Results of Proficiency Test
Chromium VI in leather
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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 Of all Chromium compounds, primarily Chromium VI was used, but this has been replaced by the less hazardous Chromium III in most applications.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.

The Institute for Interlaboratory Studies organizes since 2014 an interlaboratory study for the determination of Chromium VI in leather. In the annual proficiency test program of 2016/2017, this proficiency test was continued.
In this interlaboratory study, 164 laboratories in 33 different countries registered for participation (see appendix 3). In this report, the results of the 2017 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 was the organizer of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. Due to lack of a sufficient amount of suitable materials it was decided to send in this proficiency test only one aged leather sample, positive on Chromium VI, labelled #17540.
The participants were requested to report 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 regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol can be downloaded from 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 gray/green leather sample was obtained from the local market. The batch was grinded and aged. After thoroughly mixing, 190 bags were filled with approximately 5 grams of leather each and vacuumized. Eight stratified randomly selected samples were tested using ISO17075 to check the homogeneity of the batch.
The test results of the homogeneity tests are shown in table 1.

	Chromium VI in mg/kg
Sample #17540-1	15.4
Sample #17540-2	16.6
Sample #17540-3	14.9
Sample #17540-4	15.4
Sample #17540-5	16.8
Sample #17540-6	16.2
Sample #17540-7	14.6
Sample #17540-8	15.4

Table 1: homogeneity test results of subsamples #17540

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

	Chromium VI in mg/kg
r (observed)	2.2
reference test method	ISO17075:2017
r (reference test method)	2.8
0.3 x R (reference test method)	2.0

Table 2: evaluation of the repeatability of subsamples #17540

The calculated repeatability of the homogeneity test results was in agreement with the extrapolated repeatability and with 0.3 times the reproducibility mentioned in the reference method ISO17075:2017. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample (labelled #17540) of approx. 5 grams was sent on March 22, 2017.

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).

It was also requested to report the test results using the indicated units on the report form and not to round the test results, but to report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluation.
Also some analytical details were requested to be reported.

To get comparable test results a detailed report form and a letter of instructions are prepared. 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 were 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 per sample and per component in the appendix 1 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report 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 reanalyses). Additional or corrected test results are used for the data analysis and the original test results are placed under 'Remarks' in the test 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 organisation 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 March 2017 (iis-protocol, version 3.4).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were 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.

In accordance to ISO 5725 the original test results per determination were submitted subsequently to Dixon's, 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 averages and 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. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualise 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 proficiency 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 of 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 some cases, a reproducibility based on former iis proficiency tests could be used.

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 result is fit-for-use.

The z-scores were calculated in accordance with:

$z_{(target)}$ = (test result – average of proficiency test) / target standard deviation

The $z_{(target)}$ scores are listed in the result tables of 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 samples. Two participants reported results after the final reporting date and one other participant did not report any test results. Not all laboratories were able to report all analyses requested. In total, 163 participants reported 183 numerical results. Observed were 5 outlying test results, which is 2.7% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed. All statistical results reported on the leather sample is summarised in appendix 1.

In 2017 a new version of test method ISO17075 was published. The 2017 version of ISO17075 was split up into two parts: colorimetric method (part 1) and chromatographic method (part 2). In the previous version of ISO17075 (2007 version) only the colorimetric method was described.

Cr VI - colorimetric: The determination of Chromium VI at a concentration level of 16.7 mg/kg was not problematic. Only three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with ISO17075:2017 (part 1).

Cr VI - chromatographic: The determination of Chromium VI at a concentration level of 17.3 mg/kg was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with ISO17075:2017 (part 2).

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 (standard deviation*2.8) and the target reproducibility, derived from the official test method ISO17075:2017 are presented in the next table.

Parameter	unit	N	Average	2.8 * sd	R(target)
Chromium VI – colorimetric	mg/kg	149	16.69	6.91	6.90
Chromium VI – chromatographic	mg/kg	29	17.30	4.99	5.00

Table 3: performance overview for sample #17540

From the above table, it can be concluded, without further statistical calculations, that the participating laboratories have no problems with the analysis of Chromium VI in leather, when compared to the target reproducibility requirements of the ISO17075 method (part1 and 2), for this sample.

4.3 EVALUATION OF GROUP RESULTS AGAINST LIMITS FOR CHROMIUM VI

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 (part 1 and 2). Examples of regulations can be found in table 4.

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 4: Regulation on Chromium VI

When the results of this interlaboratory study were compared to this limit, it may be noticed that all, except one, 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 #17540, all, except one, of the laboratories would not release this sample to the consumer market.

5 COMPARISON WITH PREVIOUS INTERLABORATORY STUDIES

The observed variation in the test results for Chromium VI in the 2017 PT is in agreement in comparison with the variation as observed in the previous PTs, see below table.

Component	*May 2017*	*April 2016*	*February 2015*	*February 2014*	*Target*
Cr VI (colorimetric)	15%	29%	33%	19 – 31%	15%
Cr VI (chromatographic)	10%	n.e.	n.e.	n.e.	10%

Table 5: development of the uncertainties over the years

6 DISCUSSION

In this PT also some analytical details were asked (see appendix 2) to use for further statistical analyses. The majority (78%) of the participants is ISO/IEC 17025 accredited for the determination of Chromium VI in leather.

