
17	0,087	-0,04	-1,5	
18	0,17	0,04	1,3	

4.3 Mercury in mg/kg

Characteristics	
Number of results	17
Number of outliers	1
Mean	2,25
Median	2,02
Robust mean ($\bar{x}$)	2,07
Robust standard deviation (S^*)	0,37
Target standard deviation ($\hat{\sigma}$) (Horwitz)	0,296
Target standard deviation (ASU § 64 LFGB L00.00-135 for Information)	0,394
Lower limit of target range ($\bar{x} - 2 \hat{\sigma}$)	1,47
Upper limit of target range ($\bar{x} + 2 \hat{\sigma}$)	2,66
Quotient $S^*/\hat{\sigma}$	1,3
standard uncertainty u_x	0,11
Quotient $u_x/\hat{\sigma}$	0,38
Number of results in the target range	14 (82%)



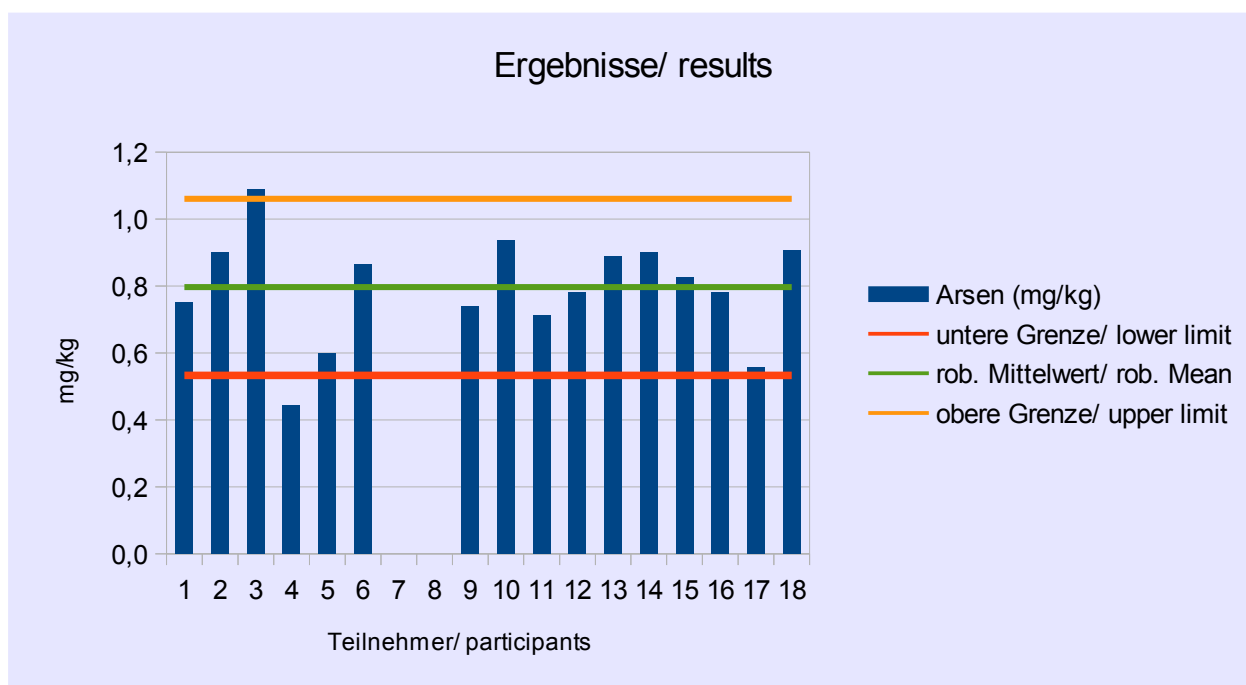


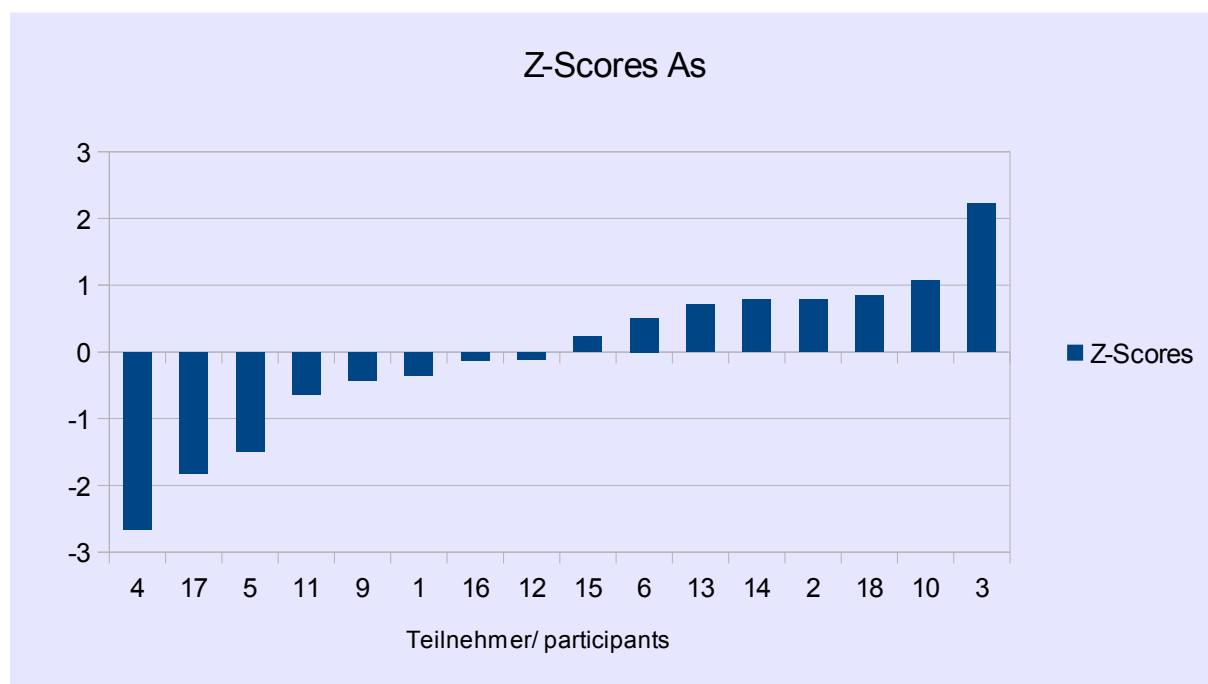
Laboratories

Teilnehmer/ participants	Quecksilber/ mercury (mg/kg)	Abweichung/ deviation	Z-Score	Bemerkung/ remark
1	1,98	-0,08	-0,3	
2	1,89	-0,17	-0,6	
3	1,71	-0,35	-1,2	
4	2,6	0,53	1,8	
5	< 0,01		(<-6,9)	result below target range
6	2,26	0,20	0,7	
7	0,77	-1,29	-4,4	
8	0,07	-1,99	-6,7	
9	2,47	0,41	1,4	
10	1,91	-0,15	-0,5	
11	2,17	0,11	0,4	
12	2,2	0,14	0,5	
13	2,16	0,10	0,3	
14	1,87	-0,19	-0,7	
