REPORTS OF FINNISH ENVIRONMENT
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1 INTRODUCTION

The Finnish Environment Institute (SYKE) carried out the proficiency test for leaching testing of the contaminated soil sample with the one stage and the two stage batch leaching test. These tests are used as compliance tests in evaluating the waste landfill disposal. The contaminated soil sample was distributed to the participants for determination of leached amounts of inorganic substances (As, Ba, Cd, Cr, Cu, Fe, Mo, Ni, Pb, Sb, Se and Zn), dissolved organic carbon (DOC), and determination of conductivity and pH value of the eluates in April 2008. The test was carried out in accordance with the international guidelines, ISO/IEC Guide 43 1 [1], ILAC Requirements [2], ISO 13528 [3] and IUPAC Recommendations [4]. SYKE is the Proficiency Testing Provider no. PT01 accredited by the Finnish Accreditation Service. The proficiency testing service in SYKE conforms to the requirements of the Guide ISO/IEC 43-1:1997. However, the organising of proficiency tests for the leaching testing is not included in the accredited scope.

2 ORGANIZING THE PROFICIENCY TEST

2.1 Responsibilities

Organizing laboratory:	Finnish Environment Institute (SYKE) Hakuninmaantie 6, 00430 Helsinki Fax. +358 20 490 2890
Coordinator :	Irma Mäkinen Tel. +358 40 823 9956 e-mail:irma.makinen@ymparisto.fi
Experts:	Kati Vaajasaari, Golder Associates Oy (the expert: leaching tests) Raija Ivalo, Pirkanmaa Regional Environment Centre (the expert: sample preparation and taking part in testing) Olli Järvinen (the analytical expert: ICP-MS, conductivity, pH, DOC) Timo Sara-Aho (the analytical expert: ICP-OES)

2.2 Participants

In total, 18 laboratories from Finland, Denmark and Greece participated in this proficiency test (Appendix 1).

2.3 Samples and their delivery

The test sample was prepared from the soil sample contaminated by wood preservatives (CCA), in which the main pollutants were arsenic, chromium and copper. The material was air dried and sieved through a 0.5 mm and divided into 32 bottles.

The sample was delivered 7 April 2008. It was requested to be analyzed and reported before 27 August 2008.

The requested tests were as follows:

Codes	Test
R1	One stage leaching test[1)] (analysis of metals, DOC, conductivity and pH)
R21	Two stage leaching test - L/S2[2)] (analysis of metals, DOC, conductivity and pH)
R22	Two stage leaching test - L/S10[3)] (analysis of metals, DOC, conductivity and pH)

[1)] at the liquid to solid ratio 10 litres/kg
[2)] at the liquid to solid ratio of 2 litres/kg
[3)] at the liquid to solid ratio of 2 litres/kg + 8 litres/kg

The feasibility of the soil sample for one stage and two stage batch leaching test was obtained by analysis of inorganic substances and DOC (Appendix 2). The ratio R1/R22 (R1 = L/S10 in one stage test, R22 = L/S10 in two stage leaching test) was mainly about one. Thus the sample was regarded to be appropriate for the batch leaching tests.

Five participants were interested to carry out also the percolation test. However, in preliminary testing, the soil texture was obtained to be too fine-grained and, therefore the percolation test was regarded technically unsuitable for studied soil material.

2.4 Homogeneity testing and distribution of particle size

Homogeneity was tested by analyzing the total concentrations of As, Cr, Cu and Zn in the soil sample as duplicate determinations from six subsamples. (Appendix 3). According to the homogeneity test results, the soil sample was considered homogenous.

Particle size distribution was also tested from one sub sample. The results showed, that the percentage of the particles below 200 µm was 51.2 % (Appendix 3). The sample material was homogenous and it was expected to be easier to treat in comparing with waste samples in general.

2.5 Comments sent by the participants

The participants did not comment the carrying out the proficiency test.

2.6 Analytical methods

2.6.1 Batch leaching tests

The standard methods EN 12457-2 (one stage batch leaching test) and EN 12457-3 (two stage batch leaching test) were used to determine the leaching properties of studied components from the soil sample [5 and 6].

The one stage leaching test provides information on leaching of studied components under experimental conditions at liquid to solid ratio of 10 l/kg dry matter. In two stage leaching test, the liquid to solid ratio is 2 l/kg dry matter in the first step, and subsequently of 8 l/kg dry matter in a second step.

The concentrations of analytes from the leaching test eluates are expressed as the leached amounts (mg/kg) relative to the total mass of the soil sample.

2.6.2 Differences in participants' procedures

There were some differences in procedures used by the participants as follows (Appendix 4).

- Shaking or mixing equipment varied
  - mainly rotary shaker or end-over-end table was used.
- The amount of the recovered eluate varied:.
  - one stage test: 0.1-0.9 l
  - two stage test: 1st step- 0.17- 0.3 l; 2nd step – 0.15-1.35 l.
- Time between agitation and separation varied as well:
  - one stage test:15 -1440 min
  - two stage test:15-130 min.
- There were also differences in time of filtration:
  - one stage test:10 – 80 min
  - two stage test:10-260 min (in total).

There were also some differences in the measured pH-value, conductivity and the temperature mainly due to the differences in testing procedures. The soil texture was fine-grained that could have had effect on differences in the duration of separation and time between agitation and separation.

For the measurement of metals ICP-MS or ICP-OES was mainly used. DOC was measured by using IR-spectrometry (Appendix 4).

2.7 Data treatment

2.7.1 Testing of outliers and normality of data

Before the statistical treatment, the data was tested according to the Kolmogorov-Smirnov normality test and the outliers were rejected according to the Hampel test for calculation of the mean value. In this PT a few results were rejected in use of robust statistics, too. In most cases only one result was rejected.

2.7.2 Assigned values and target value for total deviation

The robust mean of the reported results was used as the assigned value for all measurands (Appendix 5). The assigned value was not estimated in determination of Cd, Mo and Se because of low leached amounts of these components (Appendix 5).

However, the assigned value was indicative in the cases, in which the standard uncertainty of the assigned value was higher than 0.3 • the target total deviation used in calculating of z score [3]. The uncertainty for the assigned values of Ba, Fe, Ni and Zn was not estimated because of relatively low leached amounts in the leaching experiment. In determination of Cu in two stage batch test (R21 and R22) the uncertainty of the assigned value was high due fairly low Cu content and the assigned value was indicative.

The target value for the total deviation used for calculation of the z scores varied from 25 % to 40 % at the 95 % confidence level (Table 1, Appendix 7). The deviations were depending on the substance and their leached amounts. The target value for the total deviation was not estimated in determination of inorganic substances with low leachability (Ba, Cd, Fe, Mo, Ni, Pb, Se and Zn).

2.7.3 Evaluation of performance

The performance evaluation was carried out by using the z scores (Appendix 7 and 10). The z score was calculated using the following equation:

$$z = \frac{(x_i - X)}{s}$$

where:

x_i = the participant's value
X = the assigned value
s = the target value for total deviation/2.

z scores were interpreted as follows:

$\lvert z \rvert \leq 2$	satisfactory results
$2 < \lvert z \rvert < 3$	questionable results
$\lvert z \rvert \geq 3$	unsatisfactory results.

3 RESULTS

3.1 Variation of the results

All participants reported the results of duplicate measurements. Table 1 shows the results of the duplicate determination in the cases, in which the laboratory performance has been evaluated.

Table 1. Results of duplicate determinations (ANOVA statistics)

Analyte	Code	Unit	Ass. value	s_w %	s_b %	s_t %	r	R
As	R1	mg/kg	28,0	1,4	10	10	**4,0**	**28**
	R21	mg/kg	8,10	4,6	14	14	**13**	**40**
	R22	mg/kg	29,5	4,7	6,3	7,9	**13**	**22**
Conductivity	R1	mS/m	0,985	5,7	17	18	**16**	**51**
	R21	mS/m	2,12	8,0	31	32	**23**	**91**
	R22	mS/m	1,19	16	33	37	**45**	**105**
Cr	R1	mg/kg	1,52	4,7	28	28	**13**	**79**
	R21	mg/kg	0,641	4,1	25	25	**12**	**71**
	R22	mg/kg	1,52	5,1	14	15	**14**	**42**
Cu	R1	mg/kg	3,13	6,9	24	25	**20**	**71**
	R21	mg/kg	1,05	7,5	46	47	**21**	**133**
	R22	mg/kg	2,72	4,3	45	46	**12**	**130**
DOC	R1	mg/kg	45,2	2,9	14	15	**8,2**	**42**
	R21	mg/kg	27,8	3,3	16	17	**9,3**	**48**
	R22	mg/kg	51,1	4,2	26	27	**12**	**76**
pH	R1		5,77	2,2	5,3	5,8	**6,2**	**16**
	R21		6,03	2,0	7,5	7,8	**5,7**	**22**
	R22		6,09	2,4	7,3	7,7	**6,8**	**22**
Sb	R1	mg/kg	0,940	3,5	18	18	**9,9**	**51**
	R21	mg/kg	0,357	4,1	14	14	**12**	**40**
	R22	mg/kg	0,990	1,4	24	24	**4,0**	**68**

in which,
Ass.value = the assigned value, s_w = the within laboratory standard deviations, s_b = the between laboratory standard deviation, s_t = the total standard deviation, r = the repeatability and R = the reproducibility

In general, in the duplicate determinations, the ratio of the laboratory standard deviation and the total standard deviation (s_t/s_w) varied from 2 to 17. It was highest for leached amounts determined from the two stage batch leaching test, in the first step R21 (Cr and Cu) and in the cumulative leaching test R22 (DOC, Cu and Sb). Also, in determination of leached amount of As from the one stage leaching test, the ratio was abnormally high (s_t/s_w = 7). The ratio s_t/s_w should be between 2 and 3 for the robust methods [7].

The standards EN 12457-2 and EN 14257-3 [5 and 6] recommend the relationships for calculating of the repeatability limit (r) and reproducibility limit (R) with 95 % statistical confidence as well as the typical expected values for r and R (Table 2).

Table 2. Relationships for the repeatability and reproducibility in the one stage and in the two stage leaching test

Parameter	Relationship	Typical expected value (r) in the one stage leaching test	Typical expected value (R) in the two stage leaching test
Repeatability, r	$r = 2 \bullet \sqrt{2} \bullet s_w$	24 %	40 % (1st step) 50 % (2nd test)
Reproducibility, R	$R = 2 \bullet \sqrt{2} \bullet s_t$.	72 %	100 %

In both the one stage leaching test and the two stage leaching test, the repeatability (r) was lower than the typical expected value (Table 1 and 2). Further, the reproducibility (R) was higher than the expected value for Cr in the one stage leaching test. In addition, in the two stage leaching test R was higher than the expected value for Cu in the 1st step and for conductivity as well as for Cu in the 2nd test. Thus the obtained repeatability and reproducibility were mainly lower than the expected values.

Table 3. Summary of the proficiency test 4/2008

Analyte	Sample	Unit	Ass. val.	Mean	Mean rob.	Md	SD rob	SD rob, %	Num. of labs	2*Targ SD%	Accepted z-val%
As	R1	mg/kg	28	28.50	27.66	28.40	3.37	12,2	15	25	87
	R21	mg/kg	8,1	8.04	8.06	8.04	0.95	11,8	15	25	93
	R22	mg/kg	29,5	29.92	29.53	29.48	1.74	5,9	15	25	80
Ba	R1	mg/kg	0,055	0.079	0.068	0.055	0.032	48	12		
	R21	mg/kg	0,039	0.049	0.042	0.031	0.029	67,6	12		
	R22	mg/kg	0,057	0.088	0.064	0.061	0.023	36	12		
Cd	R1	mg/kg		0.75	0.75	0.73	0.69	91,7	14		
	R21	mg/kg		0.090		0.090			14		
	R22	mg/kg		0.29		0.29			14		
conductivity	R1	mS/m	0,985	0.98	1.03	1.00	0.18	17,2	15	30	64
	R21	mS/m	2,12	2.29	2.17	2.11	0.55	25,2	15	30	80
	R22	mS/m	1,19	1.23	1.24	1.15	0.44	35,4	14	30	62
Cr	R1	mg/kg	1,52	1.55	1.52	1.50	0.39	25,1	14	40	86
	R21	mg/kg	0,641	0.65	0.65	0.60	0.16	25,3	14	40	86
	R22	mg/kg	1,52	1.43	1.53	1.50	0.29	18,7	14	40	79
Cu	R1	mg/kg	3,13	2.99	3.18	3.29	0.58	18,1	13	40	77
	R21	mg/kg	1,05	1.06	1.08	1.17	0.44	40,9	14	40	57
	R22	mg/kg	2,72	2.93	2.89	3.18	1.39	48,1	14	40	57
DOC	R1	mg/kg	45,2	45.39	46.10	46.00	7.60	16,5	11	25	82
	R21	mg/kg	27,8	27.89	28.31	27.80	4.91	17,4	10	25	80
	R22	mg/kg	51,1	52.50	54.24	52.40	15.43	28,4	10	25	70
Fe	R1	mg/kg	0,861	0.98	0.93	0.95	0.51	54,3	13		
	R21	mg/kg	0,234	0.28	0.25	0.19	0.17	67,3	12		
	R22	mg/kg	0,939	1.22	1.22	1.00	0.83	67,5	12		
Mo	R1	mg/kg		0.006		0.006			15		
	R21	mg/kg		0.001		0.001			13		
	R22	mg/kg		0.006		0.006			13		
Ni	R1	mg/kg	0,041	0.041	0.053	0.060	0.043	80,5	15		
	R21	mg/kg	0,007	0.007	0.008	0.009	0.002	23,9	14		
	R22	mg/kg	0,024	0.034	0.017	0.013	0.009	52,6	14		
Pb	R1	mg/kg	0,042	0.042	0.047	0.049	0.035	74,3	14		
	R21	mg/kg	0,023	0.023	0.026	0.027	0.020	76,4	15		
	R22	mg/kg	0,049	0.056	0.056	0.051	0.042	75,2	15		
pH	R1		5,77	5.74	5.90	5.90	0.31	5,3	15	6,9	80
	R21		6,03	6.06	6.03	6.06	0.43	7,1	15	6,7	73
	R22		6,09	6.12	6.09	6.09	0.45	7,4	14	6,5	71
Sb	R1	mg/kg	0,94	0.94	0.94	0.97	0.18	19,5	13	30	92
	R21	mg/kg	0,357	0.36	0.36	0.37	0.050	13,8	15	30	93
	R22	mg/kg	0,99	0.98	0.99	1.00	0.24	24,2	15	30	80
Se	R1	mg/kg		0.008		0.008			12		
	R21	mg/kg		0.003		0.003			12		
	R22	mg/kg		0.007		0.007			12		
Zn	R1	mg/kg	0,192	0.26	0.23	0.20	0.098	43,5	15		
	R21	mg/kg	0,073	0.075	0.078	0.079	0.034	43,8	15		
	R22	mg/kg	0,237	0.22	0.20	0.16	0.11	56,8	15		

Ass. val.	the assigned value
Mean	the mean value
Mean rob	robust mean
Md	the median value
SD %	the standard deviation as percent
SD rob	the robust standard deviation
SD rob %	the robust standard deviation as percents
Num of Labs	the number of participants
2•Targ. SD%	the total target deviation at 95% confidence level
Accepted z-val%	the satisfactory z values: the results (%), where $\lvert z \rvert \leq 2$.

The main pollutants in the soil sample were arsenic (As), chromium (Cr) and copper (Cu). As the Table 3 shows, the robust standard deviation was low (12 % -14 %) for arsenic and relatively low for chromium, too. However, the results varied most for copper (27 % - 50 %). The differences in the duration of separation and the time between the agitation and separation in two step leaching procedure might have had effect on pH and conductivity, and that way affecting the leaching behaviour of Cu; for instance. The pH of a leaching system can be affected in an uncontrolled manner when system is exposed to the atmosphere. On the other hand, the results were rather concurrent with each other also in determination of DOC and Sb. In the analysis of substances with low leachability (Ba, Cd, Fe, Ni, Pb, Se and Zn), the robust variation varied up to about 70 % - 90 %.

3.2 Comparison of the results obtained in the one stage and the two stage test

When the one stage and two stage test resembles each other, the ratio of the leached amounts in the one stage leaching test and the cumulative leached amount in the two stage leaching test (in this PT the ratio R1/R22) is often close to one. The laboratory 5 carried out only the two stage test and the laboratory 15 only the one stage test (Appendix 9). Therefore the ratio R1/R22 was not calculated by using the results reported by these laboratories.

The ratio R1/R22 was close to one for the main pollutants (As, Cr and Cu) and for DOC (Fig. 1). The ratios calculated from the results reported by the laboratories 11 and 16 showed some deviations.

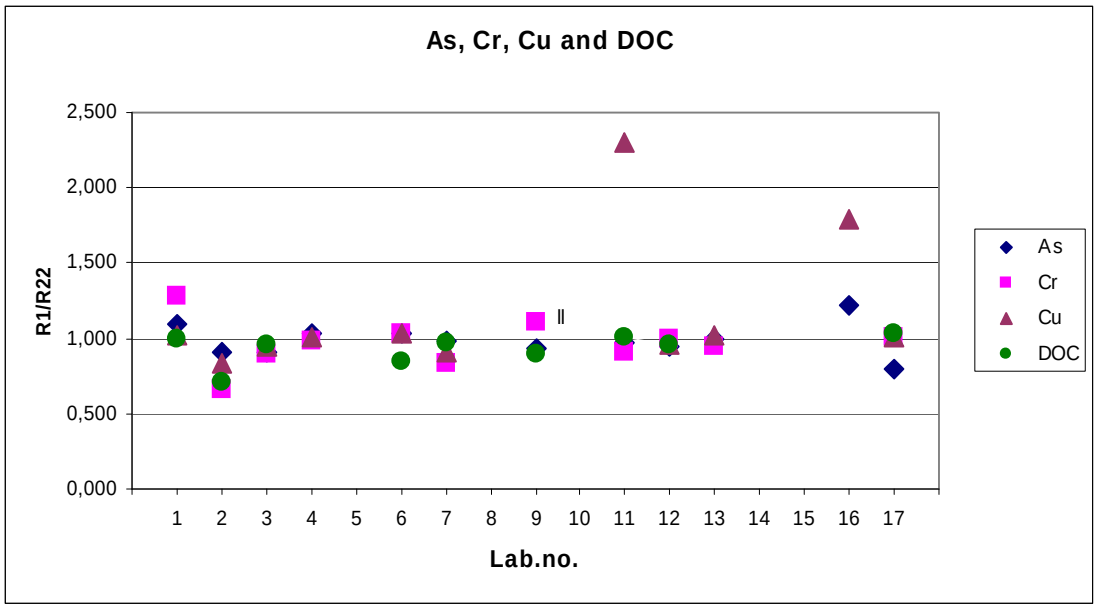

Fig 1. The ratio (R1/R22) of the leached amounts of main pollutants and DOC in the one stage leaching test and the cumulative leached amount in the two stage leaching test

4 IMPORTANT ASPECTS IN THE BATCH LEACHING TESTS

The leaching of contaminants from waste is controlled by several parameters an external factors [6]. The key factors can be briefly addressed as follows:

- Shaking procedure
    - The standard recommends to use as agitation devices end-over-end table or rotary table, which the most participants used. In sufficient shaking can lead to underestimated results.
- pH value and CO_2
    - The sensitivity of leaching to relatively small changes in pH can be significant [6]. In the one stage test the pH values of the eluates varied from 4.7 (lab 15) to 7.5 (lab 17). In the two stage test in the 1st step pH value varied from 5.35 to 7.1 and in the 2nd step from 5.45 to 7.3. In this PT pH values might have had effect on leaching behavior of e.g. Cu.
    - The pH of the leaching system can be affected in an uncontrolled manner when system is exposed to the atmosphere. Due to uptake of CO_2 and increased levels of CO_2, the pH may change during the leaching experiment.
- Particle size
    - The particle size distribution of the test material can affect the leachability of studied substances [8]. In general, the smaller the particle size, the larger the surface area of the particles in contact with water, producing higher leachability of contaminants. In this PT about 20 % of the particles were smaller than 20 µm and about 52 % of the particles were smaller than 200 µm (Appendix 3). The fine particles might have also had an effect on separation procedure by increasing the time of separation.
- Separation
    - Almost all participants used a membrane filter (0.45 µm) in separation the solid phase from the liquid phase. Some laboratories used a glass fiber filter or centrifugation as a preliminary step in filtration procedure.
    - There were also differences in the separation time. In particular, the time of separation differed most between participants in the 2nd step of the two stage test. The differences in the duration of the separation and in the time between the agitation and separation in two stage leaching procedure might have had caused some changes in pH and conductivity.
- Analysis
    - The laboratories used mainly ICP-MS or ICP-OES for analysis of metals. DOC was mainly measured by IR spectrometry.