From the reported test methods it appeared that a large majority participants tested the leather samples according to the test method ISO17075, a colorimetric test method, and one participant used §64 B LFGB 82.02-11. These two tests methods appear to be similar (both in literature searches as in the results of this proficiency test). Other used test methods are DIN53314 and GB/T22807. Four participants used an in house test method which seems to be based on ISO17075.

It is remarkable that for the leather sample used in this proficiency test, the requested analytical details, mentioned in appendix 2, appeared to have no significant influence on the test result for this sample. The calculated reproducibility is in full agreement with the reproducibility mentioned in ISO17075:2017. The aging of the sample before the start of the PT as well as the relatively high Chromium VI in sample #17540 may be causes for the high quality of the test results.

Although, it can be concluded that the group of participants have no problems with the determination of Chromium VI in this sample, 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

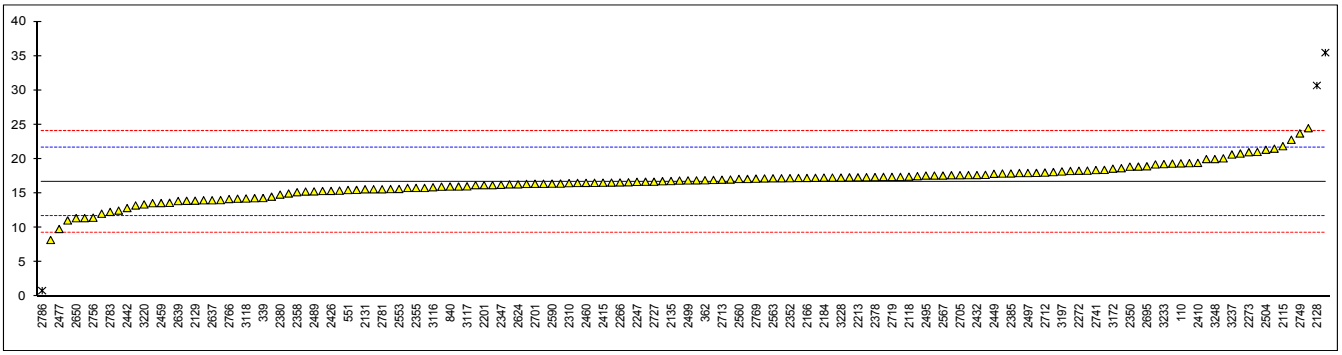
Determination of Chromium VI (colorimetric) in sample #17540; results in mg/kg

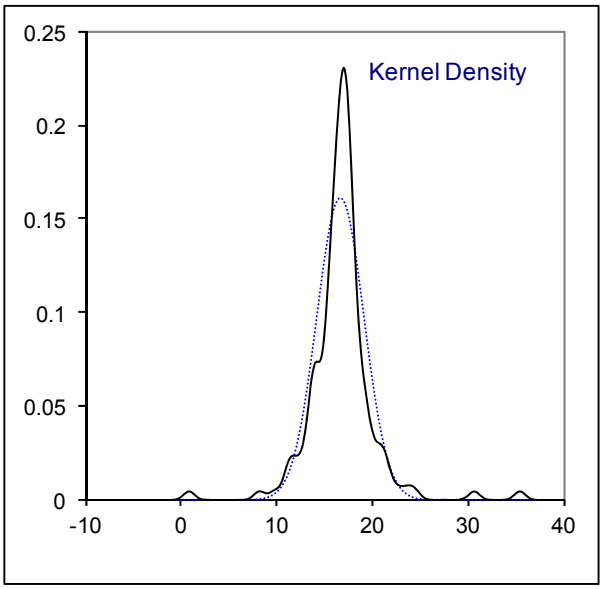
lab	method	value	mark	z(targ)	remarks
110	ISO17075-1	19.3724		1.09	
213		-----		-----	
339	ISO17075:2007	14.325		-0.96	
348	ISO17075-1	17.979		0.52	
362	ISO17075-1	16.918		0.09	
551	ISO17075-1	15.492		-0.49	
623	ISO17075-1	14.525		-0.88	
840	ISO17075-1	16		-0.28	
841	ISO17075-1	20.80		1.67	
1911	ISO17075-1	18.265		0.64	
2102	ISO17075-1	14.23		-1.00	
2115	ISO17075-1	21.875		2.10	
2118	ISO17075-1	17.433		0.30	
2128	ISO17075-1	30.7	C,R(0.01)	5.68	First reported 30.711
2129	ISO17075-1	13.94		-1.12	
2131	ISO17075-1	15.6		-0.44	
2132	ISO17075-1	17.4		0.29	
2135	ISO17075-1	16.801		0.05	
2137	ISO17075-1	16.52		-0.07	
2139	ISO17075-1	15.35		-0.54	
2159	ISO17075-1	18.31		0.66	
2165	ISO17075-1	17.642		0.39	
2166	ISO17075-1	17.252		0.23	
2184	ISO17075-1	17.3		0.25	
2201	ISO17075-1	16.20		-0.20	
2213	ISO17075-1	17.34		0.26	
2232	ISO17075-1	16.381		-0.13	
2247	ISO17075-1	16.68		0.00	
2255	ISO17075-1	15.8		-0.36	
2256	ISO17075-1	16.62		-0.03	
2266	ISO17075-1	16.6		-0.04	
2272	ISO17075-1	18.3		0.65	
2273	ISO17075-1	21.0		1.75	
2284	ISO17075-1	16.43		-0.11	
2290	ISO17075-1	16.32		-0.15	
2293	ISO17075-1	17.176		0.20	
2295	ISO17075-1	16.7		0.00	
2301		-----		-----	
2310	ISO17075-1	16.5		-0.08	
2311	ISO17075-1	16.87		0.07	
2320	ISO17075-1	16.77		0.03	
2330	ISO17075-1	20.10		1.38	
2347	ISO17075-1	16.25		-0.18	
2350	ISO17075-1	18.887		0.89	
2352	ISO17075-1	17.220		0.22	
2355	ISO17075-1	15.8		-0.36	
2357	ISO17075-1	16.4		-0.12	
2358	ISO17075-1	15.1756		-0.61	
2363	ISO17075-1	16.96		0.11	
2365	GB/T22807	16.5767		-0.05	
2369	ISO17075-1	15.8		-0.36	
2370	ISO17075-1	17.6		0.37	
2375	ISO17075-1	19.33		1.07	
2378	ISO17075-1	17.376		0.28	
2379	ISO17075-1	14.301		-0.97	
2380	ISO17075-1	14.829		-0.76	
2385	ISO17075-1	17.9		0.49	
2389	ISO17075-1	12.50		-1.70	
2390	ISO17075-1	11.405		-2.14	
2403	ISO17075-1	16.1994		-0.20	
2410	ISO17075-1	19.435		1.11	
2415	ISO17075-1	16.56		-0.05	
2419	ISO17075-1	8.225		-3.43	
2425	ISO17075-1	15.27		-0.58	
2426	ISO17075-1	15.36		-0.54	
2432	ISO17075-1	17.667		0.40	
2442	ISO17075-1	12.87		-1.55	
2449	ISO17075-1	17.853		0.47	
2455	ISO17075-1	17.247		0.23	
2459	ISO17075-1	13.597		-1.26	
2460	ISO17075-1	16.531		-0.06	
2475	ISO17075-1	17.27		0.24	
2477	DIN53314	9.819		-2.79	
2489	ISO17075-1	15.29		-0.57	
2495	ISO17075-1	17.5996		0.37	