15	1,99	-0,08	-0,3	
16	7,9	5,84	19,7	Ausreißer/outlier
17	2,245	0,18	0,6	
18	2,02	-0,05	-0,2	

4.4 Arsenic in mg/kg

Characteristics	
Number of results	16
Number of outliers	1
Mean	0,793
Median	0,805
Robust mean ($\bar{X}$)	0,797
Robust standard deviation (S^*)	0,156
Target standard deviation ($\hat{\sigma}$) (Horwitz)	0,132
Target standard deviation (ASU § 64 LFGB L00.00-135 for Information)	0,318
Lower limit of target range ($\bar{X} - 2 \hat{\sigma}$)	0,53
Upper limit of target range ($\bar{X} + 2 \hat{\sigma}$)	1,06
Quotient $S^*/\hat{\sigma}$	1,2
standard uncertainty u_x	0,049
Quotient $u_x/\hat{\sigma}$	0,37
Number of results in the target range	14 (88%)





Laboratories

Teilnehmer/ participant	Arsen (mg/kg)	Abweichung/ deviation	Z-Scores	Bemerkung/ remark
1	0,75	-0,05	-0,4	
2	0,9	0,10	0,8	
3	1,09	0,29	2,2	
4	0,44	-0,35	-2,7	Ausreißer/ outlier
5	0,6	-0,20	-1,5	
6	0,86	0,07	0,5	
7				
8				
9	0,74	-0,06	-0,4	
10	0,94	0,14	1,1	
11	0,71	-0,08	-0,6	
12	0,78	-0,01	-0,1	
13	0,89	0,09	0,7	
14	0,9	0,10	0,8	
15	0,83	0,03	0,2	
16	0,78	-0,02	-0,1	
17	0,557	-0,24	-1,8	
18	0,91	0,11	0,8	