5 EVALUATION OF PERFORMANCE

The evaluation of the participants was based on z scores which were calculated using the estimated target values for the total deviation. The calculated z scores are presented with the results of each participant (Appendix 7) and the summary of z scores is presented in Appendix 10.

The total target deviation was between 25 % and 40 %. The total target deviation was estimated on the basis of the type of the sample, the leached amounts of analytes and the results of the homogeneity testing and the uncertainties of the assigned values. Performance was not evaluated for substances with low leachability (Ba, Cd, Fe, Ni, Pb, Se and Zn).

In this PT from 78 % of the results were satisfactory. The texture of the sample was fine-grained and homogeneous, and the preparation of subsamples for the participants were regarded to be of uniform quality based on the homogeneity test results of main pollutant concentrations in subsamples. Consequently, the results can be regarded to be satisfactory. The proficiency test for the batch leaching test was carried out the second time in Finland.

6 SUMMARY

In spring 2008 the Finnish Environment Institute (SYKE) carried out the proficiency test for leaching testing of a contaminated soil sample with the one stage and the two stage batch leaching tests. In total, 18 laboratories participated in the proficiency test.

The test material was a soil sample contaminated by wood preservatives (CCA), in which the main pollutants were arsenic, chromium and copper. The sample was fine-grained sand, the percentage of the particles below 200 µm was 51.2 %.

The proficiency test was performed for the one stage batch leaching test (L/S 10, EN 12457-2) and the two stage batch leaching test (L/S2 and L/S10, EN 12457-3). From the leaching test eluates the inorganic substances (As, Ba, Cd, Cr, Cu, Mo, Ni, Pb, Sb, Se and Zn) and DOC, pH and conductivity were determined.

The participants tested the samples using the procedure described in the EN standards. However, there were differences, e.g., in agitation devices, in filtering procedure, and in duration of separation of the solid phase from the liquid phase. These differences might have had effect on the results only in very few cases.

When the one stage and two stage test resembles each other, the ratio of the results obtained in the one stage leaching test and the cumulative result obtained in the two stage leaching test (in this PT the ratio R1/R22) is often close to one. In this PT for the main pollutants (As, Cr and Cu) and DOC, the ratio R1/R22 was close to one. For other studied components, there were some deviations from one.

The evaluation of the laboratory performance was carried out using z score. In this proficiency test 78 % of the results were satisfactory, when the total target deviation varied from 25 % to 40 % at the 95 % confidence interval.

The SYKE proficiency test for leaching tests was carried out the second time. The test material texture was homogenous and consequently the results of the proficiency test can be regarded to be satisfactory.

According to the Finnish national waste landfill acceptance criteria, the batch leaching tests are mainly recommended to be used for quality control of wastes, and only in exceptional cases for basic characterization of waste materials. Primarily for basic characterization, the percolation test (CEN/TS 14405) shall be used. Above all, it is important, that laboratories develop ability to carry out the percolation test as well.

7 YHTEENVETO

SYKE järjesti pätevyyskokeen jätteen kaatopaikkakelpoisuuden arvioimiseen käytettävistä liukoisuustesteistä (yksi- ja kaksivaiheinen ravistelutesti) keväällä 2008. Pätevyyskokeeseen osallistui yhteensä 18 laboratoriota.

Testausnäytteeksi valittiin suolakyllästeellä (CCA) pilaantunut maa. Pääasialliset haitalliset yhdisteet maassa olivat arseeni, kupari ja kromi. Näyte oli hienojakoista ja suurin osa partikkeleista (51,2 %) oli pienempiä kuin 200 µm.

Osallistujat käyttivät liukoisuustestimenetelminä 1-vaiheista ravistelutestiä (L/S 10, EN 12457-2) ja 2-vaiheista ravistelutestiä (L/S2 ja L/S10, EN 12457-3). Menettelyt vastasivat pääosin standardiohjeita, mutta esim. näytteen ravistelutavassa ja nesteen ja kiinteän materiaalin suodattamiseen kuluneissa suodatusajoissa oli eroavuuksia, jotka saattoivat jonkin verran vaikuttaa tuloksiin. Ravistelutestiuutteista määritettiin epäorgaanisten aineiden (As, Ba, Cd, Cr, Cu, Mo, Ni, Pb, Sb ja Zn) sekä orgaanisen hiilen (DOC) liuenneet määrät sekä uutteiden pH ja sähkönjohtavuus.

Sekä 1-vaiheisessa että 2-vaiheisessa testissä toistettavuus ja uusittavuus olivat pääasiallisesti pienempiä kuin standardiohjeissa esitetyt rajat. Poikkeavuuksia esiintyi 2-vaiheisen testin toistettavuudessa ja uusittavuudessa. Toistettavuus oli raja-arvoa suurempi ensimmäisen vaiheen (tässä vertailussa tulokset R21) sähkönjohtavuustuloksissa. Uusittavuus ylitti tämän rajan kaksivaiheisen testin 1-vaiheen kuparituloksissa sekä 2-vaiheen (R22) sähkönjohtavuus- ja kuparituloksissa.

1-vaiheisen ravistelutestin (L/S10, R1) ja 2-vaiheisen ravistelutestin kumulatiivista (R22) eri komponenttien liuennutta määrää voidaan verrata keskenään. Yleensä testit vastaavat hyvin toisiaan, kun suhde R1/R22 on lähellä yhtä. Tärkeimpien haitallisten aineiden osalta (arseeni, kromi ja kupari) suhde R1/R22 oli pääasiallisesti lähellä yhtä. Muille analyyteille suhde R1/R22 erosi yhdestä edellä mainittua useammin.

Laboratorioiden pätevyyden arviointi tehtiin z-arvon avulla (z score). Tässä vertailussa tulosaineistossa oli tyydyttäviä tuloksia 78 %, kun vertailuarvosta (the assigned value) sallittiin 25 % – 40 % poikkeama 95 % merkitsevyystasolla.

Vertailuun käytetty maa-aines oli hienojakoista ja kohtalaisen tasalaatuista. Myös maa-aineksen orgaanisen aineksen määrä oli alhainen. Maa-aineksen hienoaines saattoi vaikeuttaa ja hidastaa ravistelutestin jälkeisen seoksen suodattamista. Pätevyyskokeen tuloksia voidaan pitää tyydyttävänä.

SYKE järjesti toisen kerran ravistelutestejä (EN-12457) koskevan pätevyyskokeen. Ravistelutestimenetelmiä käytetään kaatopaikkakelpoisuuden määrittämisessä laadunvalvontaan sekä poikkeustapauksissa pienempien jätevirtojen perusmäärittelyyn. Ensisijaisena perusmäärittelytestinä on kaatopaikkakelpoisuuskriteerien (Vna 202/2006) mukaisesti esitetty käytettäväksi kolonni- eli läpivirtaustestiä (CEN/TS 14405). Ensisijaisena laadunvalvontatestinä suositellaan käytettävän 2-vaiheista ravistelutestiä (Vna 202/2006). Ravistelumenetelmien käyttötarve ja testejä tekevien laboratorioiden määrä tulee lisääntymään tulevaisuudessa. Tästä syystä liukoisuustestimenetelmien vertailun järjestämistä pidetään tärkeänä myös jatkossa.

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APPENDIX 1. PARTICIPANTS IN THE PROFICIENCY TEST SYKE 4/2008

ALS Scandinavia, AB, Luleå, Sweden

Boliden Harjavalta Oy, Harjavalta, Finland

Boliden Kokkola Oy, Kokkola, Finland

Ekokem Oy Ab, Riihimäki, Finland

Eurofins Environment Sweden AB, Lidköping, Sweden

Eurofins Miljø A/S, Vejen, Denmark

KCL Kymenlaboratorio Oy, Kuusankoski, Finland

Laboratory of Metallurgy, National technical University of Athens, Greece

Labtium Oy, Kuopio, Finland

Lapin Vesitutkimus Oy, Rovaniemi, Finland

Neste Oil Oyj, Konsernin tutkimus ja kehittäminen, Porvoo, Finland

Novalab Oy, Karkkila, Finland

Outokumpu Tornio Works, Tornio, Finland

Pirkanmaan ympäristökeskus, Tampere, Finland

Ramboll Analytics Oy, Lahti, Finland

SGS Inspection Services Oy, Hamina, Finland

Stora Enso Oyj, Tutkimuskeskus, vesi- ja hivenaineanalyysit, Imatra, Finland

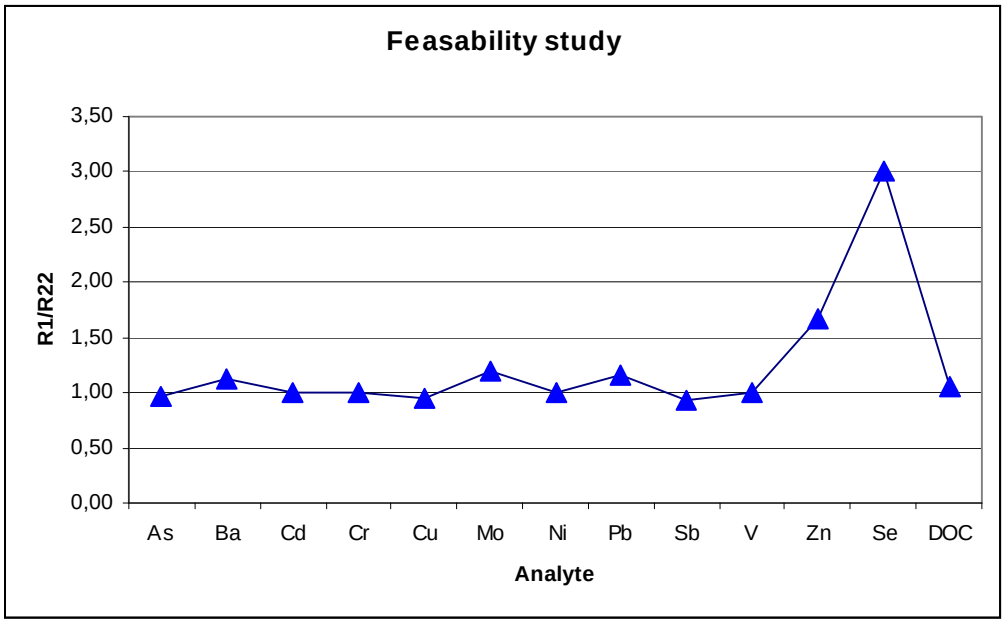
Suomen ympäristöpalvelu Oy, Oulu, Finland

APPENDIX 2. RESULTS OF THE FEASIBILITY STUDY

The feasibility of the soil sample for one stage and two stage batch leaching tests was obtained by analysis of inorganic substances and DOC .

The value R1/R22 (L/S10 in one stage test/L/S10 in two stage leaching test) was mainly about 1 except in analysis of Zn and Se because of low metal content.

Code	R1	R21	R22	R1/R22
Analyte	mg/kg	mg/kg	mg/kg	
As	27,97	7,88	28,7	**0,97**
Ba	0,54	0,046	0,48	**1,13**
Cd	0,0001	0,0001	0,0001	**1,00**
Cr	1,33	0,59	1,32	**1,01**
Cu	1,51	0,86	1,6	**0,94**
Mo	0,006	0,001	0,005	**1,20**
Ni	0,007	0,003	0,007	**1,00**
Pb	0,014	0,007	0,012	**1,17**
Sb	1,1	0,43	1,17	**0,94**
V	0,14	0,03	0,14	**1,00**
Zn	0,94	0,11	0,56	**1,68**
Se	0,003	0,001	0,001	**3,00**
DOC	40	21	38	**1,05**



APPENDIX 3. TESTING OF HOMOGENEITY AND DISTRIBUTION OF PARTICLE SIZE

Homogeneity

The soil sample was distributed into 32 bottles. Homogeneity was tested as duplicate determinations of metals (As, Cr, Cu and Zn) from six bottles. The analytical variation s_a and the between bottle variation s_{bb} was calculated using one-way variance analysis. For this proficiency test the results were recalculated by taking into account the IUPAC procedure for the treatment of homogeneity testing data and the target values of total deviation [4].

Soil sample	Conc. mg/kg	s_t%	s_t	s_a	s_a/s_t	Was s_a/s_t <0.5?	s_{bb}	s_{bb}^2	c	Was s_{bb}^2<c?
As	2760	5	138.0	42.93	0.311	Yes	17.53	307.2	6793	Yes
Cr	1452	5	72.6	34.73	0.478	Yes	14.18	201.0	3022	Yes
Cu	1094	5	54.7	17.95	0.328	Yes	7.33	53.70	1121	Yes
Zn	26.35	5	1.318	0.338	0.256	Yes	0.14	0.019	0.531	Yes

where,
Conc. = Concentration

s_t = target deviation, total target deviation/2

s_t% = target deviation as percent, total target deviation/2

s_a = analytical deviation, mean standard deviation of results in a sub sample

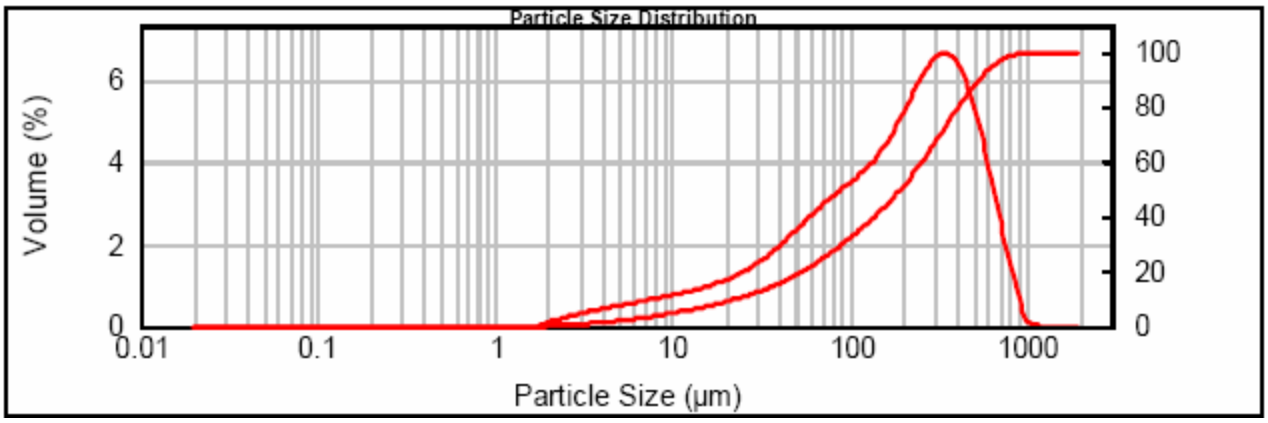
s_{bb} = between-sample deviation, standard deviation of results between sub samples

$c = F1 \bullet s_{all}^2 + F2 \bullet s_a^2$
where,
$s_{all}^2 = (0.3 s_t)^2$
F1 = 1.88 when the number of sub samples is 10, F2 = 1.01 when the number of sub samples is 10
F1 = 1.94 when the number of sub samples is 9, F2 = 1.11 when the number of sub samples is 9

Conclusion: In each case s_a/s_t<0.5 and s_{bb}^2 < c. The sample material was homogenous.

Particle size

Distribution of particle size was tested from one sample bottle by using laser diffraction.



The percentage of the particles below 200 µm was 51.2 %.

APPENDIX 4. METHODS

1) Production of eluate – one stage leaching test

Lab	Mass M_w (kg)	Extraction and shaking	T (°C)	V of leachant (l)	Liquid/Solid separation	t(min) Agitation/ Separation	t(min) Separation	VE (l) Filtered eluate	pH; γ_{25} mS/m; T (°C)
1	According to EN 12457 (2-3)								
2	0,0906	PE bottle, 1 l Rotary shaker, 8 rpm	25	0,9	Centifugation, filtration 0,45 µm	15	60	0,6	6,05; 0,9 24,6
3	0,0909	Glass bottle, 1 l, End-over-end, 10 rpm	24	0,899	Buchner, filtration membrane 0,45 µm	15			5,8 and 5,7; 1,0; 24.0 and 23.8
4	0,09	HDPE bottle, 1 l Rotary shaker, 10 rpm	23-25	0,9	Microfiltration, esther filters 0,45 µm	15	30	0,8	6,03 and 5,85; 1,0 and 0,9; 23,9 and 23,4
6	0,09	Glass bottle, 1 l, Rotary shaker, 11 rpm	23	0,9	Filtration, cellulose nitrate, 0,45 µm	< 30			5,9; 0,97 and 0,86
7	0,0907, 0,0903		20,2-21,1	0,9	Filtration, Whatman Me 25 and membrane, 0,45 µm	15	40	0,48, 0,50	5,46 and 5,51; 2,11 and 2,06
9	0,0908	HDPE bottle, 1 l, End-over-end, 5-10 rpm	Room	0,899	Centifugation, membrane 0,45 µm	20	10		5,9; < 1
10	According to EN 12457 (2-3)								
11	0,1	PE bottle, 1 l, Rotary shaker, 7 rpm	23		Filtration, cellulose nitrate, 0,45 µm	15	30	0,300	5,71 and 5,61; 0,92 and 0,96; 23
12	0,09	HDPE bottle, 1 l, End-over-end, 10 rpm	22	0,889	Filtration, glass fiber filter, and membrane, 0,45 µm	15	25		6,21 and 6,18; 1,098 and 1,089; 22,5 and 22,4
13	0,0905	HDPE bottle, 1 l Rotary shaker, 10 rpm	23	0,9	Filtration, Whatman 27 and membrane 0,8µm	15 and 55	40 and 45	0,15	5,41 and 5,44; 1,49 and 1,34; 23
14	0,09	PE bottle, 1 l Rotary shaker, 8 rpm	20	0,900	Filtration, membrane 0,45 µm	20	18 and 19	0,860	5,80 and 5,79; 0,95 and 0,92; 26,5 and 26,4
15	0,09	Glass bottle, 1l, Rotary shaker, 8,5 rpm	23	0,9	Filtration, membrane 0,45 µm	1440	60	0,12	4,70 and 5,24; 1,91 and 1,18; 23
17	0,0905	Glass bottle, 1 l, End-over-end, 8 rpm	23	0,8995	Filtration, glass filter and membrane 0,45 µm	1	80	0,872	7,5; 2,42