lab	method	value	mark	z(targ)	remarks
2497	ISO17075-1	17.961		0.52	
2499	ISO17075-1	16.891		0.08	
2501	ISO17075-1	14.989		-0.69	
2504	ISO17075-1	21.34725		1.89	
2511	ISO17075-1	17.02		0.13	
2532	ISO17075-1	15.6		-0.44	
2538	ISO17075-1	17.30		0.25	
2549	ISO17075-1	15.4		-0.52	
2553	In house	15.64		-0.43	
2560	ISO17075-1	17.1010		0.17	
2561		-----		-----	
2563	ISO17075-1	17.2		0.21	
2567	ISO17075-1	17.6		0.37	
2569	ISO17075-1	16		-0.28	
2590	ISO17075-1	16.421		-0.11	
2592	ISO17075-1	16.20		-0.20	
2602	§64LFGB82.02-11	19.4		1.10	
2605	ISO17075-1	14.0		-1.09	
2609	GB/T22807	17.35		0.27	
2618	ISO17075-1	12.048		-1.88	
2624	In house	16.32		-0.15	
2629	ISO17075-1	11.0632		-2.28	
2637	ISO17075-1	14		-1.09	
2639	GB/T22807	13.89		-1.14	
2643	ISO17075:2007	17.89		0.49	
2646	ISO17075-1	18.4225		0.70	
2650	ISO17075-1	11.3941867		-2.15	
2654	ISO17075-1	18.67		0.80	
2656		-----		-----	
2658	ISO17075-1	15.63		-0.43	
2666	ISO17075-1	17.660		0.39	
2674	ISO17075-1	17.3367		0.26	
2682	ISO17075-1	13.25		-1.40	
2695	ISO17075-1	18.95		0.92	
2701	ISO17075-1	16.3998		-0.12	
2702	ISO17075-1	15.512		-0.48	
2705	ISO17075-1	17.650		0.39	
2706		-----		-----	
2711		-----		-----	
2712	ISO17075-1	18.03		0.54	
2713	ISO17075-1	16.99		0.12	
2716	ISO17075-1	17.202		0.21	
2719	ISO17075-1	17.4		0.29	
2725	ISO17075-1	15.9773		-0.29	
2727	ISO17075-1	16.7		0.00	
2730		-----		-----	
2737	ISO17075-1	17.96		0.52	
2741	ISO17075-1	18.4		0.69	
2743	ISO17075:2007	13.58		-1.26	
2749	ISO17075-1	23.728		2.86	
2756	ISO17075-1	11.466666		-2.12	
2758		-----		-----	
2766	ISO17075-1	14.18		-1.02	
2769	ISO17075-1	17.15		0.19	
2772		17.4		0.29	
2773		-----		-----	
2777	GB/T22807	35.48	R(0.01)	7.62	
2778	GB/T22807	24.493		3.17	
2781	ISO17075-1	15.60		-0.44	
2783	ISO17075-1	12.3127	C	-1.78	First reported 173.1032
2786	ISO17075-1	0.8555	R(0.01)	-6.43	
3100	ISO17075:2007	13.63		-1.24	
3109	In house	19.2147		1.02	
3116	In house	15.90		-0.32	
3117	ISO17075-1	16.028		-0.27	
3118	ISO17075-1	14.257		-0.99	
3146		-----		-----	
3150	ISO17075-1	18.1		0.57	
3151	ISO17075-1	16.54		-0.06	
3154		-----		-----	
3160	ISO17075-1	20.00		1.34	
3172	ISO17075-1	18.6		0.78	
3185	ISO17075-1	14.03		-1.08	
3191	ISO17075-1	13.92558	C	-1.12	First reported 34.81395
3192	ISO17075-1	18.9000		0.90	
3197	ISO17075-1	18.2		0.61	
3200	ISO17075-1	16.90		0.09	
3209	ISO17075-1	17.51		0.33	

