Results of Proficiency Test
Chromium (VI) in Leather
May 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Chromium (VI) is a toxic and mutagenic substance. In the leather industry, Chromium containing substances could be used in the production process. Of all Chromium compounds, primarily Chromium (VI) was used, but this has been replaced by the less hazardous Chromium (III) in most applications. The regulations for the presence of Chromium (VI) for leather continue to become stricter. But even if no Chromium (VI) is used in the production of leather, it can still be formed from Chromium (III), when production or end-use circumstances are not controlled.

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the determination of Chromium (VI) in Leather every year. During the annual proficiency testing program 2018/2019, it was decided to continue the proficiency test for the analysis of Chromium (VI) in Leather.

In this interlaboratory study 150 laboratories in 34 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2019 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one aged leather sample of 5 grams, positive on Chromium (VI), labelled #19540.
The participants were asked to report the rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

A batch of off-white colored leather was obtained from the local market. The leather material was grinded and aged. After thoroughly mixing, 200 plastic bags were filled with approximately 5 grams of leather, vacuumed and labelled #19540. The homogeneity of the subsamples was checked by determination of Chromium (VI) in accordance with ISO17075-1 on 8 stratified randomly selected samples.

	Chromium (VI) in mg/kg
Sample #19540-1	20.20
Sample #19540-2	19.08
Sample #19540-3	19.91
Sample #19540-4	19.85
Sample #19540-5	19.23
Sample #19540-6	19.56
Sample #19540-7	19.42
Sample #19540-8	17.50

Table 1: homogeneity test results of subsamples #19540

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2, in the next table.

	Chromium (VI) in mg/kg
r (observed)	2.33
reference test method	ISO17075-1:17
0.3 * R (reference test method)	2.37

Table 2: evaluation of the repeatability of subsamples #19540

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample labelled #19540 was sent on April 17, 2019.

2.5 ANALYSES

The participants were requested to determine the content of Chromium (VI) on a leather sample, applying the analysis procedure that is routinely used in the laboratory.
It was explicitly requested to treat the sample as if it was a routine sample, but not to age nor to dry the sample nor to determine volatile matter. The amount of sample was not sufficient to allow aging and/or determine the volatile matter content. Also, it was asked to keep the sample stored dark, dry and cool (4 – 10°C) and keep sample vacuum packed until the start of extraction.

It was requested to report if the laboratory was accredited for the determination of Chromium (VI) and to report some analytical details. It was also requested to report the test results using the indicated units on the report form and not to round the results, but report as much significant figures as possible and not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no re-analyzes). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…" were in general not used in the statistical evaluation.
First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO 5725, the original test results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficienct test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In general, when no literature reproducibility is available, another target may be used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test results is fit-for-use.

The z-scores were calculated according to:

$z_{(target)}$ = (test result - average of PT) / target standard deviation

The $z_{(target)}$ scores are listed in the result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. The usual interpretation of z-scores is as follows:

$|z| < 1$ good
$1 < |z| < 2$ satisfactory
$2 < |z| < 3$ questionable
$3 < |z|$ unsatisfactory

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. Two participants did not report any test results, all other participants reported before the deadline. Not all laboratories report both analyses requested.
In total, 148 participants reported 192 numerical results. Observed were 7 outlying test results, which is 3.6% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 4.

Chromium (VI) (colorimetric): This determination was not problematic at a concentration level of 18.9 mg/kg. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with ISO17075-1:17.

Chromium (VI) (chromatographic): This determination was not problematic at a concentration level of 19.5 mg/kg. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with ISO17075-2:17.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(target)
Chromium (VI) (colorimetric)	mg/kg	134	18.92	5.57	7.75
Chromium (VI) (chromatographic)	mg/kg	51	19.50	3.26	5.38

Table 3: reproducibilities of tests on sample #19540

Without further statistical calculations, it could be concluded that there is a good compliance of the group of participating laboratories with the relevant reference test methods.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2019 WITH PREVIOUS PTS

	May 2019	April 2018	April 2017	April 2016	February 2015
Number of reporting labs	148	162	163	145	141
Number of test results	192	190	183	144	153
Number of statistical outliers	7	2	5	6	5
Percentage outliers	3.6%	1.1%	2.7%	4.2%	3.3%

Table 4: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	May 2019	April 2018	April 2017	April 2016	February 2015	Target
Chromium (VI) (colorimetric)	11%	31%	15%	29%	33%	15%
Chromium (VI) (chromatographic)	6%	31%	10%	n.e.	n.e.	10%

Table 5: development of the uncertainties over the years

The relative standard deviations observed in this PT are smaller than the relative standard deviations observed in previous PTs.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The test methods ISO17075-1 and ISO17075-2 are used by almost all of the reporting participants for the determination of Chromium (VI) with colorimetric and chromatographic method respectively.
For this PT also some analytical details were requested, see appendix 2. Based on the answers given by the participants the following can be summarized:

- About 85% of the reporting participants mentioned that they are accredited for the determination of Chromium (VI) in Leather.
- About all of the reporting participants used a test portion between 1 and 2 grams.
- Almost all of the reporting participants used an extraction time of 180 minutes
- Almost all of the reporting participants used an extraction temperature between 20°C and 25°C, ambient or room temperature.
- The pH before and after extraction was between pH 7 and pH 8 and thus in accordance with the test methods ISO17075-1:2017 and ISO17075-2:2017.
- Also the type and frequency of the shaker were requested to report. Several types of shakers were mentioned. For example “horizontal”, “orbital” or “mechanical”.
  About 80% of the reporting participants used a frequency of 100 ±10 rpm.

5 DISCUSSION

As Chromium (VI) is carcinogenic, mutagenic and toxic for reproduction, the regulations within countries tend to adopt a zero-tolerance policy. In actual practise this will mean below the detection limit of the widely accepted test method ISO17075:2017 (parts 1 and 2). Examples of regulations can be found in table 6.