2) Production of eluate – two stage leaching test

Lab	Mass M_w (kg)	Extraction and shaking	T (°C)	V of leachant (l)	Liquid/Solid separation	t(min) Agitation/ Separation	t(min) Separation	VE (l) Filtered eluate	pH; γ 25 mS/m; T (°C)
1	According to EN 12457 (2-3)								
2	0,1762	L_2: PE bottle 0,5 l L_8: PE bottle 2 l Rotary shaker, 8 rpm	24	L_2: 0,349 L_8: 1,4	Centifugation , filtration 0,45 µm	15	L_2: 55 L_8: 60	VE_1: 0,3 VE_2: 1	L/S2: 5,65; 1,7; 24,3 L/S8: 5,95; 0,8; 24,3
3	0,101	Glass bottle, 1 l, End-over-end, 10 rpm	23-24	L_2: 0,199 L_8: 0,799	Buchner, filtration membrane 0,45 µm	15		VE_1: 0,181 and 0,183	L/S2: 6,1 and 6,4; 1,7 and 1,9; 23,1 and 23,0 L/S8: 6,1 and 6,6; 0,9 and 1,2; 23,7 and 23,9
4	0,176	L_2: HDPE bottle 0,5 l L_8: HDPE bottle 2 l Rotary shaker, 10 rpm	23-25	L_2: 0,349 L_8: 1,4	Microfiltration, esther filters 0,45 µm	15	120	VE_1: 0,29 VE_2: 1,35	L/S2: 6,14 and 5,81; 2,3 and 1,84; 23,7 and 23,4 L/S8: 6,06 and 6,08; 1,73 and 0,91; 23,8 and 23,7
5	0,1758	L_2: PE bottle 0,5 l L_8: PE bottle 2 l Gerhardt RO20		L_2: 0,346 L_8: 1,4	Whatman no 6	15		VE_1: 0,28	6,2 and 6,0; 2,1 and 1,95
6	0,175	Glass bottle, 2 l, Rotary shaker, 11 rpm	23	L_2: 0,349 L_8: 1,399	Filtration, cellulose nitrate, 0,45 µm	< 30		VE_1: 0,296 and 0,262	L/S2: 5,7 and 5,8; 1,75 and 1,79; L/S8: 6,0 and 6,0; 0,84 and 0,79
7	0,1759	L_2: HDPE bottle 0,5 l L_8: HDPE bottle 2 l HAM, 10 rpm	20,2-21,1	L_2: 0,35 L_8: 1,4	Filtration, Whatman Me 25 and membrane, 0,45 µm	15	50	VE_1: 0,29 and 0,295 VE_2: 0,355 and 0,285	L/S2: 5,64; 2,75 L/S8: 5,55; 2,08
9	0,1765	HDPE bottle, 1 l, End-over-end, 5-10 rpm	Room	L_2: 0,348 L_8: 1,4	Centifugation, membrane 0,45 µm	20	10	VE_1: 0,294	5,9; 1,67
10	0,175	L_2: PE bottle 1 l L_8: PE bottle 2 l Rotary shaker, 10 rpm	21	L_2: 0,35 L_8: 1,4	Filtration, membrane 0,45 µm	34	L/S 2: 55 L/S 8: 180	VE_1: 0,29 VE_2: 1,33	L/S2: 6,18 and 6,06; 1,78 and 1,53; 22,3 L/S8: 6,42 and 6,46; 0,83 and 0,77; 22,2
11	0,176	HDPE bottle, 0,5 l and 1 l, End-over-end, 10 rpm	22	L_2: 0,349 L_8: 1,4	Filtration, glass fiber filter and membrane, 0,45 µm	130		VE_1: 0,305	L/S2: 6,36 and 6,32; 1,69 and 1,7; 22, 2 L/S8: 6,43 and 6,46; 1,29 and 01,05; 22,6
12	0,175	HDPE bottle, 2 l, End-over-end, 7 rpm	23	L_2: 0,35 L_8: 1,337	Filtration, cellulose nitrate, 0,45 µm	15	40	VE_1: 0,287 and 0,289 VE_2: 0,5	6,45 and 6,52; 1,09 and 1,19; 23
13	0,1005	HDPE bottle, 1 l, Rotary shaker, 10 µm	23	L_2: 0,1995 L_8: 0,8	Filtration, Whatman 27 and membrane 0,8 µm	10 and 45	40 and 80	VE_1: 0,164 and 0,166 VE_2: 0,15	L/S2: 5,35 and 5,46; 3,90 and 4,04; 23 L/S8: 5,45 and 5,49; 0,87; 23
17	0,176	Glass bottle, 1 l, End-over-end, 8 rpm	22	L_2: 0,349 L_8: 1,40	Filtration, glass filter and membrane 0,45 µm	2	L/S 2: 57 L/S 8: 203	VE_1: 0,302 VE_2: 1,36	L/S2: 7,1; 3,53 L/S8: 7,3; 2,33
18	0,175	HDPE-bottle, 0,5 l Rotary shaker, 10 rpm	22	L_2: 0,349 L_8: 1,4	Filtration, glass fiber filter and membrane, 0,45 µm	15-20	30-40	1: 0,165 and 0,18 2: 0,95 and 0,995	L/S2: 6,6 and 6,6; 2,4 and 2,26 L/S8: 6,4 and 6,0; 1,28 and 1,11

3) Measurement methods

Lab	Metals	DOC
1	ICP-MS	IR spectrometry
2	ICP-OES Se, Pb: GFAAS	IR spectrometry
3	ICP-AES and ICP-MS	Non-purgable organic C (NPOC),4
4	ICP-MS	
5	ICP-OES	
6	ICP-AES, ICP-MS	IR spectrometry
7	ICP-MS Ba, Fe: ICP-OES	
9	EPA 200.7 (2008)	IR spectrometry
10	GAAS Zn: FAAS	
11	ICP-MS	IR spectrometry
12	ICP-MS and ICP-OES	IR spectrometry
13	ICP-OES As, SB, Se: hydridi ICP-OES	
14	ICP-OES	
15	ICP-OES As, Pb: GAAS, Mo: FAAS	IR spectrometry
17	ICP-OES	IR spectrometry
18	ICP-OES	TOC analyzer

APPENDIX 5. EVALUATION OF ASSIGNED VALUES AND THEIR UNCERTAINTIES

R1: Results of the one stage batch test
R21: Results of the two stage batch test (L/S2)
R22: Results of the two stage batch test (L/S10)

Analyte	Sample	Assigned value[2)]	Uncertainty of the assigned value, U [1), 3)]
As mg/kg	R1	28,0	7,7 %
	R21	8,1	8,8 %
	R22	29,5	7,8 %
Ba mg/kg	R1	0,055	-
	R21	0,039	-
	R22	0,057	-
Conductivity mS/m	R1	0,985	12,2 %
	R21	2,12	16,1 %
	R22	1,19	23,7 %
Cr mg/kg	R1	1,52	18,0 %
	R21	0,641	18,4 %
	R22	1,52	15,1 %
Cu mg/kg	R1	3,13	18,6 %
	R21	1,05	33,7 %
	R22	2,72	32,6 %
DOC mg/kg	R1	45,2	12,8 %
	R21	27,8	14,5 %
	R22	51,1	19,4 %
Fe Mg/kg	R1	0,861	-
	R21	0,234	-
	R22	0,939	-
Ni mg/kg	R1	0,041	-
	R21	0,007	-
	R22	0,024	-
Pb mg/kg	R1	0,042	-
	R21	0,023	-
	R22	0,049	-
pH	R1	5,77	3,8 %
	R21	6,03	4,6 %
	R22	6,09	4,9 %
Sb mg/kg	R1	0,940	14,0 %
	R21	0,357	11,0 %
	R22	0,990	15,7 %
Zn mg/kg	R1	0,192	-
	R21	0,073	-
	R22	0,237	-

1) **$U\ \% = 2 * 1{,}25 * s_{rob}/\sqrt{p}$, where, s_{rob} = the robust standard deviation, p = the number of the results (95 % confidence level)**

2) **Cd, Mo and Se**: The assigned value and its uncertainty has not been estimated because of low concentrations of analytes for estimation of laboratory performance in the leaching tests.

3) **Ba , Fe, Ni, Pb and Zn:** The uncertainty of the assigned value has not been estimated because of low concentration of analyte (Ni and Pb) or the slight importance of analyte (Fe) dealing with its toxicity.

APPENDIX 6. EXPLANATIONS FOR THE RESULT SHEETS

Results of each participants and the summary of the results:

Sample	The code of the sample
z-Graphics	z score - the graphical presentation
z-value	z = (x - X)/s, where x = the result of the invidual participant X = the reference value (the assigned value) s = the target value for the total deviation (s_{target}).
Outl test OK	yes - the result passed the outlier test H = Hampel test (a test of mean values) In addition, in robust statistics results deviating at least > 50 % from the original robust mean have been rejected.
Assigned value	the reference value
2* Targ SD %	the target total standard deviation (95 % confidence interval).
Lab's result	the result reported by the participant (the mean value of the replicates)
Md.	Median
Mean	Mean
SD	Standard deviation
SD%	Standard deviation, %
Mean rob	Robust mean
SDrob	Robust standard deviation
SDrob %	Robust standard deviation-%
Passed	The results passed the outlier test
Missing	i.e. < DL
Num of labs	the total number of the participants

Summary on the z scores:

A - accepted ($-2 \le z \le 2$)
p - questionable ($2 < z \le 3$), positive error, the result > X
n - questionable ($-3 \le z < -2$), negative error, the result < X
P - non- accepted ($z > 3$), positive error, the result >>> X
N - non- accepted ($z < -3$), negative error, the result <<< X (X = the reference value)

Robust analysis/Calculation of the assigned values:

The items of data is sorted into increasing order, $x_1, x_2, \ldots, x_i, \ldots, x_p$.
Initial values for x^* and s^* are calculated as:
X^* = median of x_i $(i = 1 \ldots p)$
S^* = 1.483* median of $| x_i - x^* |$ $(i = 1 \ldots p)$

For each x_i is calculated:
$x_i^* = x^* - \varphi$ if $x_i < x^* - \varphi$
$x_i^* = x^* + \varphi$ if $x_i > x^* + \varphi$
$x_i^* = x_i$ otherwise

The new values of x^* and s^* are calculated from:
$X^* = \sum x_i^* / p$

$$s^* = 1.134\sqrt{\sum (x_i^* - x^*)^2 / (p-1)}$$

The robust estimates x^* and s^* can be derived by an iterative calculation, i.e. by updating the values of x^* and s^* several times, until the process converges.

LIITE 7. RESULTS OF EACH PARTICIPANT

Appendix 7.

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
Laboratory 1														
As	R1	mg/kg		0,0714	28	25	28,25	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		-0,844	8,1	25	7,245	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		-1,03	29,5	25	25,7	29,4	2,12	7,2	17	13	0	15
Ba	R1	mg/kg			0,055		0,036	0,06	0,02	44,8	10	6	4	12
	R21	mg/kg			0,039		0,027	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		0,041	0,07	0,03	50,2	8	6	5	12
Cd	R1	mg/kg					<0,00	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0,00	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,00	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		-1,39	0,985	30	0,78	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		1,64	2,12	30	2,64	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		-1,57	1,19	30	0,91	1,23	0,43	35,4	20	6	1	14
Cr	R1	mg/kg		0,74	1,52	40	1,745	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		1,36	0,641	40	0,815	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		-0,526	1,52	40	1,36	1,53	0,26	17,6	19	9	0	14
Cu	R1	mg/kg		1,32	3,13	40	3,955	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		4,71	1,05	40	2,04	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		2,13	2,72	40	3,88	2,88	1,37	47,6	25	3	0	14
DOC	R1	mg/kg		-1,72	45,2	25	35,5	46,7	7,84	16,7	18	3	0	11
	R21	mg/kg		-0,0863	27,8	25	27,5	28,7	5,73	19,9	17	2	0	10
	R22	mg/kg		-2,44	51,1	25	35,5	54,8	15,1	27,6	14	4	0	10
Fe	R1	mg/kg			0,861		1,195	0,93	0,51	55,1	20	2	2	13
	R21	mg/kg			0,234		0,5	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		0,815	1,19	0,79	66,5	17	3	2	12
Mo	R1	mg/kg					<0,00	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,00	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,00	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		0,202	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		0,009	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		0,093	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		0,073	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		0,045	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		0,046	0,05	0,04	71,0	21	0	4	15
pH	R1			-0,427	5,77	6,9	5,685	5,8	0,32	5,6	25	4	0	15
	R21			-1,88	6,03	6,7	5,65	6,03	0,43	7,2	26	3	0	15
	R22			-1,95	6,09	6,5	5,705	6,10	0,45	7,4	25	2	0	14
Sb	R1	mg/kg		-0,954	0,94	30	0,805	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		-1,83	0,357	30	0,259	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		-2,12	0,99	30	0,675	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					<0,00	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					<0,00	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0,00	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		0,179	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		0,074	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		0,134	0,19	0,08	46,0	11	9	5	15

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
Laboratory 2														
As	R1	mg/kg		0,786	28	25	30,75	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		1,66	8,1	25	9,78	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		1,13	29,5	25	33,65	29,4	2,12	7,2	17	13	0	15
Ba	R1	mg/kg			0,055		0,045	0,06	0,02	44,8	10	6	4	12
	R21	mg/kg			0,039		0,024	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		0,054	0,07	0,03	50,2	8	6	5	12
Cd	R1	mg/kg					<0,01	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0,00	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,01	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		-0,609	0,985	30	0,895	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		-1,35	2,12	30	1,69	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		-2,38	1,19	30	0,765	1,23	0,43	35,4	20	6	1	14
Cr	R1	mg/kg		0,197	1,52	40	1,58	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		3,77	0,641	40	1,125	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		2,91	1,52	40	2,405	1,53	0,26	17,6	19	9	0	14
Cu	R1	mg/kg		0,783	3,13	40	3,62	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		2,9	1,05	40	1,66	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		2,94	2,72	40	4,32	2,88	1,37	47,6	25	3	0	14
DOC	R1	mg/kg		-0,124	45,2	25	44,5	46,7	7,84	16,7	18	3	0	11
	R21	mg/kg		-0,201	27,8	25	27,1	28,7	5,73	19,9	17	2	0	10
	R22	mg/kg		1,89	51,1	25	63,15	54,8	15,1	27,6	14	4	0	10
Fe	R1	mg/kg			0,861		0,65	0,93	0,51	55,1	20	2	2	13
	R21	mg/kg			0,234		1,135	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		2,095	1,19	0,79	66,5	17	3	2	12
Mo	R1	mg/kg					<0,05	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,01	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,05	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		<0,1	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		<0,02	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		<0,1	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		0,027	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		0,06	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		0,1	0,05	0,04	71,0	21	0	4	15
pH	R1			1,41	5,77	6,9	6,05	5,8	0,32	5,6	25	4	0	15
	R21			-1,88	6,03	6,7	5,65	6,03	0,43	7,2	26	3	0	15
	R22			-0,707	6,09	6,5	5,95	6,10	0,45	7,4	25	2	0	14
Sb	R1	mg/kg		0,886	0,94	30	1,065	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		0,803	0,357	30	0,4	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		1,01	0,99	30	1,14	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					<0,05	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					<0,01	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0,05	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		0,150	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		0,048	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		0,16	0,19	0,08	46,0	11	9	5	15

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
Laboratory 3														
As	R1	mg/kg		-0,571	28	25	26	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		-0,691	8,1	25	7,4	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		-0,271	29,5	25	28,5	29,4	2,12	7,2	17	13	0	15
Ba	R1	mg/kg			0,055		<2	0,06	0,02	44,8	10	6	4	12
	R21	mg/kg			0,039		<0,7	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		<2	0,07	0,03	50,2	8	6	5	12
Cd	R1	mg/kg					<0,00	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0,00	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,00	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		0,102	0,985	30	1	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		-1,01	2,12	30	1,8	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		-0,784	1,19	30	1,05	1,23	0,43	35,4	20	6	1	14
Cr	R1	mg/kg		-0,724	1,52	40	1,3	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		-0,632	0,641	40	0,56	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		-0,23	1,52	40	1,45	1,53	0,26	17,6	19	9	0	14
Cu	R1	mg/kg		0,351	3,13	40	3,35	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		0,714	1,05	40	1,2	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		1,53	2,72	40	3,55	2,88	1,37	47,6	25	3	0	14
DOC	R1	mg/kg		0,761	45,2	25	49,5	46,7	7,84	16,7	18	3	0	11
	R21	mg/kg		-0,806	27,8	25	25	28,7	5,73	19,9	17	2	0	10
	R22	mg/kg		0,141	51,1	25	<50	54,8	15,1	27,6	14	4	0	10
Fe	R1	mg/kg			0,861		0,59	0,93	0,51	55,1	20	2	2	13
	R21	mg/kg			0,234		<0,2	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		0,605	1,19	0,79	66,5	17	3	2	12
Mo	R1	mg/kg					<0,05	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,03	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,05	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		<0,04	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		<0,02	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		<0,04	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		<0,05	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		<0,02	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		<0,05	0,05	0,04	71,0	21	0	4	15
pH	R1			-0,101	5,77	6,9	5,75	5,8	0,32	5,6	25	4	0	15
	R21			1,09	6,03	6,7	6,25	6,03	0,43	7,2	26	3	0	15
	R22			1,31	6,09	6,5	6,35	6,10	0,45	7,4	25	2	0	14
Sb	R1	mg/kg		0,248	0,94	30	0,975	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		0,056	0,357	30	0,36	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		0,0673	0,99	30	1	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					<0,01	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					<0,00	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0,01	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		<0,4	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		<0,2	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		<0,4	0,19	0,08	46,0	11	9	5	15

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
Laboratory 4														
As	R1	mg/kg		0,711	28	25	30,49	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		0,353	8,1	25	8,457	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		-0,0239	29,5	25	29,41	29,4	2,12	7,2	17	13	0	15
Ba	R1	mg/kg			0,055		<0,05	0,06	0,02	44,8	10	6	4	12
	R21	mg/kg			0,039		0,018	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		<0,06	0,07	0,03	50,2	8	6	5	12
Cd	R1	mg/kg					<0,00	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0,00	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,00	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		-0,102	0,985	30	0,97	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		-0,157	2,12	30	2,07	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		0,728	1,19	30	1,32	1,23	0,43	35,4	20	6	1	14
Cr	R1	mg/kg		-0,484	1,52	40	1,373	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		-0,0897	0,641	40	0,629	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		-0,377	1,52	40	1,405	1,53	0,26	17,6	19	9	0	14
Cu	R1	mg/kg		0,391	3,13	40	3,374	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		1,54	1,05	40	1,375	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		1,16	2,72	40	3,349	2,88	1,37	47,6	25	3	0	14
Mo	R1	mg/kg					<0,05	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,01	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,05	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		<0,04	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		<0,00	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		<0,04	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		0,078	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		0,024	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		<0,07	0,05	0,04	71,0	21	0	4	15
pH	R1			0,854	5,77	6,9	5,94	5,8	0,32	5,6	25	4	0	15
	R21			-0,272	6,03	6,7	5,975	6,03	0,43	7,2	26	3	0	15
	R22			-0,101	6,09	6,5	6,07	6,10	0,45	7,4	25	2	0	14
Sb	R1	mg/kg		1,12	0,94	30	1,098	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		0,299	0,357	30	0,373	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		0,515	0,99	30	1,067	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					<0,05	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					<0,01	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0,05	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		<0,4	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		0,10	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		<0,4	0,19	0,08	46,0	11	9	5	15
Laboratory 5														
As	R21	mg/kg		0,183	8,1	25	8,285	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		1,02	29,5	25	33,25	29,4	2,12	7,2	17	13	0	15
Ba	R21	mg/kg			0,039		<0,01	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		<0,01	0,07	0,03	50,2	8	6	5	12
Cd	R21	mg/kg					<0,01	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,01	0,29	0,01	5,5	2	0	13	14
conductivity	R21	mS/m		0,362	2,12	30	2,235	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		4,68	1,19	30	2,025	1,23	0,43	35,4	20	6	1	14
Cr	R21	mg/kg		-1,75	0,641	40	0,416	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		0,707	1,52	40	1,735	1,53	0,26	17,6	19	9	0	14
Cu	R21	mg/kg		-3,49	1,05	40	0,317	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		-2,91	2,72	40	1,135	2,88	1,37	47,6	25	3	0	14
Fe	R21	mg/kg			0,234		0,156	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		2,17	1,19	0,79	66,5	17	3	2	12
Mo	R21	mg/kg					<0,01	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,01	0,00	0,00	26,7	2	0	12	13
Ni	R21	mg/kg			0,007		<0,01	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		<0,01	0,01	0,00	51,0	6	4	9	14
Pb	R21	mg/kg			0,023		<0,01	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		0,094	0,05	0,04	71,0	21	0	4	15
pH	R21			-0,643	6,03	6,7	5,9	6,03	0,43	7,2	26	3	0	15
	R22			0,0505	6,09	6,5	6,1	6,10	0,45	7,4	25	2	0	14
Sb	R21	mg/kg		0,0934	0,357	30	0,362	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		1,65	0,99	30	1,235	0,99	0,23	23,7	28	2	0	15
Se	R21	mg/kg					<0,01	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0,01	0,00	0,00	14,6	2	0	11	12
Zn	R21	mg/kg			0,073		<0,01	0,07	0,03	41,1	17	5	3	15

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
	R22	mg/kg			0,237		0,023	0,19	0,08	46,0	11	9	5	15
Laboratory 6														
As	R1	mg/kg		0,543	28	25	29,9	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		-0,0691	8,1	25	8,03	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		-0,176	29,5	25	28,85	29,4	2,12	7,2	17	13	0	15
Ba	R1	mg/kg			0,055		<0,1	0,06	0,02	44,8	10	6	4	12
	R21	mg/kg			0,039		<0,1	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		<0,1	0,07	0,03	50,2	8	6	5	12
Cd	R1	mg/kg					0,32	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					0,08	0,08	0	0	2	0	13	14
	R22	mg/kg					0,29	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		-0,474	0,985	30	0,915	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		-1,1	2,12	30	1,77	2,20	0,58	26,3	25	4	0	15
Cr	R1	mg/kg		-1,12	1,52	40	1,18	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		-0,944	0,641	40	0,52	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		-1,23	1,52	40	1,145	1,53	0,26	17,6	19	9	0	14
Cu	R1	mg/kg		0,559	3,13	40	3,48	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		0,976	1,05	40	1,255	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		1,23	2,72	40	3,39	2,88	1,37	47,6	25	3	0	14
DOC	R1	mg/kg		0,0531	45,2	25	45,5	46,7	7,84	16,7	18	3	0	11
	R21	mg/kg		-0,23	27,8	25	27	28,7	5,73	19,9	17	2	0	10
	R22	mg/kg		0,423	51,1	25	53,8	54,8	15,1	27,6	14	4	0	10
Fe	R1	mg/kg			0,861		<1	0,93	0,51	55,1	20	2	2	13
	R21	mg/kg			0,234		<1	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		<1	1,19	0,79	66,5	17	3	2	12
Mo	R1	mg/kg					<0,1	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,1	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,1	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		<0,1	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		<0,1	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		<0,1	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		<0,1	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		<0,1	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		<0,1	0,05	0,04	71,0	21	0	4	15
pH	R1			0,653	5,77	6,9	5,9	5,8	0,32	5,6	25	4	0	15
	R21			-1,39	6,03	6,7	5,75	6,03	0,43	7,2	26	3	0	15
Sb	R1	mg/kg		-1,6	0,94	30	0,715	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		-1,81	0,357	30	0,26	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		-1,28	0,99	30	0,8	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					<0,01	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					<0,01	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0,01	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		<0,1	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		<0,1	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		<0,1	0,19	0,08	46,0	11	9	5	15