lab	method	value	mark	z(targ)	remarks
3210		-----		-----	
3214	ISO17075-1	17.7		0.41	
3216	ISO17075-1	21.035		1.76	
3218	ISO17075-1	17.104		0.17	
3220	ISO17075-1	13.40		-1.33	
3222	ISO17075-1	22.81		2.48	
3228	ISO17075-1	17.3		0.25	
3233	ISO17075-1	19.261		1.04	
3237	ISO17075-1	20.66		1.61	
3243	ISO17075-1	21.51		1.96	
3248	ISO17075-1	20		1.34	

normality	suspect
n	149
outliers	3
mean (n)	16.690
st.dev. (n)	2.4691
R(calc.)	6.914
R(ISO17075-1:17)	6.901





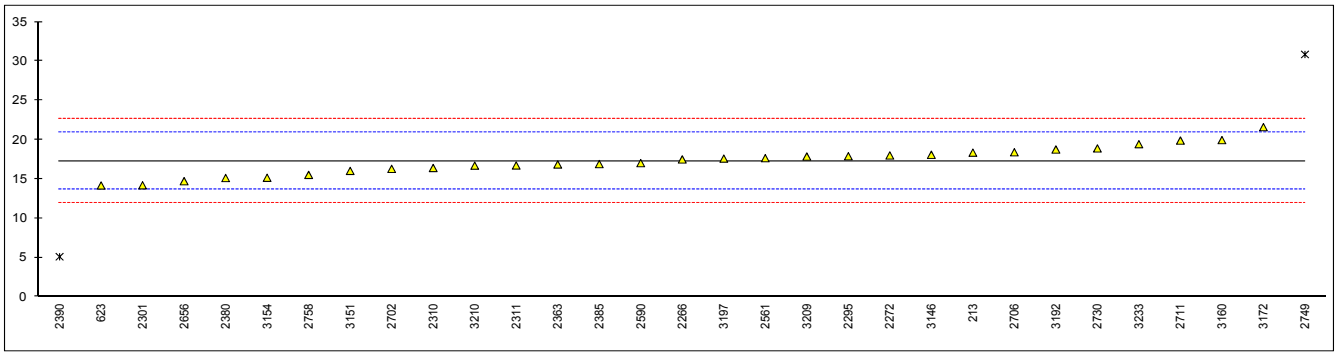
Determination of Chromium VI (chromatographic) in sample #17540; results in mg/kg

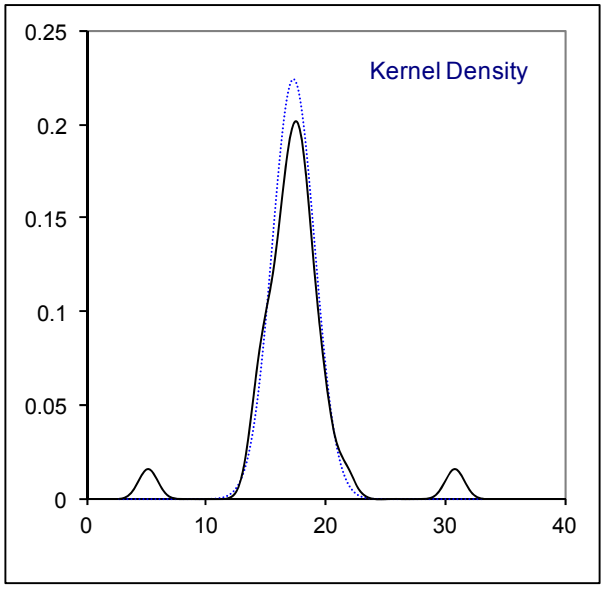
lab	Method	value	mark	z(targ)	remarks
110		-----		-----	
213	ISO17075-2	18.3575		0.59	
339		-----		-----	
348		-----		-----	
362		-----		-----	
551		NA		-----	
623	ISO17075-2	14.175		-1.75	
840		-----		-----	
841		-----		-----	
1911		-----		-----	
2102		-----		-----	
2115		-----		-----	
2118		-----		-----	
2128		-----		-----	
2129		-----		-----	
2131		-----		-----	
2132		-----		-----	
2135		-----		-----	
2137		-----		-----	
2139		-----		-----	
2159		-----		-----	
2165		-----		-----	
2166		-----		-----	
2184		-----		-----	
2201		-----		-----	
2213		-----		-----	
2232		NA		-----	
2247		-----		-----	
2255	ISO17075-2	NA		-----	
2256		-----		-----	
2266	ISO17075-2	17.49		0.11	
2272	ISO17075-2	18.0		0.39	
2273		-----		-----	
2284		-----		-----	
2290		-----		-----	
2293		-----		-----	
2295	ISO17075-2	17.9		0.34	
2301	ISO17075-2	14.20		-1.73	
2310	ISO17075-2	16.4		-0.50	
2311	ISO17075-2	16.72		-0.32	
2320		-----		-----	
2330		-----		-----	
2347		-----		-----	
2350		-----		-----	
2352		-----		-----	
2355		-----		-----	
2357		-----		-----	
2358	ISO17075-2	N/A		-----	
2363	ISO17075-2	16.85		-0.25	
2365		-----		-----	
2369		-----		-----	
2370		-----		-----	
2375		-----		-----	
2378		-----		-----	
2379		-----		-----	
2380	ISO17075-2	15.126		-1.22	
2385	ISO17075-2	16.9		-0.22	
2389		N/A		-----	
2390	ISO17075-2	5.10	C,R(0.01)	-6.83	First reported 10.81
2403		-----		-----	
2410		-----		-----	
2415		-----		-----	
2419		-----		-----	
2425		-----		-----	
2426		-----		-----	
2432		-----		-----	
2442		-----		-----	
2449		-----		-----	
2455		-----		-----	
2459		-----		-----	
2460		-----		-----	
2475		-----		-----	
2477		-----		-----	
2489		-----		-----	
2495		-----		-----	
2497		-----		-----	