Chromium (VI)	Limit	Comment
Germany: SG (Schadestoff gepruft) – label	< 3 mg/kg	As well for aging as non-aging
EU: REGULATION No 301/2014 amending Annex XVII to Regulation (EC) No 1907/2006 of the (REACH)	< 3 mg/kg	Implementation: 01-05-2014 Reported only as dry-weight

Table 6: Regulation on Chromium (VI)

When the results of this interlaboratory study were compared to this limit, it may be noticed that all participants would make identical decisions about the acceptability of the leather. When using a limit of <3 mg/kg and applying it to the reported test results for sample #19540 all participants would not release this sample to the consumer market.

6 CONCLUSION

It can be concluded that the group of participants have no problems with the determination of Chromium (VI) colorimetric and chromatographic in this PT. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

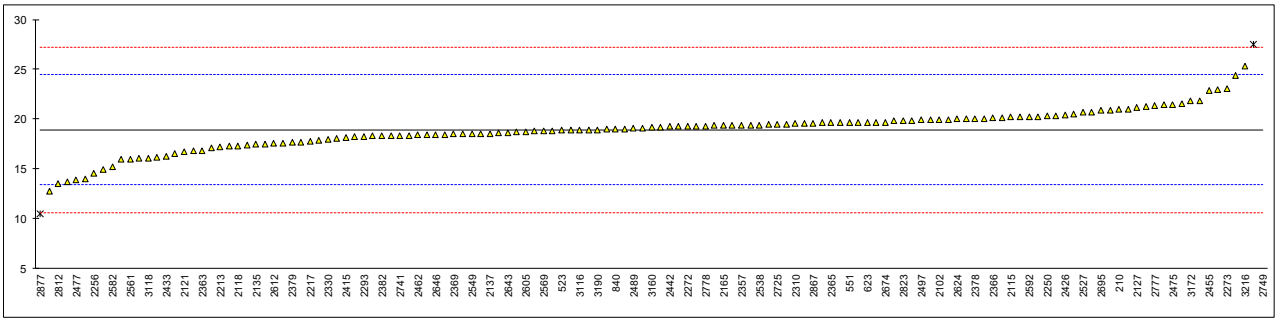
Determination of Chromium (VI) (colorimetric) in sample #19540; results in mg/kg

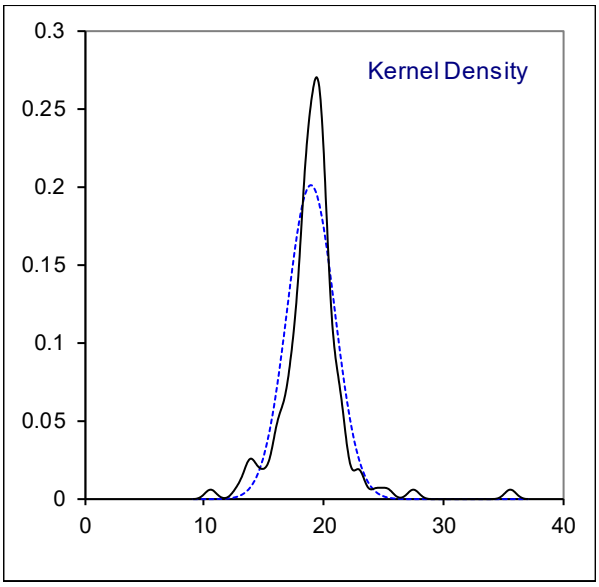
lab	method	value	mark	z(targ)	remarks
210	ISO17075-1	21		0.75	
230	ISO17075-1	21.504		0.93	
339	ISO4684/ISO17075Mod.	17.7		-0.44	
348	ISO17075-1	20.02		0.40	
362	ISO17075-1	19.422		0.18	
523	ISO17075-1	18.86		-0.02	
551	ISO17075-1	19.682	C	0.27	first reported 9.682
623	ISO17075-1	19.70		0.28	
840	ISO17075-1	18.96		0.01	
2102	ISO17075-1	19.95		0.37	
2115	ISO17075-1	20.18		0.45	
2118	ISO17075-1	17.3		-0.59	
2121	ISO17075-1	16.72		-0.80	
2127	ISO17075-1	21.2		0.82	
2129	ISO17075-1	17.3		-0.59	
2132	ISO17075-1	19.65		0.26	
2135	ISO17075-1	17.44		-0.54	
2137	ISO17075-1	18.549		-0.14	
2139	ISO17075-1	17.897		-0.37	
2146	ISO17075-1	16.037		-1.04	
2165	ISO17075-1	19.39		0.17	
2166	ISO17075-1	19.49		0.20	
2213	ISO17075-1	17.2		-0.62	
2217	ISO17075-1	17.805		-0.40	
2230	ISO17075-1	20.5	C	0.57	first reported 8.22
2241	ISO17075-1	16.56		-0.85	
2247	ISO17075-1	18.90		-0.01	
2250	ISO17075-1	20.3		0.50	
2255	ISO17075-1	18.3		-0.23	
2256	ISO17075-1	14.56		-1.58	
2266	ISO17075-1	18.8		-0.04	
2272	ISO17075-1	19.28		0.13	
2273	ISO17075-1	23.02		1.48	
2284	ISO17075-1	21.0		0.75	
2289	ISO17075-1	18.22		-0.25	
2290	ISO17075-1	18.6		-0.12	
2293	In house	18.252		-0.24	
2295	ISO17075-1	19.21		0.10	
2301	ISO17075-1	14.0		-1.78	
2310	ISO17075-1	19.52		0.22	
2311	ISO17075-1	21.8		1.04	
2330	ISO17075-1	17.97		-0.34	
2347	ISO17075-1	19.54		0.22	
2350	ISO17075-1	18.983		0.02	
2352	ISO17075-1	20.13		0.44	
2357	ISO17075-1	19.4		0.17	
2358	ISO17075-1	18.96		0.01	
2363	ISO17075-1	16.87		-0.74	
2365	ISO17075-1	19.64		0.26	
2366	ISO17075-1	20.11		0.43	
2369	ISO17075-1	18.48		-0.16	
2370	ISO17075-1	20.2		0.46	
2375	ISO17075-1	20		0.39	
2378	ISO17075-1	20.01		0.39	
2379	ISO17075-1	17.65		-0.46	
2380	ISO17075-1	19.62		0.25	
2382	ISO17075-1	18.3		-0.23	
2385	ISO17075-1	18.7		-0.08	
2390	ISO17075-1	19.37		0.16	
2410	ISO17075-1	19.7		0.28	
2415	ISO17075-1	18.15		-0.28	
2426	ISO17075-1	20.4		0.53	
2433	ISO17075-1	16.30		-0.95	
2442	ISO17075-1	19.24		0.11	
2449	ISO17075-1	19.931	C	0.36	first reported 8.372
2452		-----		-----	
2455	ISO17075-1	22.9		1.44	
2459	ISO17075-1	16.208		-0.98	
2460	ISO17075-1	15.96		-1.07	
2462	ISO17075-1	18.4		-0.19	
2475	ISO17075-1	21.48		0.92	
2477	ISO17075-1	13.901		-1.81	
2489	ISO17075-1	19.06		0.05	
2495		-----		-----	
2497	ISO17075-1	19.92		0.36	