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
Laboratory 7														
As	R1	mg/kg		-3,26	28	25	16,6	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		-2,62	8,1	25	5,45	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		-3,39	29,5	25	17	29,4	2,12	7,2	17	13	0	15
Ba	R1	mg/kg			0,055		0,1	0,06	0,02	44,8	10	6	4	12
	R21	mg/kg			0,039		<0,02	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		<0,1	0,07	0,03	50,2	8	6	5	12
Cd	R1	mg/kg					<0,00	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0,00	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,00	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		7,45	0,985	30	2,085	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		2,09	2,12	30	2,785	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		4,71	1,19	30	2,03	1,23	0,43	35,4	20	6	1	14
Cr	R1	mg/kg		-2,14	1,52	40	0,87	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		-1,22	0,641	40	0,485	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		-1,55	1,52	40	1,05	1,53	0,26	17,6	19	9	0	14
Cu	R1	mg/kg		3,15	3,13	40	5,1	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		0,476	1,05	40	1,15	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		5,39	2,72	40	5,65	2,88	1,37	47,6	25	3	0	14
DOC	R1	mg/kg		1,29	45,2	25	52,5	46,7	7,84	16,7	18	3	0	11
	R21	mg/kg		1,81	27,8	25	34,1	28,7	5,73	19,9	17	2	0	10
	R22	mg/kg		0,43	51,1	25	53,85	54,8	15,1	27,6	14	4	0	10
Fe	R1	mg/kg			0,861		<0,5	0,93	0,51	55,1	20	2	2	13
	R21	mg/kg			0,234		<0,1	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		<0,5	1,19	0,79	66,5	17	3	2	12
Mo	R1	mg/kg					<0,03	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,00	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,03	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		0,04	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		0,009	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		0,045	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		0,02	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		0,015	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		0,03	0,05	0,04	71,0	21	0	4	15
pH	R1			-1,43	5,77	6,9	5,485	5,8	0,32	5,6	25	4	0	15
	R21			-2	6,03	6,7	5,625	6,03	0,43	7,2	26	3	0	15
	R22			-2,85	6,09	6,5	5,525	6,10	0,45	7,4	25	2	0	14
Sb	R1	mg/kg		-2,02	0,94	30	0,655	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		-2,28	0,357	30	0,235	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		-2,32	0,99	30	0,645	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					<0,02	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					<0,00	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0,02	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		0,385	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		0,08	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		0,41	0,19	0,08	46,0	11	9	5	15
Laboratory 8														
As	R1	mg/kg		-1	28	25	24,5	27,7	3,01	10,8	21	9	0	15
Ba	R1	mg/kg			0,055		0,07	0,06	0,02	44,8	10	6	4	12
Cd	R1	mg/kg					<0,00	0,74	0,56	75,0	4	0	12	14
conductivity	R1	mS/m		-0,609	0,985	30	0,895	1,05	0,21	20,3	19	9	1	15
Cr	R1	mg/kg		-1,55	1,52	40	1,05	1,51	0,37	24,9	22	6	0	14
Cu	R1	mg/kg		0,0319	3,13	40	3,15	3,21	0,58	18,2	17	9	0	13
DOC	R1	mg/kg		-0,566	45,2	25	42	46,7	7,84	16,7	18	3	0	11
Fe	R1	mg/kg			0,861		0,33	0,93	0,51	55,1	20	2	2	13
Mo	R1	mg/kg					<0,02	0,00	0,00	14,5	2	0	14	15
Ni	R1	mg/kg			0,041		0,06	0,05	0,05	91,5	9	2	9	15
Pb	R1	mg/kg			0,042		0,02	0,04	0,03	69,8	19	2	3	14
pH	R1			-0,854	5,77	6,9	5,6	5,8	0,32	5,6	25	4	0	15
Sb	R1	mg/kg		-1,38	0,94	30	0,745	0,94	0,18	19,1	24	2	0	13
Se	R1	mg/kg					<0,01	0,00	0,00	47,5	2	0	11	12
Zn	R1	mg/kg			0,192		0,13	0,22	0,09	42,9	14	6	5	15

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
Laboratory 9														
As	R1	mg/kg		-0,286	28	25	27	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		-0,593	8,1	25	7,5	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		-0,149	29,5	25	28,95	29,4	2,12	7,2	17	13	0	15
Ba	R1	mg/kg			0,055		0,042	0,06	0,02	44,8	10	6	4	12
	R21	mg/kg			0,039		0,014	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		0,042	0,07	0,03	50,2	8	6	5	12
Cd	R1	mg/kg					<0,00	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0,00	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,00	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m			0,985	30	<1	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		-1,42	2,12	30	1,67	2,20	0,58	26,3	25	4	0	15
	R22	mS/m			1,19	30	<1	1,23	0,43	35,4	20	6	1	14
Cr	R1	mg/kg		0,641	1,52	40	1,715	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		0,234	0,641	40	0,671	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		0,0822	1,52	40	1,545	1,53	0,26	17,6	19	9	0	14
DOC	R1	mg/kg		-1,27	45,2	25	38	46,7	7,84	16,7	18	3	0	11
	R21	mg/kg		0,0575	27,8	25	28	28,7	5,73	19,9	17	2	0	10
	R22	mg/kg		-1,33	51,1	25	42,6	54,8	15,1	27,6	14	4	0	10
Fe	R1	mg/kg			0,861		0,96	0,93	0,51	55,1	20	2	2	13
	R21	mg/kg			0,234		0,153	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		0,467	1,19	0,79	66,5	17	3	2	12
Mo	R1	mg/kg					<0,00	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,00	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,00	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		0,017	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		0,003	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		0,013	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		0,047	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		0,008	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		0,024	0,05	0,04	71,0	21	0	4	15
pH	R1			0,653	5,77	6,9	5,9	5,8	0,32	5,6	25	4	0	15
	R21			-0,643	6,03	6,7	5,9	6,03	0,43	7,2	26	3	0	15
	R22			-2,48	6,09	6,5	5,6	6,10	0,45	7,4	25	2	0	14
Sb	R1	mg/kg		1,31	0,94	30	1,125	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		0,896	0,357	30	0,405	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		1,18	0,99	30	1,165	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					0,008	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					0,002	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					0,006	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		0,144	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		0,044	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		0,135	0,19	0,08	46,0	11	9	5	15
Laboratory 10														
As	R21	mg/kg		1,64	8,1	25	9,755	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		0,542	29,5	25	31,5	29,4	2,12	7,2	17	13	0	15
Cd	R21	mg/kg					<0,00	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,01	0,29	0,01	5,5	2	0	13	14
conductivity	R21	mS/m		-1,46	2,12	30	1,655	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		-2,19	1,19	30	0,8	1,23	0,43	35,4	20	6	1	14
Cr	R21	mg/kg		2,5	0,641	40	0,961	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		2,42	1,52	40	2,255	1,53	0,26	17,6	19	9	0	14
Cu	R21	mg/kg		0,119	1,05	40	1,075	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		0,515	2,72	40	3	2,88	1,37	47,6	25	3	0	14
Ni	R21	mg/kg			0,007		0,009	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		<0,03	0,01	0,00	51,0	6	4	9	14
Pb	R21	mg/kg			0,023		0,038	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		0,090	0,05	0,04	71,0	21	0	4	15
pH	R21			0,446	6,03	6,7	6,12	6,03	0,43	7,2	26	3	0	15
	R22			1,77	6,09	6,5	6,44	6,10	0,45	7,4	25	2	0	14
Sb	R21	mg/kg		1,18	0,357	30	0,42	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		0,808	0,99	30	1,11	0,99	0,23	23,7	28	2	0	15
Zn	R21	mg/kg			0,073		0,089	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		<0,41	0,19	0,08	46,0	11	9	5	15

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
Laboratory 11														
As	R1	mg/kg		0,371	28	25	29,3	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		-0,203	8,1	25	7,895	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		0,231	29,5	25	30,35	29,4	2,12	7,2	17	13	0	15
Ba	R1	mg/kg			0,055		0,046	0,06	0,02	44,8	10	6	4	12
	R21	mg/kg			0,039		0,075	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		0,186	0,07	0,03	50,2	8	6	5	12
Cd	R1	mg/kg					<0,02	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0,02	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,02	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		-0,643	0,985	30	0,89	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		-0,755	2,12	30	1,88	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		-0,28	1,19	30	1,14	1,23	0,43	35,4	20	6	1	14
Cr	R1	mg/kg		0,0658	1,52	40	1,54	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		-0,394	0,641	40	0,590	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		0,592	1,52	40	1,7	1,53	0,26	17,6	19	9	0	14
Cu	R1	mg/kg		0	3,13	40	3,13	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		-2,6	1,05	40	0,504	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		-2,5	2,72	40	1,36	2,88	1,37	47,6	25	3	0	14
DOC	R1	mg/kg		0,434	45,2	25	47,65	46,7	7,84	16,7	18	3	0	11
	R21	mg/kg		-1,24	27,8	25	23,5	28,7	5,73	19,9	17	2	0	10
	R22	mg/kg		-0,556	51,1	25	47,55	54,8	15,1	27,6	14	4	0	10
Fe	R1	mg/kg			0,861		0,987	0,93	0,51	55,1	20	2	2	13
	R21	mg/kg			0,234		0,191	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		1,25	1,19	0,79	66,5	17	3	2	12
Mo	R1	mg/kg					<0,02	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,02	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,02	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		<0,04	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		<0,04	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		<0,04	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		0,049	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		<0,02	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		0,060	0,05	0,04	71,0	21	0	4	15
pH	R1			-0,553	5,77	6,9	5,66	5,8	0,32	5,6	25	4	0	15
	R21			1,51	6,03	6,7	6,335	6,03	0,43	7,2	26	3	0	15
	R22			2	6,09	6,5	6,485	6,10	0,45	7,4	25	2	0	14
Sb	R1	mg/kg		0,0248	0,94	30	0,943	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		0	0,357	30	0,357	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		0,0438	0,99	30	0,996	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					<0,02	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					<0,02	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0,02	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		<0,20	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		<0,20	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		<0,20	0,19	0,08	46,0	11	9	5	15

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
Laboratory 12														
As	R1	mg/kg		0,0714	28	25	28,25	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		-0,0988	8,1	25	8	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		0,0814	29,5	25	29,8	29,4	2,12	7,2	17	13	0	15
Ba	R1	mg/kg			0,055		0,835	0,06	0,02	44,8	10	6	4	12
	R21	mg/kg			0,039		0,12	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		0,545	0,07	0,03	50,2	8	6	5	12
Cd	R1	mg/kg					<0.01	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0.01	0,08	0	0	2	0	13	14
	R22	mg/kg					<0.01	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		0,745	0,985	30	1,095	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		-1,34	2,12	30	1,695	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		-0,112	1,19	30	1,17	1,23	0,43	35,4	20	6	1	14
Cr	R1	mg/kg		-0,526	1,52	40	1,36	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		-0,593	0,641	40	0,565	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		-0,526	1,52	40	1,36	1,53	0,26	17,6	19	9	0	14
Cu	R1	mg/kg		-2,47	3,13	40	1,585	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		-0,905	1,05	40	0,86	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		-1,96	2,72	40	1,655	2,88	1,37	47,6	25	3	0	14
DOC	R1	mg/kg		-0,735	45,2	25	41,05	46,7	7,84	16,7	18	3	0	11
	R21	mg/kg		-1,45	27,8	25	22,75	28,7	5,73	19,9	17	2	0	10
	R22	mg/kg		-1,31	51,1	25	42,7	54,8	15,1	27,6	14	4	0	10
Fe	R1	mg/kg			0,861		0,3	0,93	0,51	55,1	20	2	2	13
	R21	mg/kg			0,234		0,06	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		0,315	1,19	0,79	66,5	17	3	2	12
Mo	R1	mg/kg					0,005	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					0,001	0,00	0	0	2	0	12	13
	R22	mg/kg					0,006	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		0,013	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		0,004	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		0,012	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		0,016	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		0,003	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		0,015	0,05	0,04	71,0	21	0	4	15
pH	R1			2,13	5,77	6,9	6,195	5,8	0,32	5,6	25	4	0	15
	R21			1,53	6,03	6,7	6,34	6,03	0,43	7,2	26	3	0	15
	R22			1,79	6,09	6,5	6,445	6,10	0,45	7,4	25	2	0	14
Sb	R1	mg/kg		1,42	0,94	30	1,14	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		1,36	0,357	30	0,43	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		1,38	0,99	30	1,195	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					<0.00	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					<0.00	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0.00	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		0,725	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		0,16	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		0,565	0,19	0,08	46,0	11	9	5	15

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
Laboratory 13														
As	R1	mg/kg		0,286	28	25	29	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		0	8,1	25	8,1	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		-0,0746	29,5	25	29,23	29,4	2,12	7,2	17	13	0	15
Cd	R1	mg/kg					<0,02	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0,00	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,02	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		2,91	0,985	30	1,415	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		5,82	2,12	30	3,97	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		-1,79	1,19	30	0,87	1,23	0,43	35,4	20	6	1	14
Cr	R1	mg/kg		-0,214	1,52	40	1,455	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		-0,554	0,641	40	0,57	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		0,0329	1,52	40	1,53	1,53	0,26	17,6	19	9	0	14
Cu	R1	mg/kg		0,0319	3,13	40	3,15	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		1,26	1,05	40	1,315	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		0,68	2,72	40	3,09	2,88	1,37	47,6	25	3	0	14
Fe	R1	mg/kg			0,861		0,85	0,93	0,51	55,1	20	2	2	13
	R21	mg/kg			0,234		0,18	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		1,364	1,19	0,79	66,5	17	3	2	12
Mo	R1	mg/kg					<0,10	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,02	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,10	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		<0,10	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		<0,02	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		<0,10	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		<0,40	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		<0,08	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		<0,40	0,05	0,04	71,0	21	0	4	15
pH	R1			-1,73	5,77	6,9	5,425	5,8	0,32	5,6	25	4	0	15
	R21			-3,09	6,03	6,7	5,405	6,03	0,43	7,2	26	3	0	15
	R22			-3,13	6,09	6,5	5,47	6,10	0,45	7,4	25	2	0	14
Sb	R1	mg/kg		0,709	0,94	30	1,04	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		0,187	0,357	30	0,367	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		0,0404	0,99	30	0,996	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					<0,02	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					<0,00	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0,02	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		1,885	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		0,912	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		1,623	0,19	0,08	46,0	11	9	5	15
Laboratory 14														
As	R1	mg/kg		0,193	28	25	28,68	27,7	3,01	10,8	21	9	0	15
conductivity	R1	mS/m		-0,338	0,985	30	0,935	1,05	0,21	20,3	19	9	1	15
Cr	R1	mg/kg		1,74	1,52	40	2,05	1,51	0,37	24,9	22	6	0	14
Fe	R1	mg/kg			0,861		1,535	0,93	0,51	55,1	20	2	2	13
Mo	R1	mg/kg					<0,01	0,00	0,00	14,5	2	0	14	15
Ni	R1	mg/kg			0,041		<0,01	0,05	0,05	91,5	9	2	9	15
pH	R1			0,126	5,77	6,9	5,795	5,8	0,32	5,6	25	4	0	15
Zn	R1	mg/kg			0,192		0,235	0,22	0,09	42,9	14	6	5	15
Laboratory 15														
As	R1	mg/kg		-7,11	28	25	3,11	27,7	3,01	10,8	21	9	0	15
Ba	R1	mg/kg			0,055		<0,1	0,06	0,02	44,8	10	6	4	12
Cd	R1	mg/kg					1,175	0,74	0,56	75,0	4	0	12	14
conductivity	R1	mS/m		3,79	0,985	30	1,545	1,05	0,21	20,3	19	9	1	15
Cr	R1	mg/kg		1,28	1,52	40	1,91	1,51	0,37	24,9	22	6	0	14
Cu	R1	mg/kg		-0,0719	3,13	40	3,085	3,21	0,58	18,2	17	9	0	13
DOC	R1	mg/kg		2,22	45,2	25	57,75	46,7	7,84	16,7	18	3	0	11
Fe	R1	mg/kg			0,861		1,215	0,93	0,51	55,1	20	2	2	13
Mo	R1	mg/kg					<1	0,00	0,00	14,5	2	0	14	15
Ni	R1	mg/kg			0,041		<0,1	0,05	0,05	91,5	9	2	9	15
Pb	R1	mg/kg			0,042		0,077	0,04	0,03	69,8	19	2	3	14
pH	R1			-4,02	5,77	6,9	4,97	5,8	0,32	5,6	25	4	0	15
Zn	R1	mg/kg			0,192		<0,1	0,22	0,09	42,9	14	6	5	15

Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assigned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Passed	R-adjusted	Missing	Num of labs
Laboratory 16														
As	R1	mg/kg		-1,29	28	25	23,5	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		-1,28	8,1	25	6,8	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		-2,79	29,5	25	19,2	29,4	2,12	7,2	17	13	0	15
Cd	R1	mg/kg					<0,02	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0,00	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,01	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		12,8	0,985	30	2,88	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		1,4	2,12	30	2,565	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		1,54	1,19	30	1,465	1,23	0,43	35,4	20	6	1	14
Cu	R1	mg/kg		-1,33	3,13	40	2,3	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		-3,05	1,05	40	0,41	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		-2,65	2,72	40	1,28	2,88	1,37	47,6	25	3	0	14
Mo	R1	mg/kg					<0,01	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,00	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,00	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		0,075	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		0,008	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		0,008	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		<0,01	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		0,008	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		<0,00	0,05	0,04	71,0	21	0	4	15
pH	R1			1,46	5,77	6,9	6,06	5,8	0,32	5,6	25	4	0	15
	R21			1,06	6,03	6,7	6,245	6,03	0,43	7,2	26	3	0	15
	R22			0,0253	6,09	6,5	6,095	6,10	0,45	7,4	25	2	0	14
Sb	R1	mg/kg		-0,638	0,94	30	0,85	0,94	0,18	19,1	24	2	0	13
	R21	mg/kg		-0,504	0,357	30	0,33	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		-3,43	0,99	30	0,48	0,99	0,23	23,7	28	2	0	15
Zn	R1	mg/kg			0,192		0,25	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		0,08	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		0,16	0,19	0,08	46,0	11	9	5	15
Laboratory 17														
As	R1	mg/kg		1,96	28	25	34,85	27,7	3,01	10,8	21	9	0	15
	R21	mg/kg		1,11	8,1	25	9,22	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		3,85	29,5	25	43,7	29,4	2,12	7,2	17	13	0	15
Ba	R1	mg/kg			0,055		0,211	0,06	0,02	44,8	10	6	4	12
	R21	mg/kg			0,039		0,070	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		0,147	0,07	0,03	50,2	8	6	5	12
Cd	R1	mg/kg					<0,01	0,74	0,56	75,0	4	0	12	14
	R21	mg/kg					<0,00	0,08	0	0	2	0	13	14
	R22	mg/kg					<0,01	0,29	0,01	5,5	2	0	13	14
conductivity	R1	mS/m		9,61	0,985	30	2,405	1,05	0,21	20,3	19	9	1	15
	R21	mS/m		4,83	2,12	30	3,655	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		5,74	1,19	30	2,215	1,23	0,43	35,4	20	6	1	14
Cr	R1	mg/kg		3,37	1,52	40	2,545	1,51	0,37	24,9	22	6	0	14
	R21	mg/kg		1,85	0,641	40	0,878	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		3,31	1,52	40	2,525	1,53	0,26	17,6	19	9	0	14
Cu	R1	mg/kg		-2,28	3,13	40	1,7	3,21	0,58	18,2	17	9	0	13
	R21	mg/kg		-2,6	1,05	40	0,503	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		-1,91	2,72	40	1,68	2,88	1,37	47,6	25	3	0	14
DOC	R1	mg/kg		10,8	45,2	25	106,5	46,7	7,84	16,7	18	3	0	11
	R21	mg/kg		5,57	27,8	25	47,15	28,7	5,73	19,9	17	2	0	10
	R22	mg/kg		8,2	51,1	25	103,5	54,8	15,1	27,6	14	4	0	10
Fe	R1	mg/kg			0,861		2,195	0,93	0,51	55,1	20	2	2	13
	R21	mg/kg			0,234		0,687	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		2,245	1,19	0,79	66,5	17	3	2	12
Mo	R1	mg/kg					<0,1	0,00	0,00	14,5	2	0	14	15
	R21	mg/kg					<0,02	0,00	0	0	2	0	12	13
	R22	mg/kg					<0,1	0,00	0,00	26,7	2	0	12	13
Ni	R1	mg/kg			0,041		<0,1	0,05	0,05	91,5	9	2	9	15
	R21	mg/kg			0,007		<0,02	0,00	0,00	21,4	8	4	8	14
	R22	mg/kg			0,024		<0,10	0,01	0,00	51,0	6	4	9	14
Pb	R1	mg/kg			0,042		0,153	0,04	0,03	69,8	19	2	3	14
	R21	mg/kg			0,023		0,037	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		0,115	0,05	0,04	71,0	21	0	4	15
pH	R1			7,94	5,77	6,9	7,35	5,8	0,32	5,6	25	4	0	15
	R21			5,79	6,03	6,7	7,2	6,03	0,43	7,2	26	3	0	15
	R22			5,61	6,09	6,5	7,2	6,10	0,45	7,4	25	2	0	14