5. Documentation

5.1 Primary data in mg/kg

5.1.1 Lead

Teilnehmer/ participant	Ergebnis/ result	DLA-Nr Probe I/ sample I	DLA-Nr Probe II/ sample II	Ergebnis I/ result I	Ergebnis II/ result II
	mg/kg			mg/kg	mg/kg
1	7,8	48	60	7,95	7,65
2	8,74	4	56	8,88	8,61
3	9,3	22	32	9,2	9,4
4	7,576	8	61	6,787	8,364
5	1,05	30	2	1,01	1,09
6	7,81	58	14	7,83	7,78
7	10,52	1	44	9,97	11,06
8					
9	5,08	6	29	4,9256	5,2427
10	8,24	37	64	7,93	8,54
11	7,33	18	36	7,63	7,03
12	8,17	28	59	7,813	8,526
13	8,35	17	35	8,04	8,65
14	8,84	3	49	8,802	8,878
15	5,32	15	62	5,37	5,27
16	2,0	22	63	2,01	2,03
17	7,399	5	39	7,305	7,493

5.1.2 Cadmium

Teilnehmer/ participant	Ergebnis/ result	DLA-Nr Probe I/ sample I	DLA-Nr Probe II/ sample II	Ergebnis I/ result I	Ergebnis II/ result II
	mg/kg			mg/kg	mg/kg
1	0,14	48	60	0,14	0,14
2	0,134	4	56	0,134	0,133
3	0,123	22	32	0,124	0,121
4	0,156	8	61	0,153	0,158
5	0,13	30	2	0,11	0,15
6	0,141	58	14	0,142	0,139
7	0,137	1	44	0,138	0,135
8					
9	0,08	6	29	0,0732	0,0853
10	0,127	37	64	0,126	0,128
11	0,132	18	36	0,136	0,127
12	0,136	28	59	0,144	0,127
13	0,15	17	35	0,15	0,15
14	0,153	3	49	0,15	0,156
15	0,094	15	62	0,090	0,098
16	0,075	22	63	0,073	0,077
17	0,087	5	39	0,075	0,099

5.1.3 Mercury

Teilnehmer/ paticipant	Ergebnis/ result	DLA-Nr Probe I/ sample I	DLA-Nr Probe II/ sample II	Ergebnis I/ result I	Ergebnis II/ result II
	mg/kg			mg/kg	mg/kg
1	1,98	48	60	2,11	1,85
2	1,89	4	56	1,93	1,85
3	1,71	22	32	1,65	1,76
4	2,595	8	61	2,48	2,71
5	<0,01	30	2	<0,01	<0,01
6	2,26	58	14	2,3	2,22
7	0,77	1	44	0,81	0,73
8	0,07	25	46	0,05	0,09
9	2,47	6	29	2,9385	2,005
10	1,91	37	64	1,99	1,82
11	2,17	18	36	2,2	2,14
12	2,202	28	59	2,156	2,247
13	2,16	17	35	2,19	2,13
14	1,872	3	49	1,925	1,819
15	1,985	15	62	1,963	2,006
16	7,9	22	63	7,7	8,11
17	2,245	5	39	2,355	2,135
18	2,015	26	45	2,02	2,01

5.1.4 Arsenic

Teilnehmer/ participants	Ergebnis/ result	DLA-Nr Probe I/ sammel I	DLA-Nr Probe II/ sample II	Ergebnis I/ result I	Ergebnis II/ result II
	mg/kg			mg/kg	mg/kg
1	0,75	48	60	0,8	0,7
2	0,901	4	56	0,896	0,905
3	1,09	22	32	0,97	1,21
4	0,444	8	61	0,421	0,467
5	0,6	30	2	0,56	0,64
6	0,864	58	14	0,87	0,857
7					
8					
9	0,74	6	29	0,7169	0,7585
10	0,938	37	64	0,955	0,92
11	0,713	18	36	0,707	0,719
12	0,783	28	59	0,812	0,754
13	0,89	17	35	0,92	0,86
14	0,9	3	49	0,876	0,924
15	0,827	15	62	0,806	0,848
16	0,78	22	63	0,756	0,81
17	0,557	5	39	0,516	0,597
18	0,908	26	45	0,906	0,91

5.2 DLA-portion-numbers and homogeneity

5.2.1 Repeatability standard deviation of participants

The repeatability standard deviation was calculated like under 5.1 documented.