lab	method	value	mark	z(targ)	remarks
2501	ISO17075-1	17.61		-0.47	
2504	ISO17075-1	19.698		0.28	
2511	ISO17075-1	18.406		-0.19	
2527	ISO17075-1	20.7		0.64	
2532	ISO17075-1	18.4		-0.19	
2538	ISO17075-1	19.42		0.18	
2549	ISO17075-1	18.5		-0.15	
2553	ISO17075-1	20.32	C	0.50	first reported 8.32
2560	ISO17075-1	17.504		-0.51	
2561	ISO17075-1	15.99		-1.06	
2563	ISO17075-1	18.3		-0.23	
2569	ISO17075-1	18.8		-0.04	
2582	ISO17075-1	15.26		-1.32	
2590	ISO17075-1	19.394		0.17	
2592	ISO17075-1	20.2		0.46	
2602	In house	17.4		-0.55	
2605	ISO17075-1	18.70		-0.08	
2610		-----		-----	
2612	ISO17075-1	17.55		-0.50	
2619	ISO17075-1	19.276		0.13	
2624	ISO17075-1	19.996		0.39	
2629	ISO17075-1	24.38		1.97	
2643	ISO17075-1	18.66		-0.09	
2646	ISO17075-1	18.403		-0.19	
2656		-----		-----	
2668	ISO17075-1	18.86		-0.02	
2674	ISO17075-1	19.7		0.28	
2695	ISO17075-1	20.882		0.71	
2705	ISO17075-1	27.53	R(0.01)	3.11	
2711		-----		-----	
2725	ISO17075-1	19.4806		0.20	
2730		-----		-----	
2734		-----		-----	
2741	ISO17075-1	18.3		-0.23	
2749	In house	35.68	R(0.01)	6.05	
2756		-----		-----	
2758		-----		-----	
2777	GB/T22807	21.329		0.87	
2778	GB/T22807	19.314		0.14	
2783	ISO17075-1	12.7980439969191		-2.21	
2789		-----		-----	
2791	ISO17075-1	19.12		0.07	
2804	ISO17075-1	18.38		-0.20	
2806	ISO17075-1	19.8		0.32	
2812	ISO17075-1	13.56		-1.94	
2823	ISO17075-1	19.808		0.32	
2829	ISO17075-1	20.229		0.47	
2830	ISO17075-1	21.31		0.86	
2836	ISO17075-1	20.728		0.65	
2844	ISO17075-1	19.41		0.18	
2862	§64 LFGB 82.02-11	19.95		0.37	
2866		-----		-----	
2867	ISO17075-1	19.54		0.22	
2877	ISO17075-1	10.4958	R(0.01)	-3.04	
2891	ISO17075-1	14.94		-1.44	
3100	ISO17075-1	18.025		-0.32	
3116	ISO17075-1	18.87		-0.02	
3118	ISO17075-1	16.11		-1.02	
3146	ISO17075-1	19.83		0.33	
3149	§64 LFGB 82.02-11	20.9		0.71	
3150	ISO17075-1	17.068		-0.67	
3154	ISO17075-1	18.48		-0.16	
3160	ISO17075-1	19.195		0.10	
3172	ISO17075-1	21.79		1.04	
3190	ISO17075-1	18.92		0.00	
3192		-----		-----	
3197	ISO17075-1	18.8		-0.04	
3201	In house	22.95		1.46	
3209	ISO17075-1	18.53		-0.14	
3210		-----		-----	
3216	ISO17075-1	25.35		2.32	
3228	ISO17075-1	19.3		0.14	
3233	ISO17075-1	13.73		-1.88	
3237	ISO17075-1	21.41		0.90	
3248	ISO17075-1	16.826		-0.76	

normality	suspect
n	134
outliers	3
mean (n)	18.923
st.dev. (n)	1.9881
R(calc.)	5.567
st.dev.(ISO17075-1:17)	2.7678
R(ISO17075-1:17)	7.750