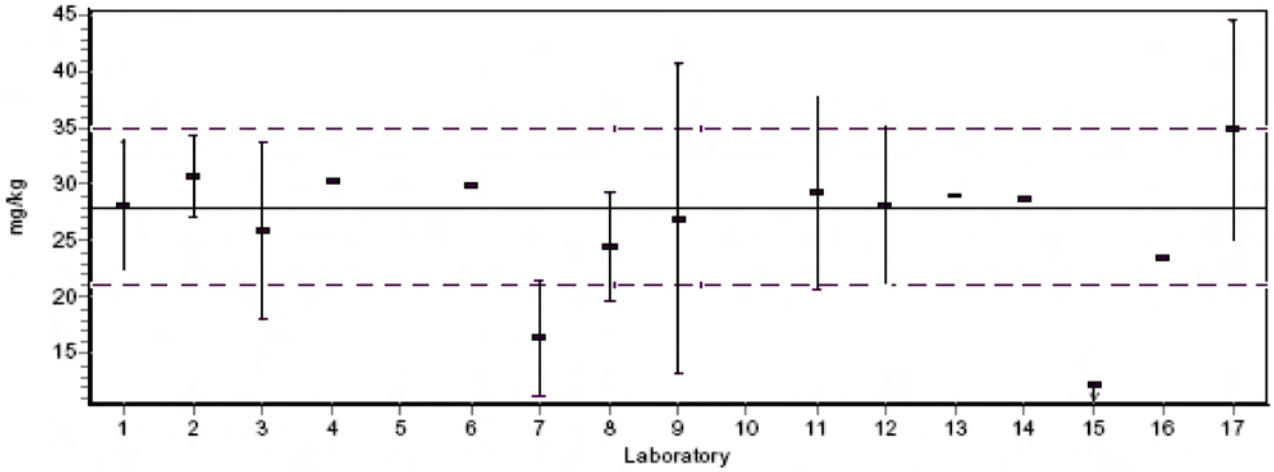
Outlier test failed: C - Cohcran, G1 - Grubbs(1-outlier algorithm), G2 - Grubbs(2-outliers algorithm), H - Hampel, M - manual

Analyte	Sample	Unit	z-Graphics -3 -2 -1 0 +1 +2 +3	Z-value	Assig-ned value	2* Targ SD%	Lab's result	R-mean	SD rob	SD rob %	Pas-sed	R-ad just-ed	Mis-sing	Num of labs
Sb	R1	mg/kg		0,851	0,94	30	1,06	0,94	0,18	19,1	24	2	0	13
Laboratory 17														
Sb	R21	mg/kg		0,513	0,357	30	0,384	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		1,75	0,99	30	1,25	0,99	0,23	23,7	28	2	0	15
Se	R1	mg/kg					<0,2	0,00	0,00	47,5	2	0	11	12
	R21	mg/kg					<0,04	0,00	9,62	3,6	2	0	11	12
	R22	mg/kg					<0,20	0,00	0,00	14,6	2	0	11	12
Zn	R1	mg/kg			0,192		0,156	0,22	0,09	42,9	14	6	5	15
	R21	mg/kg			0,073		0,057	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		0,156	0,19	0,08	46,0	11	9	5	15
Laboratory 18														
As	R21	mg/kg		0,504	8,1	25	8,61	8,04	0,91	11,3	24	6	0	15
	R22	mg/kg		0,108	29,5	25	29,9	29,4	2,12	7,2	17	13	0	15
Ba	R21	mg/kg			0,039		0,04	0,04	0,02	61,5	10	6	4	12
	R22	mg/kg			0,057		0,055	0,07	0,03	50,2	8	6	5	12
conductivity	R21	mS/m		0,66	2,12	30	2,33	2,20	0,58	26,3	25	4	0	15
	R22	mS/m		0,028	1,19	30	1,195	1,23	0,43	35,4	20	6	1	14
Cr	R21	mg/kg		0,702	0,641	40	0,731	0,64	0,15	24,5	21	7	0	14
	R22	mg/kg		-0,255	1,52	40	1,442	1,53	0,26	17,6	19	9	0	14
Cu	R21	mg/kg		0,576	1,05	40	1,171	1,06	0,47	44,9	21	7	0	14
	R22	mg/kg		1,78	2,72	40	3,688	2,88	1,37	47,6	25	3	0	14
DOC	R21	mg/kg		2,37	27,8	25	36,03	28,7	5,73	19,9	17	2	0	10
	R22	mg/kg		4,73	51,1	25	81,32	54,8	15,1	27,6	14	4	0	10
Fe	R21	mg/kg			0,234		0,530	0,27	0,18	67,9	12	7	2	12
	R22	mg/kg			0,939		0,908	1,19	0,79	66,5	17	3	2	12
Pb	R21	mg/kg			0,023		0,039	0,02	0,02	77,4	20	1	4	15
	R22	mg/kg			0,049		0,026	0,05	0,04	71,0	21	0	4	15
pH	R21			2,82	6,03	6,7	6,6	6,03	0,43	7,2	26	3	0	15
	R22			0,556	6,09	6,5	6,2	6,10	0,45	7,4	25	2	0	14
Sb	R21	mg/kg		0,243	0,357	30	0,37	0,36	0,04	11,7	21	9	0	15
	R22	mg/kg		-0,31	0,99	30	0,944	0,99	0,23	23,7	28	2	0	15
Zn	R21	mg/kg			0,073		0,08	0,07	0,03	41,1	17	5	3	15
	R22	mg/kg			0,237		0,277	0,19	0,08	46,0	11	9	5	15

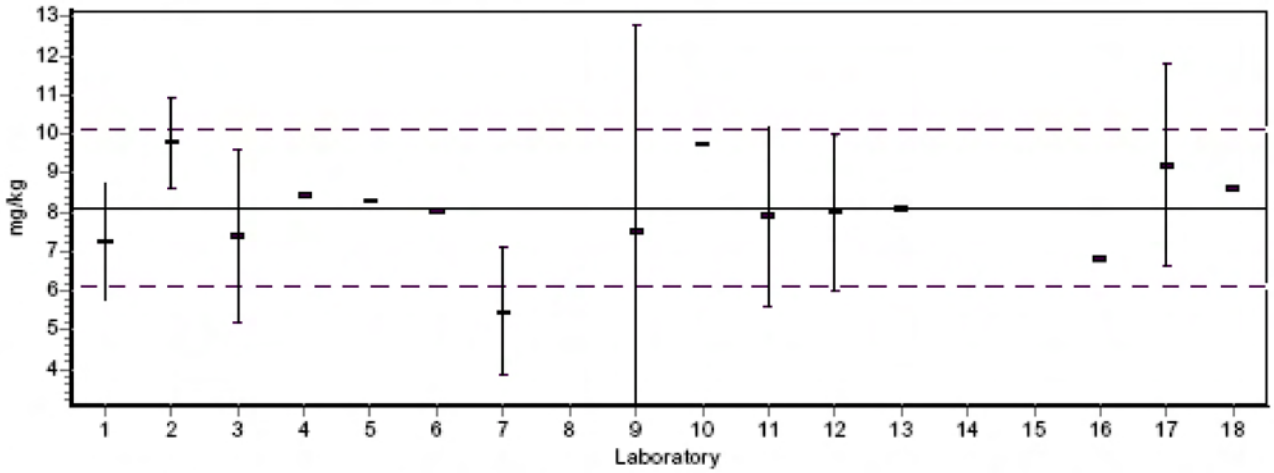
LIITE 8. RESULTS AND THEIR UNCERTAINTIES REPORTED BY THE PARTICIPANTS

Appendix 8.

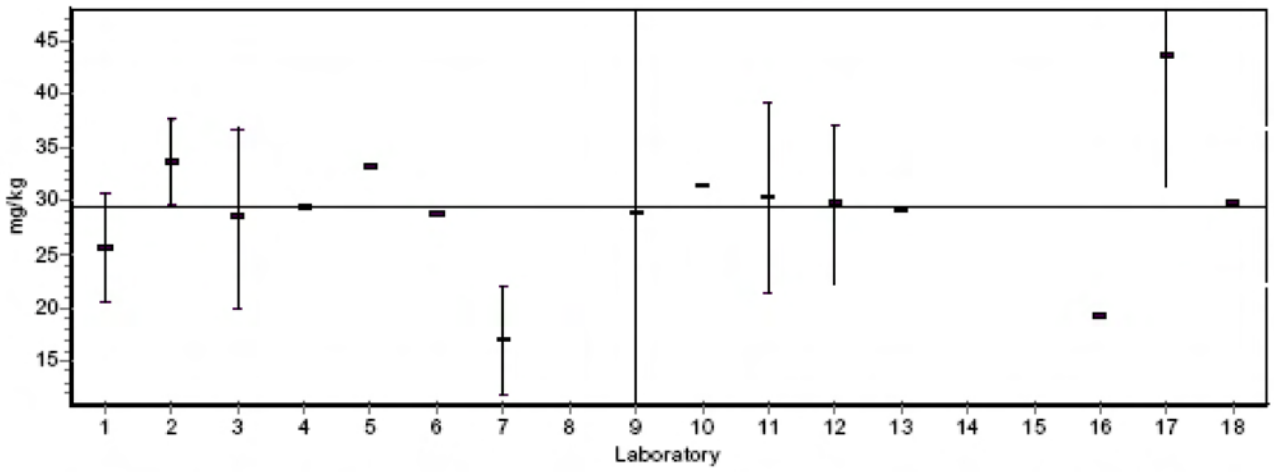
Analyytti *(Analyte)* As Näyte *(Sample)* R1

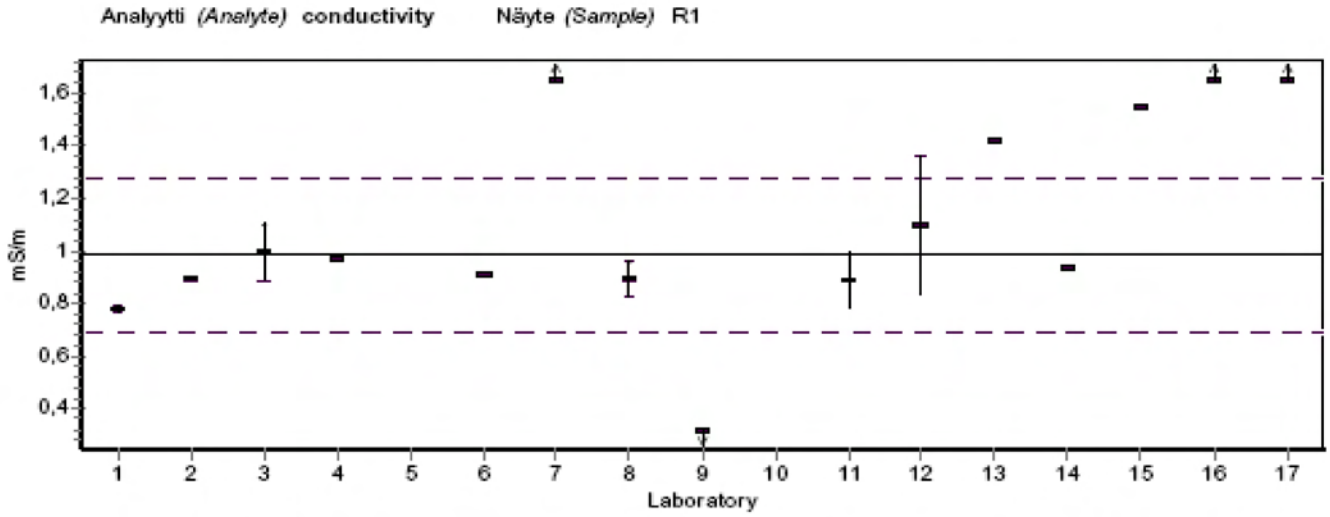


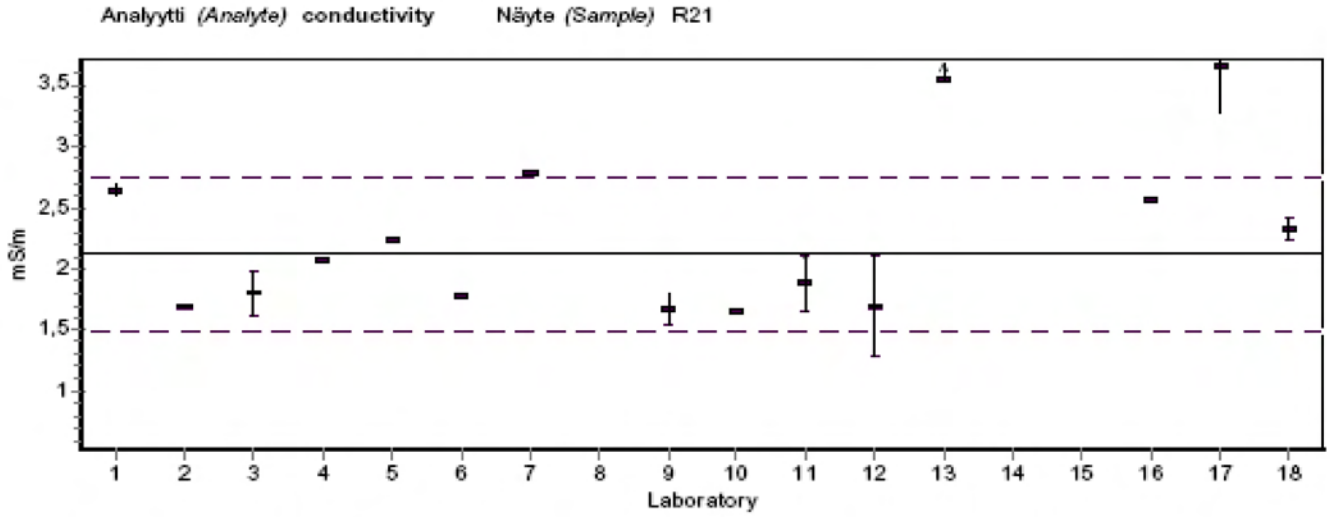
Analyytti *(Analyte)* As Näyte *(Sample)* R21

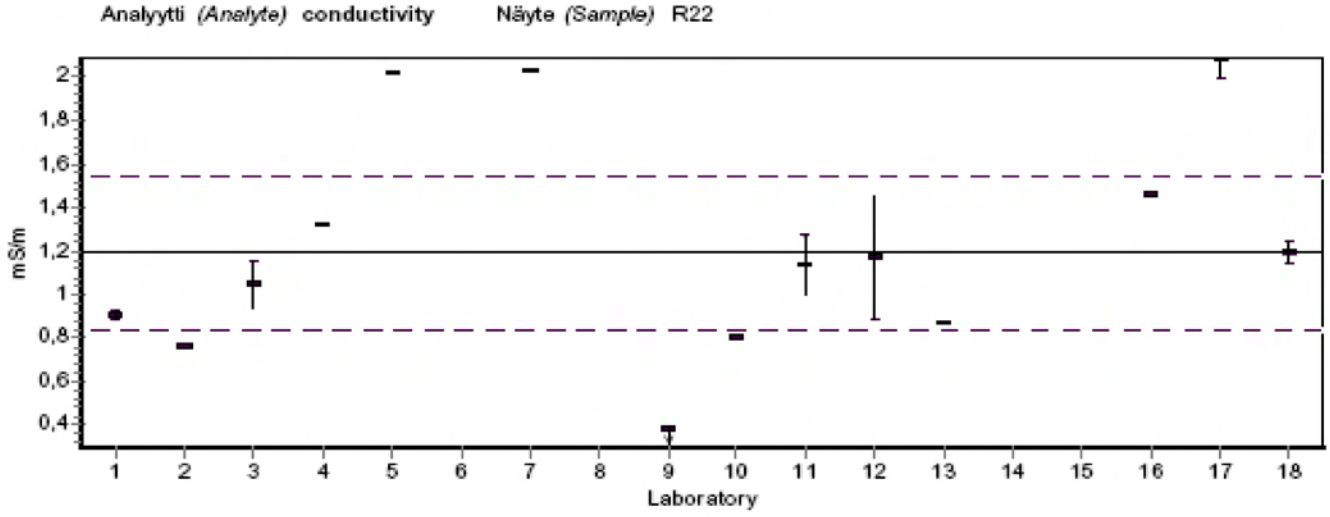


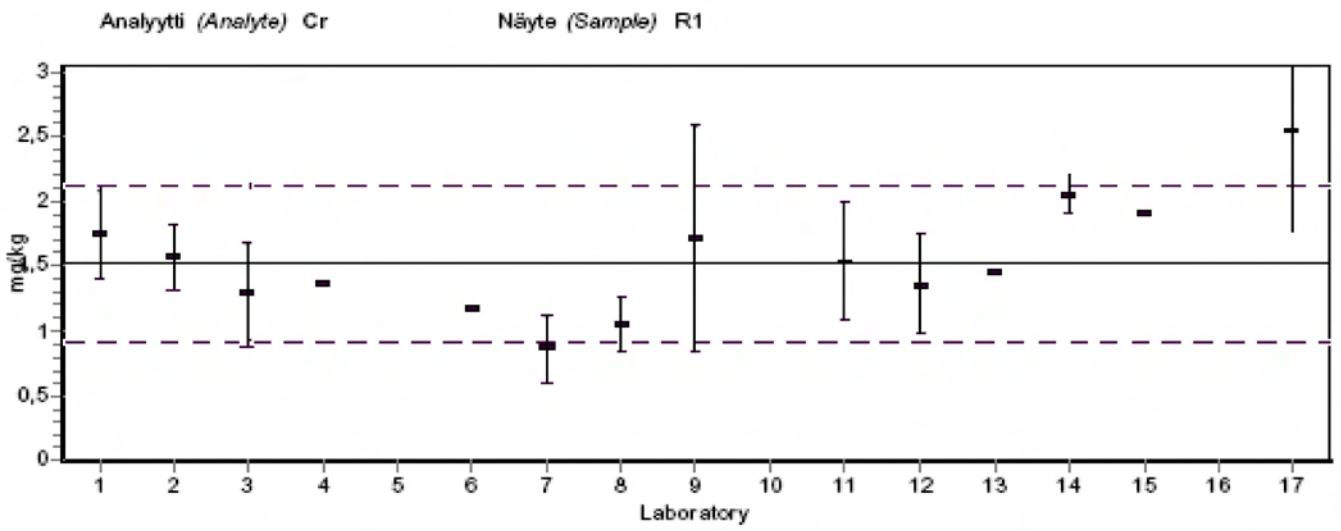
Analyytti *(Analyte)* As Näyte *(Sample)* R22