lab	Method	value	mark	z(targ)	remarks
2499		-----		-----	
2501		-----		-----	
2504		N/A		-----	
2511		-----		-----	
2532		-----		-----	
2538		-----		-----	
2549		-----		-----	
2553		-----		-----	
2560	ISO17075-2	ND		-----	
2561	ISO17075-2	17.65		0.20	
2563		-----		-----	
2567		-----		-----	
2569		-----		-----	
2590	ISO17075-2	17.023		-0.15	
2592		-----		-----	
2602		-----		-----	
2605		-----		-----	
2609		-----		-----	
2618		-----		-----	
2624		-----		-----	
2629		-----		-----	
2637		-----		-----	
2639		-----		-----	
2643		-----		-----	
2646		-----		-----	
2650		-----		-----	
2654		-----		-----	
2656	ISO17075-2	14.72		-1.44	
2658		-----		-----	
2666		-----		-----	
2674		-----		-----	
2682		-----		-----	
2695		-----		-----	
2701		-----		-----	
2702	ISO17075-2	16.291		-0.56	
2705		-----		-----	
2706	ISO17075-2	18.406		0.62	
2711	ISO17075-2	19.89		1.45	
2712		-----		-----	
2713		-----		-----	
2716		-----		-----	
2719		-----		-----	
2725		-----		-----	
2727		-----		-----	
2730	ISO17075-2	18.89		0.89	
2737		-----		-----	
2741		-----		-----	
2743		-----		-----	
2749	ISO17075-2	30.851	R(0.01)	7.58	
2756		-----		-----	
2758	ISO17075-2	15.52	C	-1.00	First reported 23.24
2766		-----		-----	
2769		-----		-----	
2772		-----		-----	
2773		-----		-----	
2777		-----		-----	
2778		-----		-----	
2781		-----		-----	
2783		-----		-----	
2786		-----		-----	
3100		-----		-----	
3109		-----		-----	
3116		-----		-----	
3117		-----		-----	
3118		-----		-----	
3146	ISO17075-2	18.08		0.44	
3150		-----		-----	
3151	ISO17075-2	16.03		-0.71	
3154	ISO17075-2	15.159		-1.20	
3160	ISO17075-2	19.95		1.48	
3172	ISO17075-2	21.6		2.41	
3185		-----		-----	
3191		-----		-----	
3192	ISO17075-2	18.7550		0.81	
3197	ISO17075-2	17.6		0.17	
3200		-----		-----	
3209	ISO17075-2	17.86		0.31	
3210	In house	16.70		-0.34	

lab	Method	value	mark	z(targ)	remarks
3214		-----		-----	
3216		-----		-----	
3218		-----		-----	
3220		-----		-----	
3222		-----		-----	
3228		N/A		-----	
3233	In house	19.435		1.20	
3237		-----		-----	
3243		-----		-----	
3248		-----		-----	

normality	OK
n	29
outliers	2
mean (n)	17.299
st.dev. (n)	1.7816
R(calc.)	4.988
R(ISO17075-2:17)	5.003