RSD = 10.5%



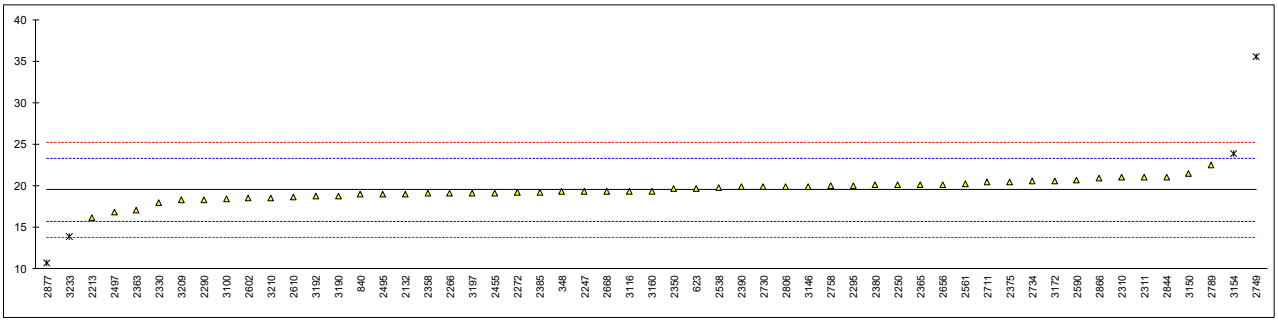


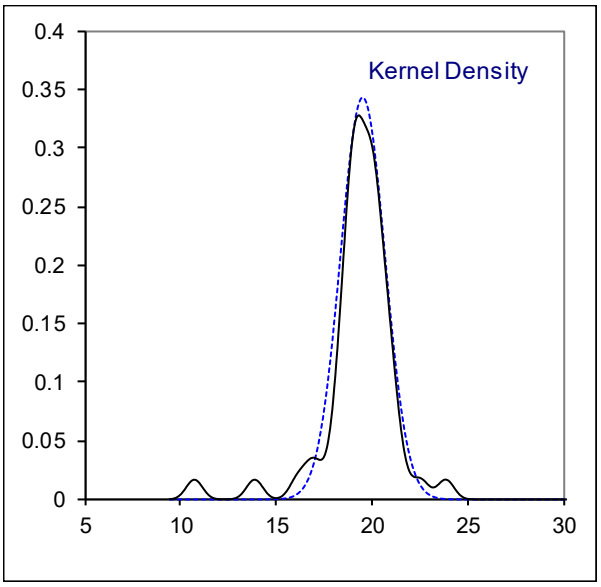
Determination of Chromium (VI) (chromatographic) in sample #19540; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
230		-----		-----	
339		-----		-----	
348	ISO17075-2	19.26		-0.12	
362		-----		-----	
523		-----		-----	
551		-----		-----	
623	ISO17075-2	19.69		0.10	
840	ISO17075-2	18.93		-0.30	
2102		-----		-----	
2115		-----		-----	
2118		-----		-----	
2121		-----		-----	
2127		-----		-----	
2129		-----		-----	
2132	ISO17075-2	18.98		-0.27	
2135		-----		-----	
2137		-----		-----	
2139		-----		-----	
2146		-----		-----	
2165		-----		-----	
2166		-----		-----	
2213	ISO17075-2	16.1		-1.77	
2217		-----		-----	
2230		-----		-----	
2241		-----		-----	
2247	ISO17075-2	19.33		-0.09	
2250	ISO17075-2	20.1		0.31	
2255		-----		-----	
2256		-----		-----	
2266	ISO17075-2	19.1		-0.21	
2272	ISO17075-2	19.16		-0.18	
2273		-----		-----	
2284		NA		-----	
2289		-----		-----	
2290	ISO17075-2	18.3		-0.62	
2293		-----		-----	
2295	ISO17075-2	20.0		0.26	
2301		-----		-----	
2310	ISO17075-2	21		0.78	
2311	ISO17075-2	21		0.78	
2330	ISO17075-2	17.91		-0.83	
2347		-----		-----	
2350	ISO17075-2	19.64		0.07	
2352		-----		-----	
2357		-----		-----	
2358	ISO17075-2	19.09		-0.21	
2363	ISO17075-2	17.10		-1.25	
2365	ISO17075-2	20.15		0.34	
2366		-----		-----	
2369		-----		-----	
2370		-----		-----	
2375	ISO17075-2	20.5		0.52	
2378		-----		-----	
2379		-----		-----	
2380	ISO17075-2	20.08		0.30	
2382		-----		-----	
2385	ISO17075-2	19.2		-0.15	
2390	ISO17075-2	19.86		0.19	
2410		-----		-----	
2415		-----		-----	
2426		-----		-----	
2433		-----		-----	
2442		-----		-----	
2449		-----		-----	
2452		-----		-----	
2455	ISO17075-2	19.101		-0.21	
2459		-----		-----	
2460		-----		-----	
2462		-----		-----	
2475		-----		-----	
2477		-----		-----	
2489		-----		-----	
2495	ISO17075-2	18.934		-0.29	
2497	ISO17075-2	16.78		-1.42	