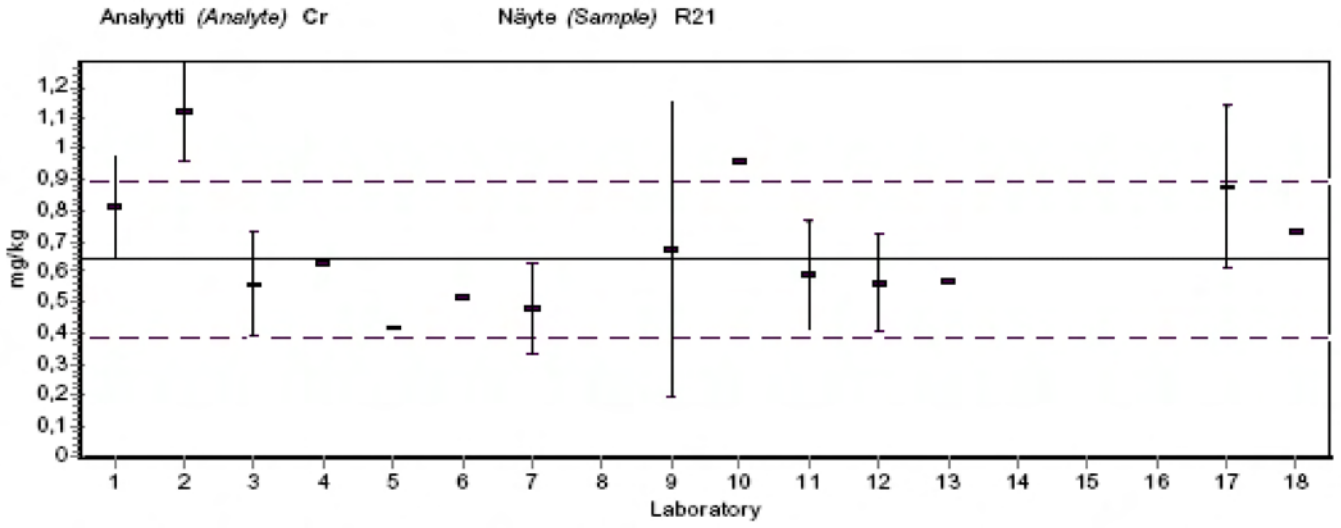


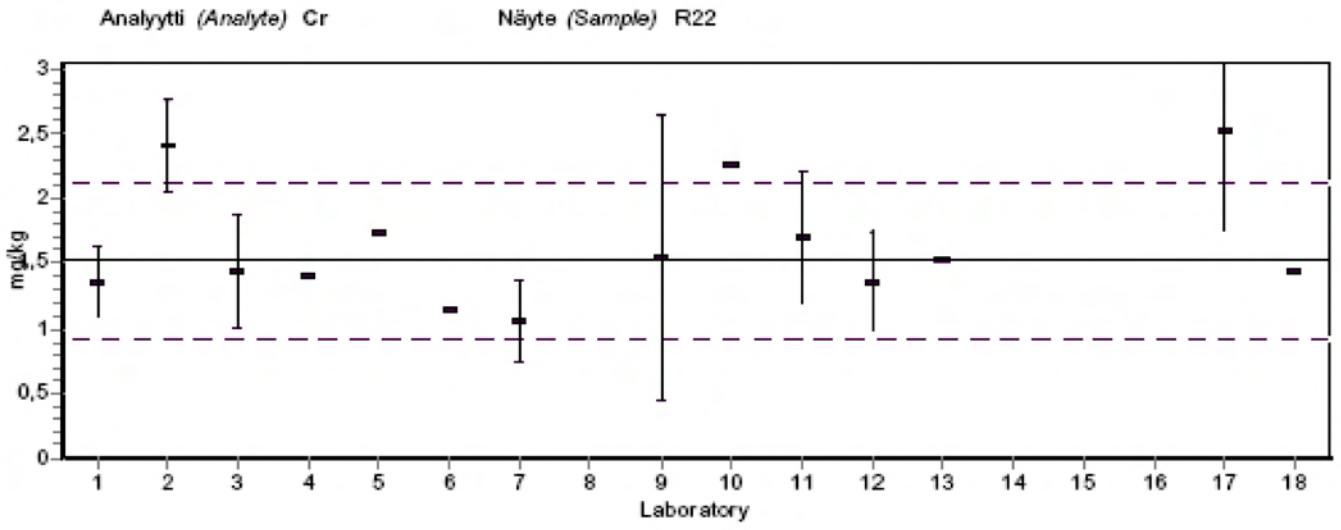

Analyytti (Analyte) conductivity
Näyte (Sample) R1
mS/m
1,6
1,4
1,2
1
0,8
0,6
0,4
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory

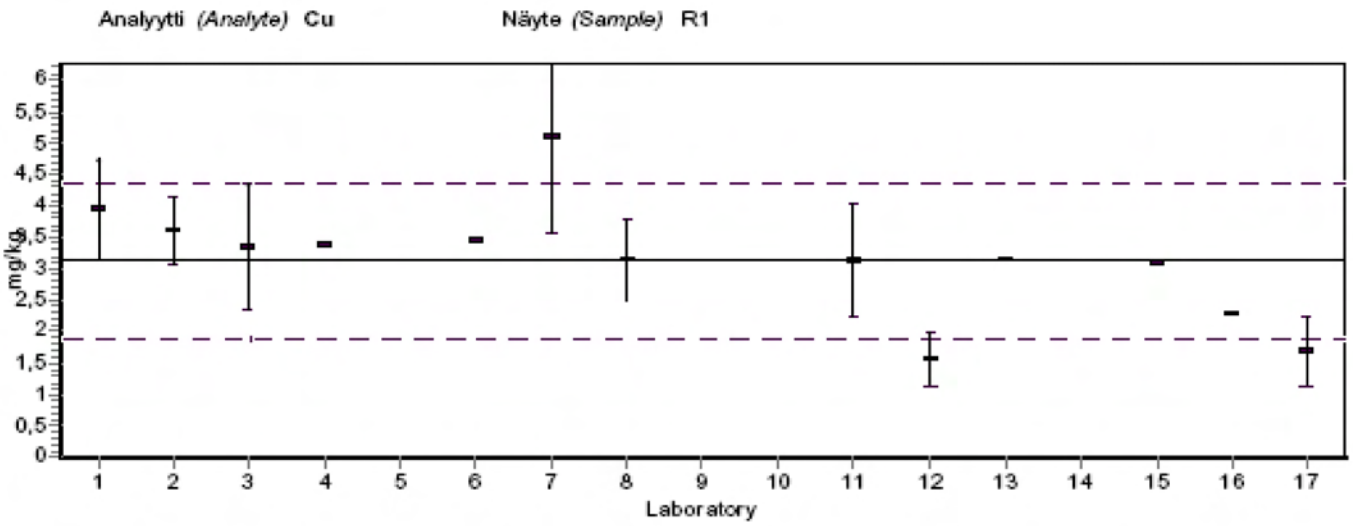

Analyytti (Analyte) conductivity
Näyte (Sample) R21
mS/m
3,5
3
2,5
2
1,5
1
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
Laboratory

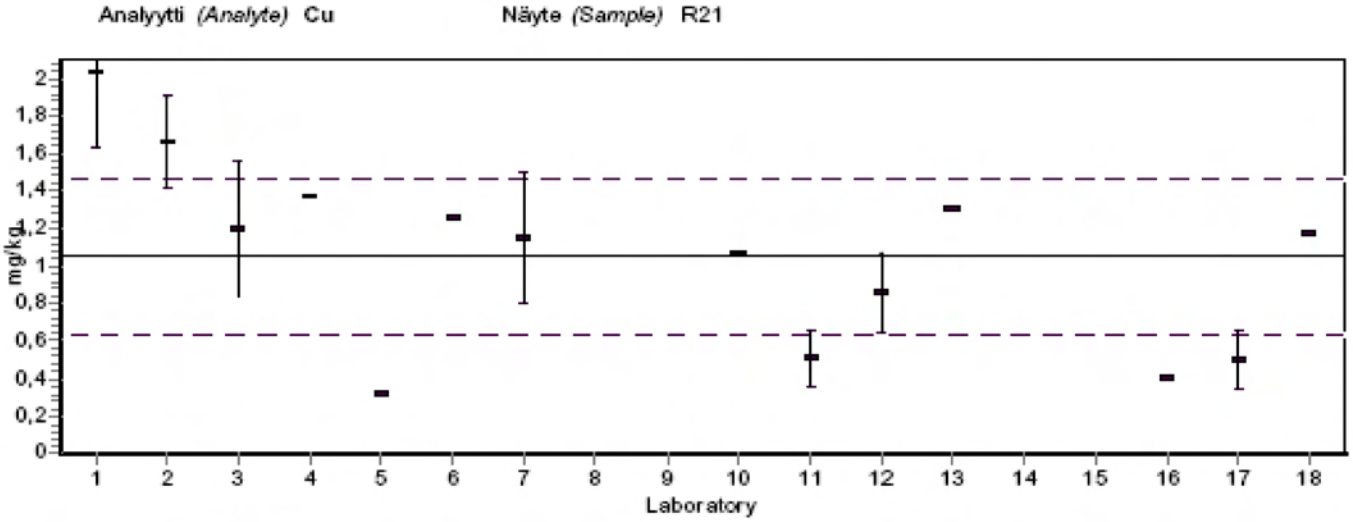

Analyytti (Analyte) conductivity
Näyte (Sample) R22
mS/m
2
1,8
1,6
1,4
1,2
1
0,8
0,6
0,4
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
Laboratory