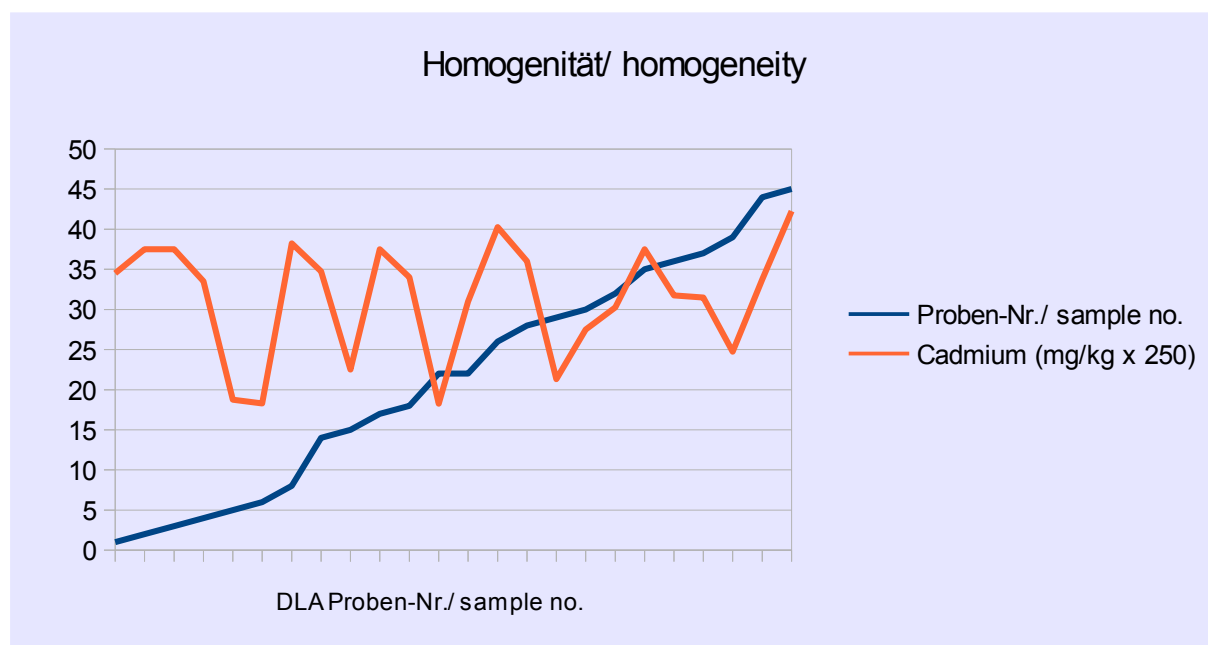
It is to 0,013 mg/kg = 10,3 % of X (= 0,13 mg/kg) for cadmium.

For comparison ASU § 64 LFGB L00.00-135 (11):

For cadmium (Scampi): 0,008 mg/kg = 9,5 % of X (= 0,08 mg/kg)

5.2.2 Comparison of sample number/test result

From the comparison of the rising sample-numbers and measured cadmium-concentrations (x250) can be seen on homogeneity.



5.3 Analytical methods

Inst.	Method	Details	Accredited
1	Pb, Cd, Hg, As: DIN EN ISO 17294-2	1. no 2. EN 15763 3. 400 mg 4. INCT-MPH-2 5. (simple) linear	yes
2	As, Cd, Pb: ASU L 00.00-19/1; DIN EN 15763	1. no 2. Microwave pressure digestion 3. 0,5 g 4. BCR [®] CRM - 281, NIST SRM 1515, IRMM BCR [®] 150 5. external calibration with intern standard	yes
	Hg: ASU L 00.00-19/1; ASU L 00.00-19-4	1. no 2. Microwave pressure digestion 3. 0,5 g 4. BCR [®] CRM - 281, NIST SRM 1515, IRMM BCR [®] 150 5. external calibration	yes
3	As: WI-D-247/1: ICP-MS, ⁷⁵ As with collision cell	1. shake for 1 minute 2. Microwave digestion with HNO ₃ /H ₂ O ₂ 3. 0,5 g 4. ERM-CE278k Mussel tissue 5. External - 5 standards	yes
	Cd: WI-D-247/1: ET-AAS	1. shake for 1 minute 2. Microwave digestion with HNO ₃ /H ₂ O ₂ 3. 0,5 g 4. ERM-CE278k mussel tissue 5. External - 5 standards	yes
	Hg: method based on WI-D-424: cold vapour furnace trapping ET-AAS	1. shake for 1 minute 2. Microwave digestion with HF/HNO ₃ /H ₂ O ₂ 3. 0,1 g 4. BR463 tuna fish 5. External - 5 standards	no
	Pb: WI-D-247/1: ICP-MS, ²⁰⁶⁻²⁰⁷⁻²⁰⁸ Pb (sum)	1. shake for 1 minute 2. Microwave digestion with HF/HNO ₃ /H ₂ O ₂ 3. 0,5 g 4. ERM-CE278k mussel tissue 5. External - 5 standards	yes
4	As, Cd, Pb: graphite tube AAS	1. yes 2. Pressure digestion, wet 3. approx. 0,25 g 4. yes 5. External	yes
	Hg: FIMS-AAS	1. yes 2. Pressure digestion, wet 3. approx. 0,25 g 4. yes 5. External	yes