APPENDIX 2

Summary of reported analytical details

Lab	ISO/IEC17025 accredited	Pretreatment	particle size before analysis	time between grinding and extraction
110	Yes	Used as received	NA	NA
213	No	Used as received		
339	No	Used as received	As received	No grinding/cutting
348	No	Used as received	as received	less than a minute
362	Yes	Used as received		
551	Yes	Used as received		
623	No	Used as received		Immediate
840	Yes	Used as received		
841	---	---		
1911	No	Used as received	used as received	-
2102	Yes	Used as received		
2115	Yes	Grinded	-	-
2118	Yes	Used as received	as received	none
2128	Yes	Used as received		< 5 min.
2129	Yes	Used as received		
2131	No	Used as received	used as received	10
2132	Yes	Used as received	Same as received	immediately
2135	Yes	Used as received		10
2137	Yes	Used as received		
2139	Yes	Cut	5 mm	About 10 minutes.
2159	Yes	Used as received	N/A (grinded)	300
2165	Yes	Used as received	N/A	N/A
2166	Yes	Used as received		
2184	Yes	Used as received	use as received	NA
2201	Yes	Used as received	<1mm	as soon as possible
2213	Yes	Used as received		
2232	Yes	Used as received	NA	NA
2247	Yes	Used as received	test performed on as is sample , it was grinded one	NA
2255	Yes	Used as received	NA	NA
2256	Yes	Used as received	N/A	N/A
2266	Yes	Used as received		14400
2272	No	Used as received	Powder	N/A
2273	Yes	Used as received	POWDER	N/A
2284	Yes	Used as received	less than 2mm*2mm	NA
2290	---	---		
2293	No	Used as received	0.5 cm	0 minutes the samples was used as received
2295	Yes	Used as received		
2301	Yes	Cut	-	-
2310	Yes	Used as received	in received condition	Performed test in received condition
2311	No	Used as received	Sample received in grinded form	Sample received in grinded form
2320	Yes	Used as received		
2330	Yes	Used as received	N/A	N/A
2347	Yes	Used as received		
2350	Yes	Cut	3 ~ 5 mm	As soon as
2352	Yes	Used as received	/	15 minutes
2355	Yes	Cut	4mm	10min
2357	Yes	Used as received		
2358	Yes	Used as received	N/A	N/A
2363	Yes	Used as received	3*3mm	
2365	Yes	Used as received	Used as received.	NA
2369	No	Used as received		
2370	Yes	Used as received	Powder flocculent.	About 30 minutes.
2375	Yes	Cut	3x3mm	
2378	Yes	Grinded	powder	3 min
2379	Yes	Grinded	2 x 2 mm	Around 190 min
2380	Yes	Used as received	N/A	N/A
2385	Yes	Used as received		
2389	Yes	Used as received		
2390	Yes	Used as received	As received tested	After degressing within 5 minutes
2403	Yes	Used as received	4 mm	10 min
2410	Yes	Grinded	2 mm ~ 3 mm	5 day
2415	Yes	Cut	3 mm by 3mm	30 min
2419	Yes	Used as received		
2425	Yes	Used as received	As received	N/A
2426	Yes	Grinded	-	-

Lab	ISO/IEC17025 accredited	Pretreatment	particle size before analysis	time between grinding and extraction
2432	---	---		
2442	No	Used as received	It was grinding samples	5 minutes
2449	Yes	Grinded	very small	30min
2455	Yes	Used as received	2 mm	0
2459	Yes	Used as received	Grinded sample as received	5 minutes
2460	Yes	Used as received	was used as received	15 minutes
2475	No	Used as received		
2477	Yes	Used as received		
2489	Yes	Used as received	3 mm	-
2495	Yes	Used as received	ND	NA
2497	Yes	Used as received		
2499	Yes	Used as received		
2501	Yes	Used as received	N/A	N/A
2504	Yes	Used as received	2mm	-
2511	No	Used as received		
2532	Yes	Used as received	3mm	Sample used for extraction as received. no further cut prior analysis.
2538	Yes	Used as received		
2549	Yes	Used as received	Used as received	Not applicable, sample tested as received
2553	Yes	Used as received	5 mm*5 mm	NA
2560	Yes	Used as received	As received	10 mins
2561	No	Used as received		
2563	Yes	Used as received	0,1 - 3 mm	< 10 miin.
2567	Yes	Used as received	3 x 3 mm	--
2569	Yes	Used as received		
2590	Yes	Used as received		The sample was extracted immediately from the packaging, without being further cut
2592	Yes	Cut	< 4 mm	1 h
2602	Yes	Used as received	not measured	not measured because used as received
2605	Yes	Used as received	<1mm	No
2609	Yes	Used as received		
2618	Yes	Used as received		
2624	No	Used as received	/	/
2629	Yes	Used as received		
2637	Yes	Used as received		
2639	Yes	Used as received	powder	not mentioned
2643	Yes	Used as received	5 mm	extraction : 180 minutes(3 hours)
2646	Yes	Used as received	Used as received.	Used as received.
2650	Yes	Used as received	The same than we received	-
2654	Yes	Used as received		
2656	No	Used as received	as received	no grinding/cutting ; used as received
2658	Yes	Used as received		3 hours
2666	Yes	Used as received		15 min
2674	Yes	Used as received	just the same as received,big ones 3mm*3mm,others smaller	about 10 minutes
2682	---	---		
2695	Yes	Used as received	2 mm	10 minutes
2701	Yes	Used as received		2 weeks
2702	No	Grinded		
2705	No	Used as received		/
2706	Yes	Used as received	n/a	n/a
2711	No	Used as received		
2712	Yes	Grinded	as sample received	--
2713	Yes	Used as received	Grinded sample	-
2716	Yes	Used as received		
2719	Yes	Used as received		
2725	No	Used as received	as received.	960 min
2727	Yes	Used as received	the sample was used as received.	The sample wasn´t grinded neither cutted. Was used as received.
2730	No	Used as received	The sample was used as received and it was already grinded when we received it.	The sample was used as received and it was already grinded when we received it.
2737	Yes	Used as received		About 180minute
2741	Yes	Used as received	Grinded as receive	2 minutes
2743	Yes	Used as received	the sample was already grinded	the sample was already grinded
2749	Yes	Used as received		
2756	Yes	Grinded	4mm	We don't know
2758	No	Used as received	-	-
2766	Yes	Used as received	1 mm	Immediate
2769	Yes	Used as received	Used as received	Used as received
2772	Yes	Used as received	4mm	Used as received