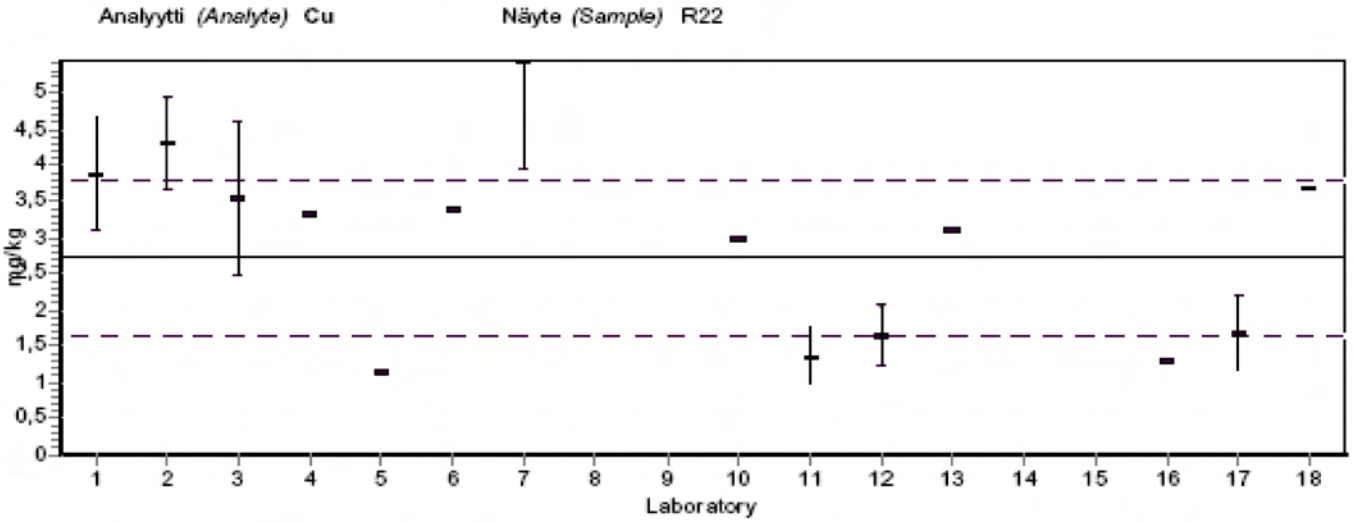

Analyytti (Analyte) Cr
Näyte (Sample) R1
mg/kg
Laboratory

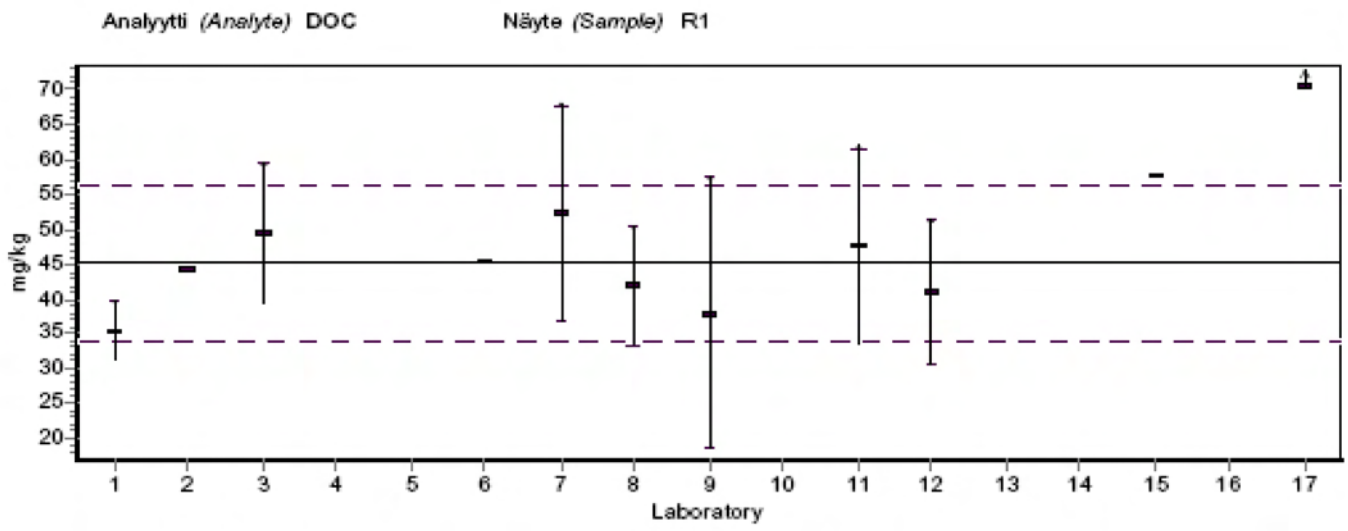

Analyytti (Analyte) Cr
Näyte (Sample) R21
mg/kg
Laboratory

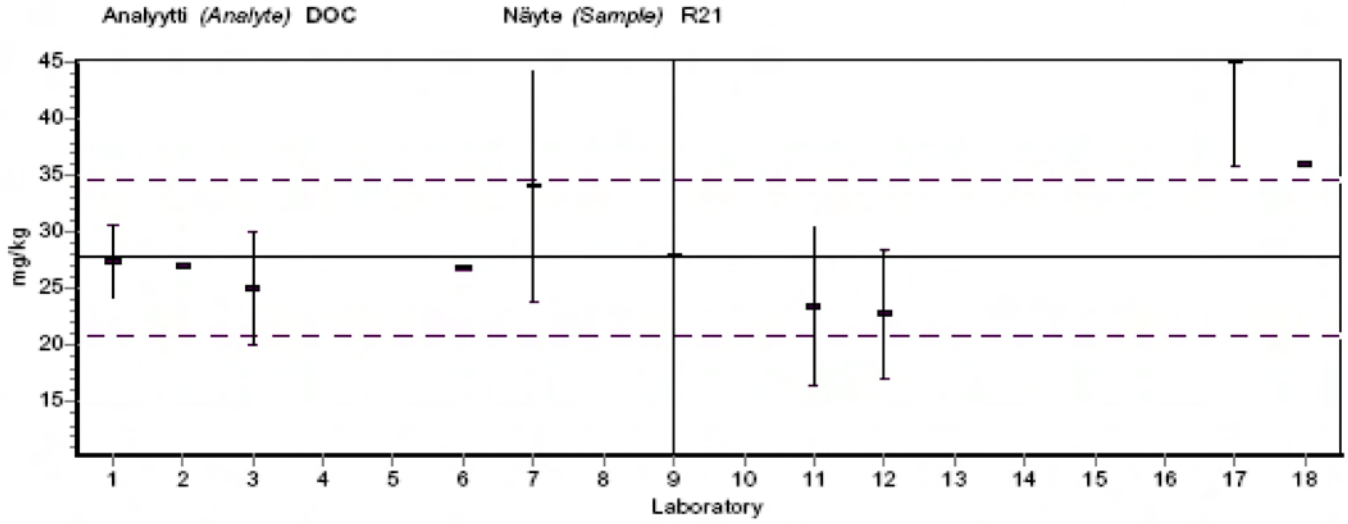

Analyytti (Analyte) Cr
Näyte (Sample) R22
mg/kg
Laboratory

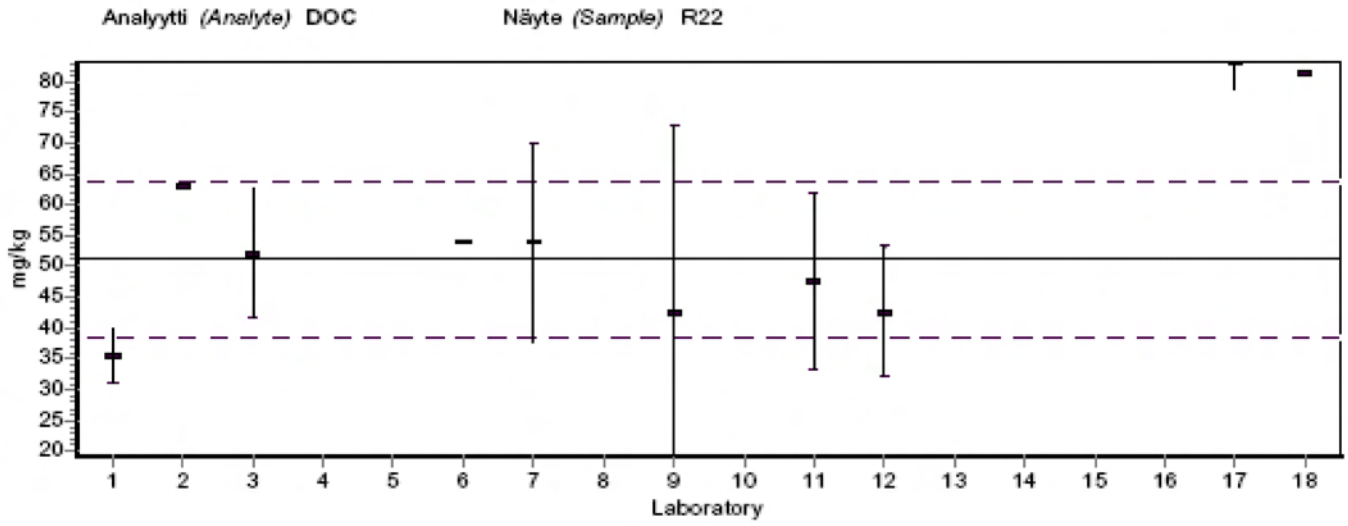

Analyytti (Analyte) Cu
Näyte (Sample) R1
mg/kg
Laboratory

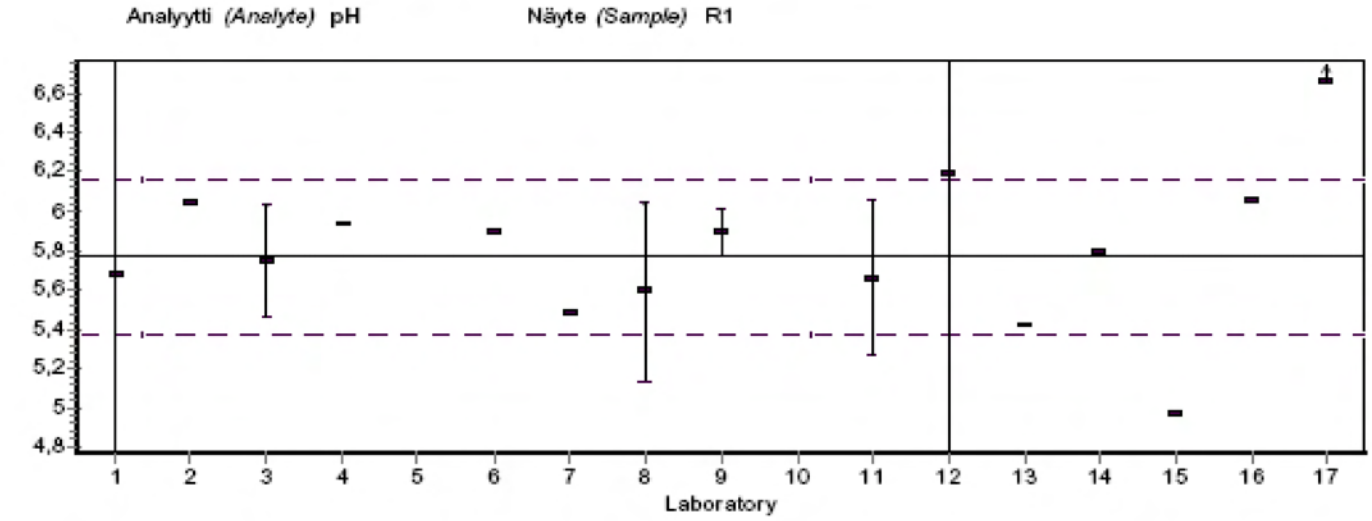

Analyytti (Analyte) Cu
Näyte (Sample) R21
mg/kg
Laboratory

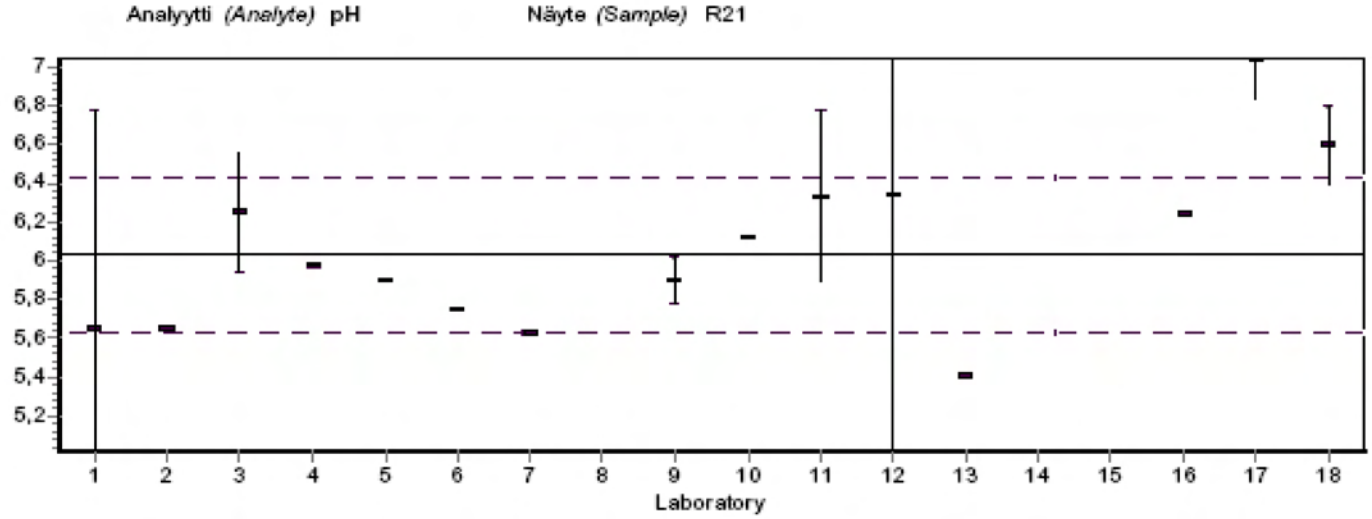

Analyytti (Analyte) Cu
Näyte (Sample) R22
mg/kg
Laboratory

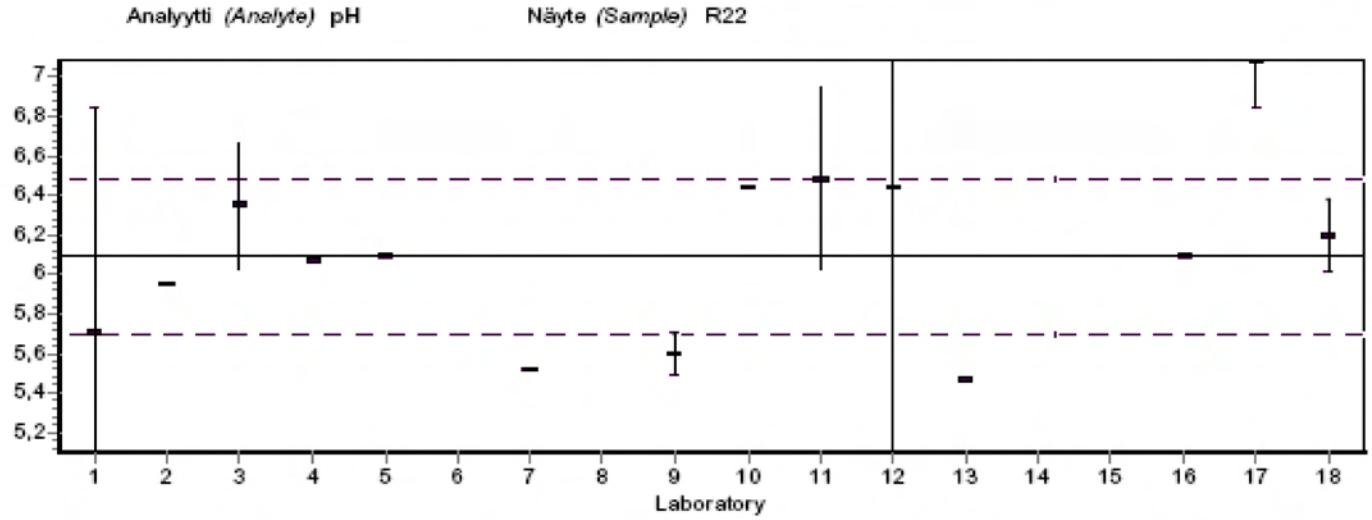

Analyytti (Analyte) DOC
Näyte (Sample) R1
mg/kg
70
65
60
55
50
45
40
35
30
25
20
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory

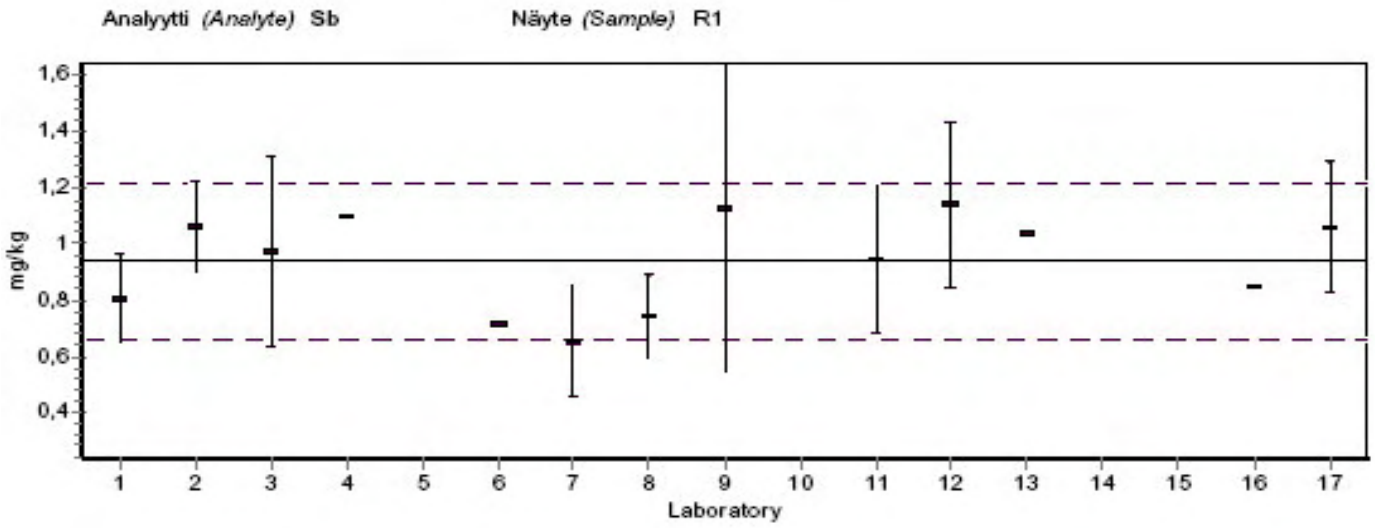

Analyytti (Analyte) DOC
Näyte (Sample) R21
mg/kg
45
40
35
30
25
20
15
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
Laboratory

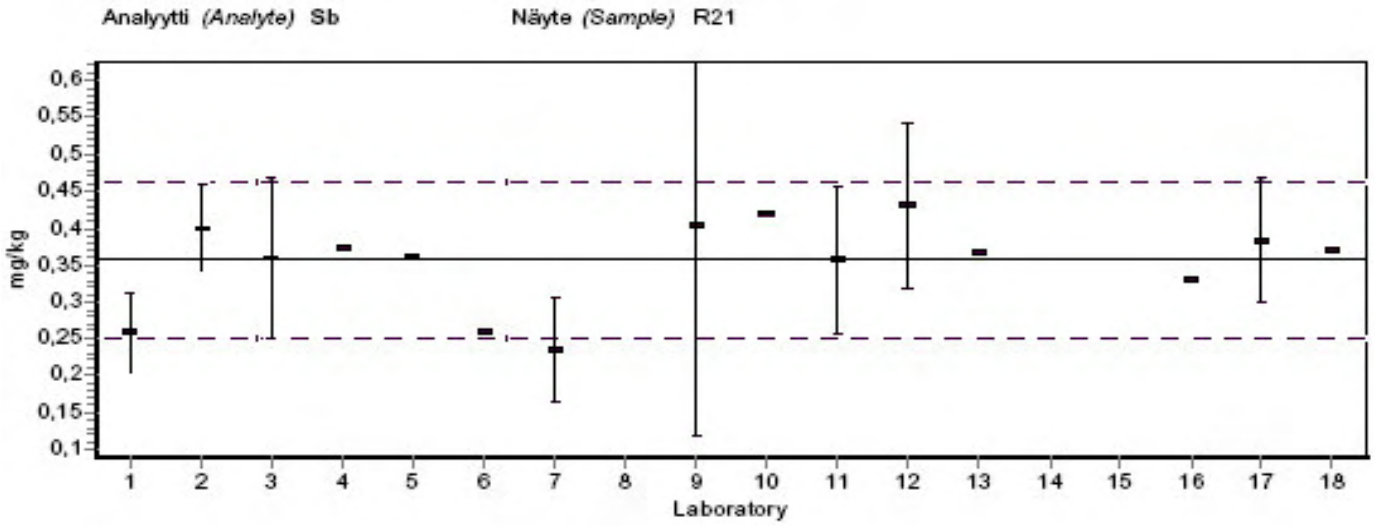

Analyytti (Analyte) DOC
Näyte (Sample) R22
mg/kg
80
75
70
65
60
55
50
45
40
35
30
25
20
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
Laboratory

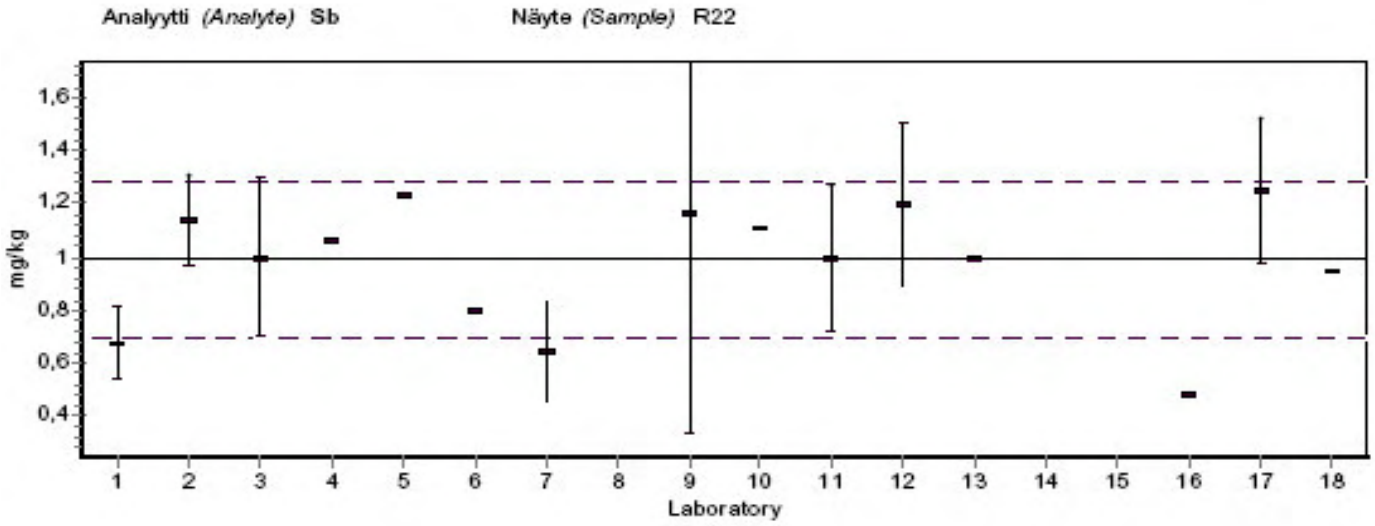

Analyytti (Analyte) pH
Näyte (Sample) R1
6,6
6,4
6,2
6
5,8
5,6
5,4
5,2
5
4,8
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory


Analyytti (Analyte) pH
Näyte (Sample) R21
7
6,8
6,6
6,4
6,2
6
5,8
5,6
5,4
5,2
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
Laboratory


Analyytti (Analyte) pH
Näyte (Sample) R22
7
6,8
6,6
6,4
6,2
6
5,8
5,6
5,4
5,2
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
Laboratory


Analyytti (Analyte) Sb
Näyte (Sample) R1
mg/kg
1,6
1,4
1,2
1
0,8
0,6
0,4
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Laboratory


Analyytti (Analyte) Sb
Näyte (Sample) R21
mg/kg
0,6
0,55
0,5
0,45
0,4
0,35
0,3
0,25
0,2
0,15
0,1
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
Laboratory


Analyytti (Analyte) Sb
Näyte (Sample) R22
mg/kg
1,6
1,4
1,2
1
0,8
0,6
0,4
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
Laboratory

LIITE 9. ORGINAL RESULTS REPORTED BY THE PARTICIPANTS

Appendix 9.

Analyte	Sample	Unit	1			2			3			4			5			6		
As	R1	mg/kg	28,5	28,0	1	30,5	31,0	1	26	26	1	30,12	30,86	1				30,2	29,6	1
	R21	mg/kg	7,21	7,28	1	9,76	9,80	1	7,4	7,4	1	8,502	8,412	1	8,26	8,31	1	7,95	8,11	1
	R22	mg/kg	25,3	26,1	1	33,4	33,9	1	29	28	1	29,758	29,066	1	33,3	33,2	1	28,5	29,2	1
Ba	R1	mg/kg	0,034	0,038	1	0,044	0,047	1	<2	<2	1	<0,05	<0,05	1				<0,1	<0,1	1
	R21	mg/kg	0,026	0,028	1	0,024	0,024	1	<0,7	<0,7	1	0,020	0,016	1	<0,01	<0,01	1	<0,1	<0,1	1
	R22	mg/kg	0,046	0,037	1	0,052	0,057	1	<2	<2	1	<0,06	<0,05	1	<0,01	<0,01	1	<0,1	<0,1	1
Cd	R1	mg/kg	<0,001	<0,001	1	<0,015	<0,015	1	<0,004	<0,004	1	<0,004	<0,004	1				0,32	0,32	1
	R21	mg/kg	<0,001	<0,001	1	<0,003	<0,003	1	<0,003	<0,003	1	<0,000	<0,000	1	<0,01	<0,01	1	0,08	0,08	1
	R22	mg/kg	<0,001	<0,001	1	<0,015	<0,015	1	<0,004	<0,004	1	<0,004	<0,004	1	<0,01	<0,01	1	0,28	0,30	1
conductivity	R1	mS/m	0,79	0,77	1	0,90	0,89	1	1,0	1,0	1	1,04	0,90	1				0,97	0,86	1
	R21	mS/m	2,64	2,64	1	1,69	1,69	1	1,9	1,7	1	2,30	1,84	1	2,36	2,11	1	1,75	1,79	1
	R22	mS/m	0,93	0,89	1	0,77	0,76	1	1,2	0,9	1	1,73	0,91	1	2,10	1,95	1			
Cr	R1	mg/kg	1,82	1,67	1	1,58	1,58	1	1,3	1,3	1	1,415	1,331	1				1,16	1,20	1
	R21	mg/kg	0,80	0,83	1	1,11	1,14	1	0,56	0,56	1	0,679	0,580	1	0,403	0,429	1	0,51	0,53	1
	R22	mg/kg	1,38	1,34	1	2,38	2,43	1	1,4	1,5	1	1,455	1,356	1	1,82	1,65	1	1,09	1,20	1
Cu	R1	mg/kg	4,13	3,78	1	3,55	3,69	1	3,3	3,4	1	3,280	3,469	1				3,61	3,35	1
	R21	mg/kg	2,13	1,95	1	1,65	1,67	1	1,2	1,2	1	1,493	1,256	1	0,276	0,359	1	1,25	1,26	1
	R22	mg/kg	3,97	3,79	1	4,20	4,44	1	3,6	3,5	1	3,521	3,177	1	1,19	1,08	1	3,45	3,33	1
DOC	R1	mg/kg	35,8	35,2	1	45,0	44,0	1	49	50	1							46,0	45,0	1
	R21	mg/kg	27,2	27,8	1	26,1	28,1	1	24	26	1							26,0	28,0	1
	R22	mg/kg	35,4	35,6	1	62,7	63,6	1	<50	52	1							55,2	52,4	1
Fe	R1	mg/kg	1,42	0,97	1	0,65	0,65	1	0,57	0,61	1							<1	<1	1
	R21	mg/kg	0,43	0,57	1	1,10	1,17	1	<0,2	<0,2	1				0,123	0,189	1	<1	<1	1
	R22	mg/kg	0,86	0,77	1	1,95	2,24	1	0,56	0,65	1				2,32	2,02	1	<1	<1	1
Mo	R1	mg/kg	<0,005	<0,005	1	<0,05	<0,05	1	<0,05	<0,05	1	<0,05	<0,05	1				<0,1	<0,1	1
	R21	mg/kg	<0,005	<0,005	1	<0,01	<0,01	1	<0,03	<0,03	1	<0,01	<0,01	1	<0,01	<0,01	1	<0,1	<0,1	1
	R22	mg/kg	<0,005	<0,005	1	<0,05	<0,05	1	<0,05	<0,05	1	<0,05	<0,05	1	<0,01	<0,01	1	<0,1	<0,1	1
Ni	R1	mg/kg	0,209	0,196	1	<0,1	<0,1	1	<0,04	<0,04	1	<0,04	<0,04	1				<0,1	<0,1	1
	R21	mg/kg	0,009	0,009	1	<0,02	<0,02	1	<0,02	<0,02	1	<0,008	<0,008	1	<0,01	<0,01	1	<0,1	<0,1	1
	R22	mg/kg	0,097	0,089	1	<0,1	<0,1	1	<0,04	<0,04	1	<0,04	<0,04	1	<0,01	<0,01	1	<0,1	<0,1	1
Pb	R1	mg/kg	0,073	0,073	1	0,028	0,027	1	<0,05	<0,05	1	0,070	0,087	1				<0,1	<0,1	1
	R21	mg/kg	0,044	0,047	1	0,057	0,063	1	<0,02	<0,02	1	0,031	0,017	1	<0,01	0,014	1	<0,1	<0,1	1
	R22	mg/kg	0,042	0,051	1	0,096	0,104	1	<0,05	<0,05	1	<0,07	<0,06	1	0,098	0,090	1	<0,1	<0,1	1
pH	R1		5,71	5,66	1	6,1	6,0	1	5,8	5,7	1	6,03	5,85	1				5,9	5,9	1
	R21		5,65	5,65	1	5,6	5,7	1	6,4	6,1	1	5,81	6,14	1	6,0	5,8	1	5,7	5,8	1
	R22		5,79	5,62	1	6,0	5,9	1	6,6	6,1	1	6,06	6,08	1	6,2	6,0	1			
Sb	R1	mg/kg	0,815	0,796	1	1,06	1,07	1	0,98	0,97	1	1,098	1,097	1				0,72	0,71	1
	R21	mg/kg	0,26	0,258	1	0,40	0,40	1	0,36	0,36	1	0,376	0,370	1	0,350	0,374	1	0,24	0,28	1
	R22	mg/kg	0,67	0,68	1	1,14	1,14	1	1,0	1,0	1	1,070	1,063	1	1,26	1,21	1	0,78	0,82	1
Se	R1	mg/kg	<0,002	<0,002	1	<0,05	<0,05	1	<0,01	<0,01	1	<0,05	<0,05	1				<0,01	<0,01	1
	R21	mg/kg	<0,002	<0,002	1	<0,01	<0,01	1	<0,006	<0,006	1	<0,01	<0,01	1	<0,01	<0,01	1	<0,01	<0,01	1
	R22	mg/kg	<0,002	<0,002	1	<0,05	<0,05	1	<0,01	<0,01	1	<0,05	<0,05	1	<0,01	<0,01	1	<0,01	<0,01	1
Zn	R1	mg/kg	0,191	0,167	1	0,151	0,150	1	<0,4	<0,4	1	<0,4	<0,4	1				<0,1	<0,1	1
	R21	mg/kg	0,078	0,071	1	0,049	0,047	1	<0,2	<0,2	1	0,10	<0,08	1	<0,01	0,011	1	<0,1	<0,1	1
	R22	mg/kg	0,139	0,129	1	0,163	0,157	1	<0,4	<0,4	1	<0,4	<0,4	1	0,020	0,027	1	<0,1	<0,1	1
Analyte	**Sample**	**Unit**	**7**			**8**			**9**			**10**			**11**			**12**		
As	R1	mg/kg	16,6	16,6	1	25	24		26,7	27,3	1				29,4	29,2		28.2	28.3	1
	R21	mg/kg	5,4	5,5	1				7,5	7,5	1	8,81	10,7	1	7,72	8,07		8.0	8.0	1
	R22	mg/kg	16,9	17,1	1				29,5	28,4	1	28,3	34,7	1	30,0	30,7		30.1	29.5	1
Ba	R1	mg/kg	0,1	0,1	1	0,08	0,06		0,0449	0,0403	1				0,0494	0,0433		0.81	0.86	1
	R21	mg/kg	<0,02	<0,02	1				0,0148	0,0149	1				0,0842	0,0671		0.12	0.12	1
	R22	mg/kg	<0,1	<0,1	1				0,0389	0,0454	1				0,215	0,158		0.58	0.51	1
Cd	R1	mg/kg	<0,001	<0,01	1	<0,002	<0,002		<0,000	<0,000	1				<0,020	<0,020		<0.01	<0.01	1
	R21	mg/kg	<0,002	<0,003	1				<0,000	<0,000	1	<0,002	<0,002	1	<0,020	<0,020		<0.01	<0.01	1
	R22	mg/kg	<0,001	<0,001	1				<0,000	<0,000	1	<0,010	<0,010	1	<0,020	<0,020		<0.01	<0.01	1
conductivity	R1	mS/m	2,11	2,06	1	0,92	0,87		<1		1				0,92	0,86		1.10	1.09	1
	R21	mS/m	2,75	2,82	1				1,67		1	1,78	1,53	1	1,80	1,96		1.69	1.70	1
	R22	mS/m	2,08	1,98	1				<1		1	0,83	0,77	1	1,09	1,19		1.29	1.05	1
Cr	R1	mg/kg	0,87	0,87	1	1,1	1,0		1,77	1,66	1				1,56	1,52		1.34	1.38	1
	R21	mg/kg	0,48	0,49	1				0,684	0,658	1	0,952	0,970	1	0,561	0,620		0.57	0.56	1
	R22	mg/kg	1,0	1,1	1				1,51	1,58	1	2,32	2,19	1	1,65	1,75		1.39	1.33	1
Cu	R1	mg/kg	5,0	5,2	1	3,2	3,1								3,12	3,14		1.64	1.53	1
	R21	mg/kg	1,2	1,1	1							1,06	1,09	1	0,577	0,432		0.89	0.83	1
	R22	mg/kg	5,7	5,6	1							3,07	2,93	1	1,47	1,25		1.66	1.65	1
DOC	R1	mg/kg	54,0	51,0	1	41	43		38	-	1				48,4	46,9		41.9	40.2	1
	R21	mg/kg	34,1	34,1	1				28	-	1				23,1	23,9		23.0	22.5	1
	R22	mg/kg	52,4	55,3	1				42,6	-	1				47,1	48,0		43.3	42.1	1
Fe	R1	mg/kg	<0,5	<0,5	1	0,42	0,24		0,999	0,921	1				0,995	0,980		0.26	0.34	1
	R21	mg/kg	<0,1	0,1	1				0,156	0,15	1				0,137	0,246		0.05	0.07	1