Inst.	Method	Details	Accredited
5	As, Cd, Hg, Pb: ICP-MS	1. - 2. Microwave digestion 3. approx. 0,38 g 4. LCG NCSZC 2009 5. external	no
6	As, Pb: DIN EN ISO 17294-2	1. - 2. Microwave pressure digestion with H2O2/HNO3 3. 500 mg 4. NCS ZC 73013 spinach 5. -	yes
	Cd: DIN EN ISO 15586	1. - 2. Microwave pressure digestion with H2O2/HNO3 3. 500 mg 4. NCS ZC 73013 spinach 5. -	yes
	Hg: US EPA method 7473	1. - 2. - 3. 50 mg 4. ERM BD-150 skimmed milk powder 5. - 6. DMA (Direct mercury analyser)	yes
7	Cd: Q 192	1. - 2. Microwave 3. 0,5 g 4. - 5. -	yes
	Hg: Q 195	1. - 2. Microwave 3. 0,5 g 4. - 5. -	no
	Pb: Q 198	1. - 2. Microwave 3. 0,5 g 4. - 5. -	yes
8	Hg: NNI EN 13806 2003	1. - 2. - 3. 0,5 g 4. - 5. -	yes
9	As, Hg, Pb: Microwave digestion with HNO3 and 30% H2O2	1. yes 2. no 3. 0,5 g 4. FAPAS 07190 5. yes	yes
	Cd: Microwave digestion with HNO3 and 30% H2O2	1. yes 2. no 3. 1,0 g 4. FAPAS 07190 5. yes	yes
10	Cd, Pb: Z-GFAAS	1. mixed 2. Microwave 3. approx. 100 mg 4. CRM DORM 3 5. 4 point calibration	yes

Inst.	Method	Details	Accredited
	As: ICP-MS	1. mixed 2. Microwave 3. approx. 100 mg 4. CRM DORM 3 5. 6 point calibration	yes
	Hg: cV-GFAAS	1. mixed 2. Microwave 3. approx. 100 mg 4. CRM DORM 3 5. 10 point calibration	yes
11	Cd, Pb: ASU L 00.00-19/3 DIN EN 14083	1. mixed 2. LFGB §64 Abs. L 00.00-19/1 to 19/6 DIN EN 13805 3. 1 g 4. BCR 414 5. multi-point calibration	yes
	As: ASU L 00.00-19/6 DIN EN 14627	1. mixed 2. LFGB §64 Abs. L 00.00-19/1 to 19/6 DIN EN 13805 3. 1 g 4. BCR 414 5. multi-point calibration	yes
	Hg: ASU L 00.00-19/4 DIN EN 13806	1. mixed 2. LFGB §64 Abs. L 00.00-19/1 to 19/6 DIN EN 13805 3. 1 g 4. BCR 414 5. multi-point linear calibration	yes
12	As, Cd, Pb: ICP/MS	1. shaken 2. Microwave pressure digestion 3. 0,7 - 0,9 g 4. - 5. external calibration	yes
	Hg: cold vapour AAS	1. shaken 2. Microwave pressure digestion 3. 0,7 - 0,9 g 4. - 5. external calibration	yes
13	As, Cd, Pb: DIN EN 15763:2009 (mod.) and DIN EN 15763:2009 (mod.)	1. Mixed with plastic spatula 2. Microwave pressure digestion 3. 0,1 g 4. Wheat flour 5. external	no
	Hg: DIN EN 15763:2009 (mod.) and DIN EN 15763:2009 (mod.)	1. Mixed with plastic spatula 1. Microwave pressure digestion 2. 0,05 g 3. - 4. external	no
14	As: graphite tube AAS	1. - 2. Microwave 3. 0,3 g 4. Spinach powder and milk powder 5. Standard addition	yes
	Cd, Pb: graphite tube AAS	1. - 2. Microwave 3. 0,3 g 4. Spinach powder and milk powder 5. external calibration	yes