lab	method	value	mark	z(targ)	remarks
2501		-----		-----	
2504		-----		-----	
2511		-----		-----	
2527		-----		-----	
2532		-----		-----	
2538	ISO17075-2	19.77		0.14	
2549		-----		-----	
2553		-----		-----	
2560		-----		-----	
2561	ISO17075-2	20.23		0.38	
2563		-----		-----	
2569		-----		-----	
2582		-----		-----	
2590	ISO17075-2	20.710		0.63	
2592		-----		-----	
2602	ISO17075-2	18.5		-0.52	
2605		-----		-----	
2610	ISO17075-2	18.68		-0.43	
2612		-----		-----	
2619		-----		-----	
2624		-----		-----	
2629		-----		-----	
2643		-----		-----	
2646		-----		-----	
2656	ISO17075-2	20.16		0.34	
2668	ISO17075-2	19.33		-0.09	
2674		-----		-----	
2695		-----		-----	
2705		-----		-----	
2711	ISO17075-2	20.47		0.51	
2725		-----		-----	
2730	ISO17075-2	19.86		0.19	
2734	ISO17075-2	20.513		0.53	
2741		-----		-----	
2749	ISO17075-2	35.54	R(0.01)	8.35	
2756		-----		-----	
2758	ISO17075-2	19.96		0.24	
2777		-----		-----	
2778		-----		-----	
2783		-----		-----	
2789	ISO17075-2	22.51		1.57	
2791		-----		-----	
2804		-----		-----	
2806	ISO17075-2	19.9		0.21	
2812		-----		-----	
2823		-----		-----	
2829		-----		-----	
2830		-----		-----	
2836		-----		-----	
2844	ISO17075-2	21.06		0.81	
2862		-----		-----	
2866	ISO17075-2	20.9		0.73	
2867		-----		-----	
2877	ISO17075-2	10.6970	R(0.01)	-4.58	
2891		-----		-----	
3100	ISO17075-2	18.415		-0.56	
3116	ISO17075-2	19.33		-0.09	
3118		-----		-----	
3146	ISO17075-2	19.92		0.22	
3149		-----		-----	
3150	ISO17075-2	21.514		1.05	
3154	ISO17075-2	23.84	R(0.05)	2.26	
3160	ISO17075-2	19.37		-0.07	
3172	ISO17075-2	20.55		0.55	
3190	ISO17075-2	18.80		-0.36	
3192	ISO17075-2	18.74		-0.39	
3197	ISO17075-2	19.1		-0.21	
3201		-----		-----	
3209	ISO17075-2	18.26		-0.64	
3210	In house	18.533		-0.50	
3216		-----		-----	
3228		-----		-----	
3233	ISO17075-2	13.85	C,R(0.01)	-2.94	first reported 14.25
3237		-----		-----	
3248		-----		-----	

normality	suspect	
n	51	
outliers	4	
mean (n)	19.498	
st.dev. (n)	1.1647	RSD = 6.0%
R(calc.)	3.261	
st.dev.(ISO17075-2:17)	1.9203	
R(ISO17075-2:17)	5.377	





APPENDIX 2

Summary of reported analytical details

lab	ISO/IEC17025 accredited	sample intake (g)	time between grinding/cutting and extraction	extraction time (min)	extraction temp (°C)
210	No				
230	Yes	1g			
339	No	2	-	180	Room Temp
348	No	2	10-30 min	180	20ºC
362	Yes	2.0014	The sample was received grinded/cutted. The extraction was started immediately after the sample was opened.	180 min	20.8
523	No	1.0 g	5 min	180 min	25 °C
551	Yes	1.0029g		180 min.	20°C.
623	Yes	1 g	10	180	25
840	Yes	1.0000 g	NA	180 mins	25oC
2102	Yes	2 gram	Grinding/cutting is not performed	180	20
2115	Yes	1 g	0	180 min	25 °C
2118	Yes	1.996 g	The sample was used for extraction as received (0 min).	180 minutes	Room temp (24°C)
2121	Yes	2.036 g and 1.0172 g	the sample was already grinded	3h00	Ambient temp
2127	Yes	0,95	the sample was already shredded	3 hours	room temp
2129	No				
2132	Yes	2 grams	5 mins.	180 mins.	25°C
2135	Yes	2.031		180 minutes	20 °C
2137	Yes	1 g		180 minutes	23.8
2139	Yes	1g	About 10 minutes	180 minutes	21 °C
2146	No	2 g		3h +/- 5 min	ambient, about 20 dgs
2165	Yes	1	60	180	22
2166	Yes	2,00	--	180	20
2213	Yes	2.0052		240 minutes	23
2217	Yes	around 2,0 g			
2230	Yes	1g	Received the sample and extracted immediatly.	180 min	Room Temp
2241	Yes	1	0	180	23
2247	Yes: ISO17075-1	2.0089(gm)	Sample has been tested as received, no grinding/cutting was done.	180min shaking total process time: 360min	room temp
2250	Yes	2 g	10 min.	180 min.	20°C
2255	Yes	0.9398	25	180	22-23
2256	Yes	1	no further cut or grind received PT sample	180	25
2266	Yes	2g for 17075-1 0.5g for 17075-2	one hour	3 hours	20°C
2272	Yes	2gram	30min	180min	room temp, around 25
2273	Yes	2.008	N/A	180	21.0
2284	Yes	2.0007	Grinded sample received	180	Room temp
2289	Yes	2.0	No Grinding/cutting	180min	At room temp
2290	Yes				
2293	No	2.0049g	5	180	20
2295	Yes	2 grams	2 minutes	180 minutes	room temp
2301	Yes	1		180	room temp
2310	Yes	2.00g	Sample used as such	3hrs± 5mins	25°C
2311	Yes	1		180	22
2330	Yes	1 gram		180 minutes	27 °C
2347	Yes	1g			
2350	Yes	about 2.0 g	Cutting	180 mins	Room temp
2352	Yes	2g	10min	180min	23.2
2357	Yes	2g		180 mins	22.5
2358	Yes	0.8g	N/A	180 minutes	Room temp
2363	Yes	2g	30min	3h	22
2365	Yes	1g	no more grinding/cutting.	180min	18~28
2366	Yes	1g per 50 ml extraction solvent	NA	180 min.	25
2369	Yes	2g	/	180min	23
2370	Yes	2.0055 g	No grinding/cutting for this sample.	180 min	27
2375	Yes	1,0034 gr	-	180	Room Temp
2378	Yes	2g	10mins	180mins	25
2379	No	1.0052	180 minutes	180 minutes	24.2 dgs