Analyte	Sample	Unit	7			8			9			10			11			12		
Fe	R22	mg/kg	<0,5	<0,5	1				0,462	0,472	1				1,14	1,36		0.35	0.28	1
Mo	R1	mg/kg	<0,03	<0,03	1	<0,02	<0,02		<0,005	<0,005	1				<0,020	<0,020		0.006	0.005	1
	R21	mg/kg	<0,006	<0,006	1				<0,001	<0,001	1				<0,020	<0,020		0.001	0.001	1
	R22	mg/kg	<0,03	<0,03	1				<0,005	<0,005	1				<0,020	<0,020		0.005	0.007	1
Ni	R1	mg/kg	0,06	0,02	1	0,06	<0,02		0,0186	0,0172	1				<0,040	<0,040		0.014	0.013	1
	R21	mg/kg	0,01	0,009	1				0,0034	0,0041	1	0,0090	0,0105	1	<0,040	<0,040		0.004	0.004	1
	R22	mg/kg	0,05	0,04	1				0,0139	0,0126	1	<0,032	<0,033	1	<0,040	<0,040		0.012	0.013	1
Pb	R1	mg/kg	0,02	0,02	1	0,02	0,02		0,0449	0,0492	1				0,0496	0,0501		0.014	0.018	1
	R21	mg/kg	0,01	0,02	1				0,0089	0,0090	1	0,0435	0,0327	1	<0,020	<0,020		0.003	0.004	1
	R22	mg/kg	0,03	0,03	1				0,0253	0,0244	1	0,0963	0,0844	1	0,0635	0,0570		0.017	0.014	1
pH	R1		5,46	5,51	1	5,5	5,7		5,9		1				5,71	5,61		6.21	6.18	1
	R21		5,64	5,61	1				5,9		1	6,18	6,06	1	6,20	6,47		6.36	6.32	1
	R22		5,55	5,50	1				5,6		1	6,42	6,46	1	6,45	6,52		6.43	6.46	1
Sb	R1	mg/kg	0,66	0,65	1	0,75	0,74		1,06	1,19	1				0,945	0,942		1.13	1.15	1
	R21	mg/kg	0,24	0,23	1				0,406	0,404	1	0,393	0,447	1	0,358	0,356		0.43	0.43	1
	R22	mg/kg	0,64	0,65	1				1,16	1,17	1	1,10	1,12	1	0,998	0,995		1.19	1.20	1
Se	R1	mg/kg	<0,02	<0,02	1	<0,01	<0,01		0,011	0,0059	1				<0,020	<0,020		<0.001	<0.001	1
	R21	mg/kg	<0,004	<0,004	1				0,0025	0,0027	1				<0,020	<0,020		<0.001	<0.001	1
	R22	mg/kg	<0,02	<0,02	1				0,0060	0,0072	1				<0,020	<0,020		<0.001	<0.001	1
Zn	R1	mg/kg	0,39	0,38	1	0,13	0,13		0,146	0,143	1				<0,20	<0,20		0.76	0.69	1
	R21	mg/kg	0,09	0,07	1				0,0446	0,0436	1	0,0995	0,0803	1	<0,20	<0,20		0.16	0.16	1
	R22	mg/kg	0,43	0,39	1				0,13	0,141	1	<0,416	<0,400	1	<0,20	<0,20		0.54	0.59	1
Analyte	**Sample**	**Unit**	**13**			**14**			**15**			**16**			**17**			**18**		
As	R1	mg/kg	29,00	29,00	1	28,71	28,64	1	3,24	2,98	1	23	24	1	35,1	34,6	1			
	R21	mg/kg	8,40	7,80	1							6,8	6,8	1	9,33	9,11	1	8,61	8,61	1
	R22	mg/kg	29,46	28,99	1							19,2	19,2	1	43,1	44,3	1	30,3	29,5	1
Ba	R1	mg/kg							<0,1	<0,1	1				0,203	0,219	1			
	R21	mg/kg													0,0696	0,0718	1	0,046	0,034	1
	R22	mg/kg													0,146	0,148	1	0,065	0,045	1
Cd	R1	mg/kg	<0,02	<0,02	1				1,21	1,14	1	<0,02	<0,02	1	<0,01	<0,01	1			
	R21	mg/kg	<0,004	<0,004	1							<0,004	<0,004	1	<0,002	<0,002	1			
	R22	mg/kg	<0,02	<0,02	1							<0,016	<0,016	1	<0,01	<0,01	1			
conductivity	R1	mS/m	1,49	1,34	1	0,95	0,92	1	1,91	1,18	1	3,27	2,49	1	2,39	2,42	1			
	R21	mS/m	3,90	4,04	1							2,23	2,90	1	3,54	3,77	1	2,40	2,26	1
	R22	mS/m	0,870	0,870	1							1,35	1,58	1	2,10	2,33	1	1,28	1,11	1
Cr	R1	mg/kg	1,47	1,44	1	2,06	2,04	1	2,01	1,81	1				2,66	2,43	1			
	R21	mg/kg	0,588	0,552	1										0,889	0,867	1	0,721	0,741	1
	R22	mg/kg	1,56	1,50	1										2,40	2,65	1	1,517	1,368	1
Cu	R1	mg/kg	2,90	3,40	1				2,74	3,43	1	2,3	2,3	1	1,77	1,63	1			
	R21	mg/kg	1,42	1,21	1							0,4	0,42	1	0,503	0,504	1	1,181	1,161	1
	R22	mg/kg	3,00	3,18	1							1,28	1,28	1	1,60	1,76	1	3,818	3,558	1
DOC	R1	mg/kg							59,4	56,1	1				106	107	1			
	R21	mg/kg													48,4	45,9	1	36,03	36,03	1
	R22	mg/kg													102	105	1	84,91	77,74	1
Fe	R1	mg/kg	0,800	0,900	1	1,53	1,54	1	1,39	1,04	1				2,29	2,10	1			
	R21	mg/kg	0,168	0,192	1										0,689	0,685	1	0,480	0,581	1
	R22	mg/kg	1,91	0,818	1										2,05	2,44	1	1,132	0,684	1
Mo	R1	mg/kg	<0,10	<0,10	1	<0,01	<0,01	1	<1	<1	1	<0,01	<0,01	1	<0,1	<0,1	1			
	R21	mg/kg	<0,02	<0,02	1							<0,002	<0,002	1	<0,02	<0,02	1			
	R22	mg/kg	<0,10	<0,10	1							<0,008	<0,008	1	<0,1	<0,1	1			
Ni	R1	mg/kg	<0,10	<0,10	1	<0,01	<0,01	1	<0,1	<0,1	1	0,07	0,08	1	<0,1	<0,1	1			
	R21	mg/kg	<0,02	<0,02	1							0,008	0,008	1	<0,02	<0,02	1			
	R22	mg/kg	<0,10	<0,10	1							0,008	0,008	1	<0,10	<0,10	1			
Pb	R1	mg/kg	<0,40	<0,40	1				0,089	0,066	1	<0,01	0,01	1	0,160	0,146	1			
	R21	mg/kg	<0,08	<0,08	1							0,01	0,006	1	0,0414	0,0340	1	0,052	0,027	1
	R22	mg/kg	<0,40	<0,40	1							<0,008	0,016	1	0,119	0,112	1	0,022	0,030	1
pH	R1		5,41	5,44	1	5,80	5,79	1	4,70	5,24	1	6,02	6,1	1	7,2	7,5	1			
	R21		5,35	5,46	1							6,22	6,27	1	7,1	7,3	1	6,6	6,6	1
	R22		5,45	5,49	1							6,08	6,11	1	7,3	7,1	1	6,4	6,0	1
Sb	R1	mg/kg	1,04	1,04	1							0,9	0,8	1	1,07	1,05	1			
	R21	mg/kg	0,366	0,368	1							0,32	0,34	1	0,390	0,379	1	0,380	0,360	1
	R22	mg/kg	0,994	0,998	1							0,48	0,48	1	1,26	1,24	1	0,949	0,939	1
Se	R1	mg/kg	<0,02	<0,02	1										<0,2	<0,2	1			
	R21	mg/kg	<0,004	<0,004	1										<0,04	<0,04	1			
	R22	mg/kg	<0,02	<0,02	1										<0,20	<0,20	1			
Zn	R1	mg/kg	1,67	2,10	1	0,20	0,27	1	<0,1	<0,1	1	0,3	0,2	1	0,136	0,176	1			
	R21	mg/kg	1,62	0,204	1							0,08	0,08	1	0,0490	0,0656	1	0,072	0,088	1
	R22	mg/kg	2,25	0,995	1							0,16	0,16	1	0,159	0,153	1	0,315	0,239	1

LIITE 10. SUMMARY OF THE Z SCORES
Appendix 10.

Analyte	Sample\Lab	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	%
As	R1	A	A	A	A	.	A	**N**	A	A	.	A	A	A	A	**N**	A	A	.	87
	R21	A	A	A	A	A	A	n	.	A	A	A	A	A	.	.	A	A	A	93
	R22	A	A	A	A	A	A	**N**	.	A	A	A	A	A	.	.	n	**P**	A	80
Ba	R1	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R21	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R22	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
Cd	R1	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R21	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R22	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
conductivity	R1	A	A	A	A	.	A	**P**	A	.	.	A	A	p	A	**P**	**P**	**P**	.	64
	R21	A	A	A	A	A	A	p	.	A	A	A	A	**P**	.	.	A	**P**	A	80
	R22	A	n	A	A	**P**	.	**P**	.	.	n	A	A	A	.	.	A	**P**	A	62
Cr	R1	A	A	A	A	.	A	n	A	A	.	A	A	A	A	A	.	**P**	.	86
	R21	A	**P**	A	A	A	A	A	.	A	p	A	A	A	.	.	.	A	A	86
	R22	A	p	A	A	A	A	A	.	A	p	A	A	A	.	.	.	**P**	A	79
Cu	R1	A	A	A	A	.	A	**P**	A	.	.	A	n	A	.	A	A	n	.	77
	R21	**P**	p	A	A	**N**	A	A	.	.	A	n	A	A	.	.	**N**	n	A	57
	R22	p	p	A	A	n	A	**P**	.	.	A	n	A	A	.	.	n	A	A	57
DOC	R1	A	A	A	.	.	A	A	A	A	.	A	A	.	.	p	.	**P**	.	82
	R21	A	A	A	.	.	A	A	.	A	.	A	A	.	.	.	.	**P**	p	80
	R22	n	A	A	.	.	A	A	.	A	.	A	A	.	.	.	.	**P**	**P**	70
Fe	R1	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R21	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R22	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
Mo	R1	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R21	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R22	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
Ni	R1	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R21	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R22	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
Pb	R1	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R21	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R22	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
pH	R1	A	A	A	A	.	A	A	A	A	.	A	p	A	A	**N**	A	**P**	.	80
	R21	A	A	A	A	A	A	n	.	A	A	A	A	**N**	.	.	A	**P**	p	73
	R22	A	A	A	A	A	.	n	.	n	A	A	A	**N**	.	.	A	**P**	A	71
Sb	R1	A	A	A	A	.	A	n	A	A	.	A	A	A	.	.	A	A	.	92
	R21	A	A	A	A	A	A	n	.	A	A	A	A	A	.	.	A	A	A	93
	R22	n	A	A	A	A	A	n	.	A	A	A	A	A	.	.	**N**	A	A	80
Se	R1	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R21	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R22	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
Zn	R1	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R21	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
	R22	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	.	
%		81	76	100	100	75	100	33	100	94	75	90	90	78	100	33	67	33	79	
Accredited		yes		yes						yes			yes					yes	yes	

A - accepted ($-2 \leq Z \leq 2$), p - questionable ($2 < Z \leq 3$), n - questionable ($-3 \leq Z < -2$), P - non-accepted ($Z > 3$), N - non-accepted ($Z < -3$),

%* - percentage of accepted results

Totally accepted, % In all: 78

Documentation page

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Author(s)	Irma Mäkinen, Kati Vaajasaari, Olli Järvinen, Raija Ivalo, Keijo Tervonen and Markku Ilmakunnas	
Title of publication	SYKE Proficiency Test 4/2008 Leaching testing of a solid waste – the one stage and the two stage batch leaching test	
Parts of publication/ other project publications	The publication is available on the internet: www.ymparisto.fi/julkaisut	
Abstract	In spring 2008 the Finnish Environment Institute (SYKE) carried out the proficiency test for leaching testing of a contaminated soil sample with the one stage and the two stage batch leaching tests. In total, 18 laboratories participated in the proficiency test. The test material was a soil sample contaminated by wood preservatives (CCA), in which the main pollutants were arsenic, chromium and copper. The proficiency test was performed for the one stage batch leaching test (L/S 10, EN 12457-2) and the two stage batch leaching test (L/S2 and L/S10, EN 12457-3). From the leaching test eluates the inorganic substances (As, Ba, Cd, Cr, Cu, Mo, Ni, Pb, Sb, Se and Zn) and DOC, pH and conductivity were determined. The evaluation of the laboratory performance was carried out using z score. In this proficiency test 78 % of the results were satisfactory, when the total target deviation varied from 25 % to 40 % at the 95 % confidence interval. The SYKE proficiency test for leaching tests was carried out the second time. The test material texture was homogenous and consequently the results of the proficiency test can be regarded to be satisfactory.	
Keywords	leaching test, batch leaching test, characterization of waste, waste landfill acceptance criteria, environmental laboratories, proficiency test, interlaboratory comparisons	
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Tekijä(t)	Irma Mäkinen, Kati Vaajasaari, Olli Järvinen, Timo Sara-Aho, Raija Ivalo, Keijo Tervonen ja Markku Ilmakunnas	
Julkaisun nimi	SYKE Pätevyyskoe 4/2008 Liukoisuustestit kiinteille jätteille – 1-vaiheinen ja 2-vaiheinen ravistelutesti	
Julkaisun osat/ muut saman projektin tuottamat julkaisut	Julkaisu on myös saatavana internetissä: www.ymparisto.fi/julkaisut	
Tiivistelmä	SYKE järjesti pätevyyskokeen jätteen kaatopaikkakelpoisuuden arvioimiseen käytettävistä liukoisuustesteistä keväällä 2008. Pätevyyskokeeseen osallistui yhteensä 18 laboratoriota. Testausnäytteeksi valittiin suolakyllästeellä (CCA) pilaantunut maa. Pääasialliset haitalliset yhdisteet maassa olivat arseeni, kupari ja kromi. Osallistujat käyttivät liukoisuustestimenetelminä 1-vaiheista ravistelutestiä (L/S 10, EN 12457-2) ja 2-vaiheista ravistelutestiä (L/S2 ja L/S10, EN 12457-3). Ravistelutestiuutteista määritettiin epäorgaanisten aineiden (As, Ba, Cd, Cr, Cu, Mo, Ni, Pb, Sb ja Zn) sekä orgaanisen hiilen (DOC) liuenneet määrät sekä uutteiden pH ja sähkönjohtavuus. Sekä 1-vaiheisessa että 2-vaiheisessa testissä toistettavuus ja uusittavuus olivat pääasiallisesti pienempiä kuin standardiohjeissa esitetyt rajat. Poikkeavuuksia esiintyi 2-vaiheisen testin toistettavuudessa ja uusittavuudessa. Laboratorioiden pätevyyden arviointi tehtiin z-arvon avulla ***(z score).*** Tässä vertailussa tulosaineistossa oli tyydyttäviä tuloksia 78 %, kun vertailuarvosta (***the assigned value***) sallittiin 25 % – 40 % poikkeama 95 % merkitsevyystasolla. Pätevyyskokeen tuloksia voidaan pitää tyydyttävänä.	
Asiasanat	Liukoisuustesti, ravistelutesti, jätteen karakterisointi, kaatopaikkakelpoisuus, ympäristölaboratoriot, pätevyyskoe, laboratorioiden välinen vertailukoe	
Julkaisusarjan nimi ja numero	Suomen ympäristökeskuksen raportteja 32/2008	
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Projektihankkeen nimi ja projektinumero		
Rahoittaja/ toimeksiantaja		
Projektiryhmään kuuluvat organisaatiot		
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Julkaisun kustantaja	Suomen ympäristökeskus, PL 140, 00251 Helsinki	
Painopaikka ja -aika	Helsinki 2008	
Muut tiedot		

Presentationsblad

Utgivare	Finlands Miljöcentral (SYKE)	Datum November 2008
Författare	Irma Mäkinen, Kati Vaajasaari, Olli Järvinen, Timo Sara-Aho, Raija Ivalo, Keijo Tervonen och Markku Ilmakunnas	
Publikationens titel	SYKE Provningsjämförelse 4/2008 Laktest för klassificering av avfall till deponi - ett steg och två steg skaktestet	
Publikationens delar/ andra publikationer inom samma projekt	Publicationen finns tillgänglig på internet: www.ymparisto.fi/julkaisut	
Sammandrag	Under våren 2008 genomförde Finlands Miljöcentral en provningsjämförelse, som omfattade bestämningen av utlagning av jord föorenad huvudsakligen med Cu, Cr oach As (CCA). I provningsjämförelsen deltog 18 laboratorier. Labratorierna använde ett steg och två steg skaktestet vid provningsjämförelsen. Från ekstrakter bestämdes metaller (As, Ba, Cd, Cr, Cu, Mo, Ni, Pb, Sb, Se och Zn), DOC, F, pH-värdet och konduktivitet. Resultaten värderades med hjälp av z-värden. I provningsjämförelsen var 78 % av alla resultaten tillfredsställande, när 24–40 % totalavvikelsen från referensvärdet accepterades på 95 % konfidens interval. Provet var finfördelat. Resultat av denna provningsjämförelse var tillfredställande.	
Nyckelord	laktest, katrakterisering av utfall, klassificering av avfall till deponi, provningsjämfö-relse, miljölaboratorier	
Publikationsserie och nummer	Suomen ympäristökeskuksen raportteja 32/2008	
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