Lab	ISO/IEC17025 accredited	Pretreatment	particle size before analysis	time between grinding and extraction
2773	---	---		
2777	Yes	Used as received		30minutes
2778	No	Used as received		
2781	No	Used as received		
2783	No	Used as received	This was not measured, we used the sample received "as is."	Since the sample arrived ground up, we do not have time information.
2786	No	Used as received		
3100	Yes	Used as received	NA	NA
3109	Yes	Used as received	1mm x 1mm	
3116	Yes	Used as received	used as received	15 minutes
3117	Yes	Used as received	As received	Within 5 minutes
3118	Yes	Cut	3mmx3mm	
3146	Yes	Used as received		
3150	Yes	Used as received		1h
3151	Yes	Used as received		
3154	Yes	Used as received		
3160	Yes	Used as received	Used as received	-
3172	Yes	Used as received	--	--
3185	Yes	Used as received	3mm*3mm	Not apply
3191	Yes	Used as received	0.3cm-0.5cm	
3192	Yes	Used as received	Used as received	Grinding by IIS (day unknown); sample unpacked and extrakted in about 10 minutes
3197	Yes	Used as received		Immediately
3200	Yes	Used as received		
3209	Yes	Cut	1 mm	10 minutes
3210	Yes	Used as received	-	-
3214	Yes	Used as received	2-3 mm	No grinding and test directly
3216	Yes	Used as received	-	-
3218	Yes	Used as received	As received.	5 min
3220	Yes	Used as received	As received.	Immediately.
3222	Yes	Used as received	< 5 mm	1 min
3228	Yes	Used as received		
3233	No	Used as received	/	/
3237	Yes	Used as received	Used as received	
3243	---	Used as received		
3248	Yes	Used as received	Use as received	10 minutes

Summary of reported analytical details, continued

Lab	extraction solution degassed	analytical solution degassed	extraction time and temperature	pH extraction solution	pH analytical solution
110	not degassed	not degassed	180 minutes, 24 °C	8.0	8.0
213	Argon	---	3 hours at ambient temperature	8	7,7
339	Argon	Argon	180 minutes, 25°C	7.90	7.62
348	Nitrogen	---	180 minutes 20ºC	7.98	between 7.5-8
362	Nitrogen	Nitrogen	180 min, 22.8C	7.89	7.50
551	Nitrogen	Nitrogen	180 minutes and 22ºC	8,010	7,656
623	Other	Other	3 hours; room temperature	7.9	7.9
840	---	---			
841	---	---			
1911	Nitrogen	Nitrogen	180 minutes at 21°C	8.0	7.6
2102	Argon	Argon (oxygen-free)	180 minutes at 20 °C	8.0	8.0
2115	Nitrogen	Nitrogen	180 min; 25°C	8.0	7.7
2118	Nitrogen	Nitrogen	180 min, 23°C	8.0	7.7
2128	Nitrogen	Nitrogen	180 min, appr. 20°C (RT)	8,1	7,8
2129	Argon	Argon			
2131	Nitrogen	Nitrogen	180, 22	7,91	7,60; 7,18
2132	Nitrogen	not degassed	room temperature, 3 hrs	8.0	7.6
2135	Nitrogen	Nitrogen	180min 19°C	7.9	7.6
2137	not degassed	not degassed	180 min, 21°C	8.0	7.7
2139	not degassed	not degassed	180 minutes(3 h) and 21°C	pH 8.0	pH 7.6
2159	Argon (oxygen-free)	Argon (oxygen-free)	180 minutes, 25°C	7.95 (25°C)	7.91 (25°C)
2165	Argon	Argon	180 min, 23.1°C	8.03	7.58
2166	Argon (oxygen-free)	Argon (oxygen-free)	180 Min / 20°C	8,0	7,76
2184	Argon (oxygen-free)	Argon (oxygen-free)	180 mins, 22 deg C	7.5	7.8
2201	Nitrogen	Nitrogen	180 minutes and 25°C	8.00	7.64
2213	Nitrogen	Nitrogen	180 minutes and room temp.	8.0	7.95
2232	Argon (oxygen-free)	Argon (oxygen-free)	180 minutes; room temperature	8.00	7.71
2247	Nitrogen	Nitrogen	180 minutes; Temp. 20 to 22 °C	8.02	7.65
2255	Nitrogen	not degassed	180 minutes and RT (24 °C)	8.0	7.8
2256	Argon	Argon	180 minutes / 25 deg. C	8.0	7.8
2266		not degassed			
2272	Nitrogen	Nitrogen	3 hours with room temperature	8.0	7.6
2273	Nitrogen	Nitrogen	180minutes / 22 C	N/A	7.0
2284	Nitrogen	Nitrogen	180min; 22°C	8.0	7.8
2290	---	---			
2293	Argon	Argon	180 minutes 90 RPM RT	8.03	7.57
2295	Nitrogen	Nitrogen	3 hours at room temperature	8	8
2301	Argon (oxygen-free)	Nitrogen	180 minutes at RT	8.0	7.8
2310	Argon	not degassed	Time: 180 minutes; Temp.: 25 C	8	7.7
2311	Nitrogen	not degassed	3 Hours, <25°C	8.0	7.9
2320	Nitrogen	not degassed	180 minutes at 27°C	8.0	7.5-8.0
2330	Nitrogen	Nitrogen	180 Minutes and 26 oC	8.003	7.764
2347	Nitrogen	---			7.6
2350	Nitrogen	Nitrogen	3 hours, Room temperature	7.5	7.5
2352	Nitrogen	not degassed	3 hours 24°C	7.6	7.7
2355	Nitrogen	Nitrogen	3h 25°C	7.5	7.3
2357	Argon (oxygen-free)	Argon (oxygen-free)	180min,23degree	7.0~8.0	7.0~8.0
2358	Nitrogen	Nitrogen	180 mins, room temperature	7.92	7.94
2363	Nitrogen	Nitrogen	3hr 22°C	7.5	7.5
2365	Nitrogen	not degassed	3 hours,23 °C	8.00	7.64
2369	Nitrogen	Nitrogen	3hour,23°C	7.5	7.5
2370	Argon	Argon	3 hours and RT (25°C).	pH = 8.00	pH = 7.63
2375	Other	Other	23	8.0	7.6
2378	Nitrogen	Nitrogen	180 min and 25 °C	8.0	
2379	Nitrogen	Nitrogen	180 minutes , 25 degree	8.00	7.56
2380	Argon	Argon	180 minutes & 22 °C	8.0	7.71
2385	Nitrogen	Nitrogen	180	8,0	8,0
2389	Nitrogen	Nitrogen	180 minutes at 23 C	8.02	7.71
2390	Nitrogen	not degassed	180 minutes at 21.6 C	8.04	7.59
2403	Nitrogen	Nitrogen	3h ; 32.5°C	9.18	8.01
2410	Nitrogen	Nitrogen	180 min, 24 °C	8.01	7.71
2415	Nitrogen	Nitrogen	3.30 hrs 22 C	8	7.64
2419	Other	Other	180 min., 26 C	8.0	7.5
2425	Nitrogen	Nitrogen	3 Hours, Room temperature	8.0	7.8
2426	Nitrogen	Nitrogen	180 min at Room Temperature	8	7.88
2432	---	---			
2442	Argon	Argon	3 hours & 25 degree	8.01	7.85
2449	Nitrogen	Nitrogen	180min	7.9	7.6