lab	ISO/IEC17025 accredited	sample intake (g)	time between grinding/cutting and extraction	extraction time (min)	extraction temp (°C)
2380	Yes	1.00 g	5 minutes	180 minutes	22 °C
2382	Yes	1g	30min	180min	22
2385	Yes	1		180	20
2390	Yes	2.0097 gram	As received	180 minutes	22.4
2410	Yes	1 g		180 min	24
2415	Yes	2	N/A, not grinding/cutting	180	22
2426	Yes	01 gram	As revived	2 Hours	Room Temp
2433	Yes	1 g		180 min	room temp
2442	Yes	2.0085g	5 minutes	180 minutes	25
2449	Yes	1g	20	3hours	25
2452	---				
2455	Yes	17075-1: 2.0362 g 17075-2: 2.0208 g	zero minutes	180 minutes	24.1
2459	Yes	2.0	n/a	180 minutes	25 C
2460	Yes	2.0030	10 minutes	180 minutes	20
2462	Yes	2.0	10min	180min	22
2475	No	1.007g / 50ml		180	room temp
2477	Yes	2,0009 g	118 min	180 min	20 °C
2489	Yes	2.0 g	Used for testing as received.	180 minutes	25°C
2495	Yes	1	5	180	22
2497	Yes		60	180	24
2501	Yes	2.0028g	0	180 min	22℃
2504	Yes	2.00xx g	10 mins	180 mins	25.6 C
2511	---				
2527	No	0.5 g	5 minutes	180 minutes	Room temp, about 22 degrees
2532	Yes	1 gram	Sample Cutting - 5 mints ;	180 Minutes	Room Temp - 25 °C
2538	Yes	1,5	The sample was neither ground nor cut.	180	23
2549	Yes	2.0251	Not Applicable	180 minutes	25
2553	Yes	1.0	10 mins	180 mins	Room Temp 26
2560	Yes	2.000	NA	180 minutes	21
2561	Yes	2 g	N/A as sample was supplied pre-ground, therefore vacuum packed sample was opened, weighed and extraction solution added all within 10 minutes.	180	21
2563	Yes	1,61	10 minutes	180 min.	room temp
2569	Yes	2 gms	only weighing	180 Minutes	24°C
2582	Yes	2.00	-	180 min	room temp
2590	Yes	0.999 g	5 min	180 min	22.5 °C
2592	---				
2602	Yes	1,000 g	no time period, sample was grinded and made the extraction directly after opening the pouch	180 min	room temp about 21°C
2605	Yes	2 grams	No grinding/cutting the sample	180 minutes	26.5
2610	Yes	2.0121 g	The sample is received ground, so the extraction is done immediately after the weighing	180 min	23-25°C
2612	Yes	approx. 2 g	15 min	180 min +/- 5min	22,8 °C
2619	Yes	2,0062		180 min	20,08
2624	No	2,0 gr	20 min max	180 min	room temp
2629	Yes	2.0 gram	Used as sent sample, no further cutting	180 minutes	25
2643	Yes	1 g	extraction :180 minutes (3 hours)	180 minutes (3 hours)	room Temp (25)
2646	Yes	2	15 min	180 min	RT (19°C)
2656	No	2	30	180	20
2668	Yes	1.0g	Grinding sample received	180 min	Room temp
2674	Yes	1g	8 minutes	180 minutes	room temp (about 22)
2695	Yes	2,0 grams	10 minutes	180 minutes	20 °C
2705	No	3	no grinding, used as received	180	22
2711	No	1.033	30 min	180 min	25 °C
2725	No	m1:2,0067g m2:2,0083g	The simple received is grinded so there is no time.	180 min	22ºC
2730	No	2		180	room temp
2734	Yes	2	10 min	180	room
2741	Yes	2	/	180ms	23
2749	No	1g	15	180	23°C
2756	---				
2758	No	1 gram		180	room temp
2777	No	3.982	30 minutes	180 minutes	ambient

lab	ISO/IEC17025 accredited	sample intake (g)	time between grinding/cutting and extraction	extraction time (min)	extraction temp (°C)
2778	Yes	4g	20mins	3h	20-25
2783	Yes	2.0052	10	180	20
2789	Yes	2.0001g	10 minutes	3 hours	21ºC
2791	Yes	1		180	23
2804	No	2	NA	180	Room Temp.
2806	Yes				
2812	Yes	2 gr	15 min	3 hours	room temp
2823	Yes	2.0003g	N/A, samples were already ground when sent. Vacuum sealed sample was opened, weighed immediately and added straight to extraction solvent.	180 minutes.	22.5°C.
2829	Yes	2.0000	5 min	180 min	23±5 °C
2830	Yes	2.0186	Date of analysis: 21/05/2019	180 minutes	about 26 °C
2836	Yes	2,0 grams	The received sample was grinded.	180 minutes.	25°C.
2844	No	2.0000g +/- 0.2g screening 2g	1h 20 min	180 min +/- 5 min	22 ºC
2862	Yes	quantification 2x1g	sample was already grinded	3 hours	22°C
2866	Yes	2 g	material was already cut	180	22,5
2867	Yes	2.012 g	10 minutes	180 minutes	24
2877	No	2.0	NA	180 min	29°C
2891	Yes	4.01 g	1h15min	180 min	20°C
3100	Yes	2g	-	180mins	23.2℃
3116	Yes	1gram	less then 30 min	180 min	ambient temp (15 -28 °C)
3118	Yes	2.0065		180 minutes	25
3146	Yes	IC: 0.5072g Photometer: 1.0287g	Time between weigh and extract: 5-10min	180min	about 21°C
3149	Yes	1,8	directly	3 h	
3150	Yes	1		180	20
3154	Yes	2,000		180	23 °C
3160	Yes	1 g in 50 ml of extraction solution	we used the sample as it was received	180 minutes	25°C
3172	Yes	0.8		180	25
3190	Yes	1.0000	Less than 10mins.	180mins	25°C
3192	Yes	2	10	180	Room temp
3197	Yes	2,0001 g	<30 minutes	180 minutes	23C
3201	Yes	2,01 g	The sample was already ground/cut. The time period between intake and extraction was 10 minutes.	180 min	23 °C
3209	Yes	2g	Less than half an hour	180min	Room temp
3210	Yes	2.003		180	20.8
3216	Yes	aprox. 2grs		180 min	Room Temp
3228	Yes	2.0g	10min	180min	22
3233	No	1.0375g	5 min	180 min	20.4°C
3237	Yes	2 g		180 min	Room temp
3248	Yes	2.0043	60	180	20