Inst.	Method	Details	Accredited
	Hg: FIMS	1. - 2. Microwave 3. 0,3 g 4. Spinach powder and milk powder 5. external calibration	yes
15	As, CD, PB: DIN EN ISO 17294-2	1. - 2. VDLUFA Method book VII 2.1.3 3. 0,8 g/ 25 ml 4. - 5. external	-
	Hg: DIN EN 1483	1. - 2. VDLUFA Method book VII 2.1.3 3. 0,8 g/ 25 ml 4. - 5. external	-
16	As, CD, PB: DIN EN ISO 17294-2 2005-02	1. Mixed with spatula 2. Microwave pressure digestion/HNO ₃ 3. 0,20 g 4. CertiPUR (Merck) 5. Standard	yes
	Hg: MEBAK III, 2. edition 1996, chapter 5.7	1. Mixed with spatula 2. Microwave pressure digestion/HNO ₃ 3. 0,20 g 4. CertiPUR (Merck) 5. Standard	yes
17	As: VDLUFA, MB VII, 2.2.2.10	1. Mühl Retsch ZM 200 2. Microwave 3. 0,5 g 4. Enquete sample 5. 3-point calibration	yes
	Cd, Pb: DIN EN 15550-2007	1. Mühl Retsch ZM 200 2. Microwave 3. 0,25 g 4. Enquete sample 5. 3-point calibration	yes
	Hg: VDLUFA, MB VII, 2.2.2.9	1. Mühl Retsch ZM 200 2. Microwave 3. 0,25 g 4. Enquete sample 5. 3-point calibration	yes
18	As, Cd, Hg, Pb: -	1. - 2. - 3. - 4. - 5. -	-

1. = Homogenization
 2. = Hydrolization/ digestion
 3. = Sample weight
 4. = Reference material
 5. = Calibration

6. Index of participant laboratories

Teilnehmer/ Participant	Ort/ Location
BECIT GMBH	Bitterfeld-Wolfen
Bioanalytik Weißenstephan	Freising Weißenstephan
Bitburger Braugruppe GmbH	Bitburg
BML - Laboratorien GmbH	Singen
CR3-Kaffeeveredelung GmbH	Bremen
DB Lab A/S	Odense (Denmark)
FARCOS Srl	Garbagnate (MI) (Italy)
Indikator GmbH	Wuppertal
Kantonales Laboratorium Frauenfeld	Frauenfeld (Switzerland)
Knjaz Miloš A.D.	Arandelovac (Serbia)
Kommission der EG	Geel (Belgium)
Laboratorium der Urkantone	Brunnen (Switzerland)
LAVES Niedersachsen	Cuxhaven
LUFA Nord-West	Hamel
LUFA Nord-West	Oldenburg
Lufa Nordrhein-Westfalen	Münster
SGS Institut Fresenius GmbH	Taunusstein
TLR International Laboratories	Rotterdam (Netherlands)
Versuchs- und Lehranstalt für Brauerei in Berlin e.V.	Berlin
Zentrales Institut des Sanitätsdienstes der Bundeswehr Kiel	Kronshagen

6. 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. ISO 13528:2005; Statistische Verfahren für Eignungsprüfungen durch Ringversuche
7. The International Harmonised Protocol for the Proficiency Testing of Analytical Laboratories ; J.AOAC Int., 76(4), 926 - 940 (1993)
8. The International Harmonised Protocol for the Proficiency Testing of Analytical 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 function describes precision in proficiency test; M. Thompson, P.J. Lowthian; Analyst, 120, 271-272 (1995)
11. ASU §64 LFGB : L00.00-135; Bestimmung von Arsen, Cadmium, Quecksilber und Blei in Lebensmitteln (Jan. 2011)
12. ASU §64 LFGB : L00.00-19/3; Bestimmung von Blei, Cadmium, Chrom und Molybdän in Lebensmitteln (Juli 2004)
13. ASU §64 LFGB : L00.00-19/4; Bestimmung von Quecksilber in Lebensmitteln (Dezember 2003)
14. ASU §64 LFGB : L00.00-19/6; Bestimmung von Gesamtarsen in Lebensmitteln (Juli 2001)

Printed on 100% Recycling-paper