Summary of reported analytical details – continued -

lab	pH before extraction	pH after extraction	type shaker used	frequency shaker	remarks
210					
230					
339	8.0	8.0	Shaking table	230 rpm	
348	7.97	7.75 chromatographic 7.62 colorimetric	Orbital	100 rpm	
362	8.0	7.7	Orbital shaker	100 RPM	
523	7.98	7.96	Orbital shaker	100 rpm	
551	8.002	7.721	Orbital shaker	100hz	
623	7.4	7.4	Mechanical Shaker	100 min-1	
840	8.00	7.67	orital shaker	100 rpm	
2102	8.0	8.0	Orbital shaker		
2115	8.0 pH	7.72 pH	platform shaker	120 RPM	
2118	8.01	7.71	WiggenHauser OS-150 shaker	100 t/min	
2121		7.6	horizontal shaker		
2127			Edmund Bühler EB SM-32	150/min	
2129					
2132	8.0	8.0	GFL Orbital Shaker	100 rpm	
2135	8.0	7.8		80/min.	
2137	7.95	7.57			
2139	pH 8.0	pH 7.5	Orbital shaker	100 min-1.	
2146		7,5-8,0	laboratory platform shaker	150 rpm	
2165	8.0	7.6	reciprocating	100 r/min	
2166	8,00	7,63	horizontal shaker	180/Min.	
2213	8.02	7.71	Orbital shaker	100	
2217					
2230	7.95	7.80	Mechanical shaker (Horizontal)	100 +/- 10min-1	
2241	8.0	7.6	Excella E2, open air shaker	100	
2247	8.01	7.2	Orbital Shaker	100 (rpm)	
2250	8	8	Heidolph Promax 2020	117 U/min	
2255	8	7.9	Orbital	100 rpm	
2256	7.7	7.7	horizontal mechanical shaker	100 rpm	
2266	6.5	7.5	orbital mechanical stirrer	100 tour / min	
2272	8.0pH	7.55pH	orbital shaker	100rpm	
2273	8.0	7.60	Back and forward	100 rpm	
2284	8.02	7.76	Orbital shaker	100r/min	
2289	8.0	7.6	orbital shaker	100	
2290					
2293	7.95	7.57	Horizontal	100	
2295	8.0	8.0	horizantal shaker	100 rpm	
2301	8	7.9	mechanical	250 rpm	
2310	8.0pH	7.7pH	Orbital motion	110 RPM	
2311	8	7.9	MECHNICAL SHAKER	100RPM	
2330	8.001	7.561	Horizontal shaker	100 rpm	
2347					
2350	pH is 7.3	pH is 7.3	Orbital shaker	100/min	
2352	8.01	7.85	…	100times/min	
2357	7.8	7.8	Horizontal oscillation	100 /min	
2358		Between 7.0 to 8.0	Orbital shaker	100 per minute	
2363	pH=8	pH=7.8	orbital shaker	100r/min	
2365	8.01	7.68	a shaker with circular movement.	100rp/min	
2366	8.0	7-8	orbital shaker	100 R/min	
2369	8.0	7.5	rotate	100r/min	
2370	7.97	7.65	250 ml conical flask	100 rpm	
2375	8.0	7.9	Rotational	100 rpm	
2378	7.80	7.85	Constant temp. shock tank	100time/min	
2379	pH = 7.96	pH = 7.78	Horizontal shaker	100	
2380	8.0	7.60	Mechanical Shaker	100	

lab	pH before extraction	pH after extraction	type shaker used	frequency shaker	remarks
2382	8.0	7.0~8.0	Mechanical vibration	100r/min	
2385	8,00	7,8	Orbital	110/minute	
2390	7.99	7.81	Orbital shaker	100 RPM	
2410	8.0	7.74	Orbital shaker	(100 +/- 10)/min	
2415	7.98	7.86	Orbital	100 rpm	
2426	8.0	7.81	Orbital	100 rpm	
2433	8	7	mechanical	100	
2442	8.03	7.88	Mechanical shaker	100 rpm	
2449	7.83	7.96	orbital	60rpm	
2452					
2455		7.76	magnetic stirrer	~200 rpm	
2459	7.89	8.95	mechanical shaker	100 +/- 10 rpm	
2460	7.96	7.69	mechanical shaker	100 rpm	
2462	8.0	7.6	IKAHS501 digital	100r/min	
2475	8.0	7.73	horizontal shaker	100rpm	
2477	8,0	7,77	Orbital shaker	100 rpm	
2489	8	7.67	Orbital shaker	100 RPM	
2495	8.00	7.60	linear	150	
2497	8	7.6	orbital shaker	100	
2501	8.0	7.60	Horizontal shaker	100 rpm	
2504	8.03	7.62	orbital shaker	100 rpm	
2511					
2527	pH 8.0	Did not measure	Water bath, able to shake mechanically	120 times per minute	
2532	pH- 8.0	pH - 7.6	Orbital shaker	100 rpm	
2538	8,0	7,7	Horizontal Shaking Device Bühler SM 30 Control	100 rpm	
2549	8	7.8	Rotation	100 rpm	
2553	7.8	7.8	Mechanical Shaker	100 min -1	
2560	8.002	7.5-8.0	Orbital Mechanical Shaker	100±10	
2561	8.05	7.65	Orbital Shaker - Edmund Buhler	100 RPM	
2563	n. m.	7,6	orbital shaker	100 rpm	
2569	7.95	7.8	rotational shaker	100 RPM	
2582	8.0 pH	7.8 pH	Mechanical Orbital Shaker	100 rpm	
2590	8.06	7.66	Orbital shaker	100	
2592					
2602	pH 8,0	pH 7,63	Bühler SM-30 control	100 (+/- 10) U/min	
2605	8.02	7.80	ZD-8802	100r/min	
2610	8.00	7.60	orbital shaker	100 rpm	recovery 102.7%
2612	8,03	8,05	Promax 2020 horizontal shaker	93 rpm	50 mm cuvette
2619	8.0	7.5		100	
2624	8,0 pH	between 7-8	orbital shaker	100 1/min	
2629	8.01	7.59	Horizontal shaker	100 cycles per minutes	
2643	7.9	7.9	JISICO Shaking Incubator	100 min-1	
2646	8,05	7,64	Lab Shaker Edmund Bühler SM25	125 rpm	
2656	8		Mechanical	12rpm	
2668	pH-8.0	pH-7.9	Orbital shaker	100 RPM	
2674	8.04	7.59	mechanical orbital shaker ireciprocating	100Hz	
2695	8,01	7,63	orbital shaker	100 min-1	
2705	8	-	orbital shaker	150 min-1	
2711	8,1	7,8	Orbital	100 Hz	
2725	8,01	pH1=7,63 pH2=7,64	Orbital Shaker, SSL1, Stuart 6a	100 rpm	
2730	8,0	7,61	orbital shaker	100 rpm	
2734	8.0		orbital	210 rpm	
2741	8.0	7.8	Mechanical Shaker	100cycles/minute	
2749	8.0	8.0	Horizontal schüttler	60	
2756					
2758	8.0	7.7	orbital	100 rpm	
2777	7.96	7.88	horizontal shaker	50 rpm	
2778	8.0 ±0.1	7.8	Mechanical	125 times/min	
2783	8.04	7.71	Orbital	200 rpm	Used cell length: 2cm instead of 4 cm

lab	pH before extraction	pH after extraction	type shaker used	frequency shaker	remarks
2789	8.00	7.73	Orbital shaker	100 rpm	
2791	8.01	7.6	Orbital Shaker	100	
2804	8	7.6	Mechanical Shaker	100 rpm	
2806					
2812	8	7,6	mechanical	100 rpm	
2823	pH 7.99.	pH 7.60.	Orbital.	100 rpm.	
2829	8.00	7.61	orbital shaker	100 min−1	
2830	8.0	7.6	Orbitale	100 trs/min	Recovery rate: 84.37%
2836	pH init 8.0.	pH final was 7.5.	Orbital shaker.	100 RPM.	
2844	8.00	7.73	Orbital shaker	110rpm	
2862	not det.	7,7	E. Bühler SM 30C control	115 rpm	
2866	8,06	sample a) 7,61 sample b) 7,63	circular shaker	86 rpm	during the extraction procedure some of the very fine sample pieces are sticking at the glas allthough the shaking speed was reduced
2867	7.8	7.8	Water bath shaker	140rpm	
2877	8.0	7.8	Orbital shaker	100 rpm	
2891	8.07	7.61/7.62	mechanical shaker	100 rpm	
3100	7.99	7.75	HY-6D	100r/min	
3116	8.0 ± 0.1	7.5 – 8.0	Horizontal mechanical shaker	100 pre min	
3118	7.95	8.00	horizontal	100 rpm	
3146	pH 8.0	pH 7.0 - 8.0	orbital shaker	100 min-1	
3149	8,03	7,63			
3150	8,0		horizontal shaker		
3154	8,0	7,7	horizontal	100 rpm	
3160	7,96	7,2	orbital shaker	100 rpm	
3172	8.00	7.62	Mechanical Shaker	100/min	
3190	8	7.5	Horizontal reciprocating oscillation	125rpm	
3192	8,0	7,6	Orbital Shaker (GFL: Model 3017)	100 ± 10 min-1	
3197	8,0	7,98	Mechanical orbital shaker	100 rpm	
3201	8,00	7,58	Heidolph Promax 1020	108 rpm	
3209	7.2	7.5	Mechanical shaker	100 ± 10 min-1	
3210	8.06	7.65	Rotoshake	14tours/min	
3216	7.81	7.81	Mechanical Shaker	120 rpm	
3228	8.0	7.7	orbital shaker	100min-1	
3233	8.04	7.91	mechanical shaker	90 shots per min	
3237	8,00	7,52		100 rpm	
3248	7.9	7.6	mechanical shaker	60 rates per min	

APPENDIX 3

Number of participants per country

4 labs in BANGLADESH
1 lab in BELGIUM
3 labs in BRAZIL
2 labs in BULGARIA
1 lab in CAMBODIA
1 lab in ETHIOPIA
1 lab in FINLAND
8 labs in FRANCE
19 labs in GERMANY
1 lab in GUATEMALA
8 labs in HONG KONG
1 lab in HUNGARY
11 labs in INDIA
3 labs in INDONESIA
12 labs in ITALY
6 labs in KOREA
1 lab in LUXEMBOURG
1 lab in MAURITIUS
3 labs in MEXICO
2 labs in MOROCCO
24 labs in P.R. of CHINA
4 labs in PAKISTAN
1 lab in PHILIPPINES
7 labs in SPAIN
2 labs in SRI LANKA
2 labs in SWITZERLAND
2 labs in TAIWAN R.O.C.
2 labs in THAILAND
1 lab in THE NETHERLANDS
2 labs in TUNISIA
5 labs in TURKEY
2 labs in U.S.A.
2 labs in UNITED KINGDOM
5 labs in VIETNAM

APPENDIX 4

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

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