Lab	extraction solution degassed	analytical solution degassed	extraction time and temperature	pH extraction solution	pH analytical solution
2455	not degassed	not degassed	3 hours 24 C	7.82	7.54
2459	Argon	Argon	180 minutes at 25°C	8.0	8.2
2460	Argon (oxygen-free)	Argon (oxygen-free)	180 min at 20°C	7.97	7.63
2475	Nitrogen	Nitrogen	3H at ambiant temperature		
2477	Argon	Argon	3 hours 23°C	8.0	7.68
2489	Nitrogen	Nitrogen	3 hrs; room temperature	8	7.63
2495	Nitrogen	Nitrogen	3h	8.02	7.64
2497	Nitrogen	Nitrogen	180	7.6	7.7
2499	Nitrogen	Nitrogen	180 minutes and 23 °C	8,00	7,55
2501	Nitrogen	Nitrogen	180 minutes/24℃	8.0	7.6
2504	Argon (oxygen-free)	Argon (oxygen-free)	180 minutes , Temperature 25C	8.01	7.57
2511	Argon (oxygen-free)	Argon (oxygen-free)	3 hours 21°c	8,03	
2532	Nitrogen	Nitrogen	3 hrs shaking at room temp.	pH-8	pH-7.8
2538	Nitrogen	Nitrogen	180 min at room temperature	8.0	7.78
2549	Nitrogen	Nitrogen	3 hrs ; room temperature	8.0	7.6
2553	Argon (oxygen-free)	Argon (oxygen-free)	3 hrs/ 25 °C	8.0	7.8
2560	Nitrogen	Nitrogen	180 min, 22 °C	8.0	8.0
2561	Nitrogen	---	180 mins at room temp	8.01	between 7.0&8.0
2563	Argon	Argon	180 minutes; room temperature	8,0	7,62
2567	Nitrogen	Nitrogen	180 min; 23°C	8.0	7.8
2569	Nitrogen	Nitrogen	3 Hrs	8	7.5
2590	Argon (oxygen-free)	Argon (oxygen-free)	180 minutes; room temperature	8.03	7.79
2592	Nitrogen	Nitrogen	3 h , T = rt	7,6	7,6
2602	not degassed	not degassed	180 min and RT (about 20°C)	8	7,64
2605	Nitrogen	Nitrogen	3h,room temperature	8.00	7.69
2609	Nitrogen	Nitrogen	3h, 22°C	8.0	8.0
2618	Nitrogen	not degassed	3h, room temp		
2624	not degassed	not degassed	120 min room temperature	8,0	-
2629	Nitrogen	Nitrogen	3h ± 5 minutes/ 25 ± 2 0C		
2637	Argon	Argon	180 Minutes 20 C	7,8	7,5
2639	Argon (oxygen-free)	Argon (oxygen-free)	180minutes,22.5°C	8.07	7.82
2643	not degassed	not degassed	180 minutes(3 hours), (25°C)	7.9	7.9
2646	Nitrogen	Nitrogen	180min, room temperature	pH 8,00	pH 7,59
2650	Nitrogen	Nitrogen	180 minutes 21ºC	8.00	7.66
2654	Nitrogen	Nitrogen	180 min /24°C	8.0	7.7
2656	Nitrogen	not degassed	180minutes / 20°C	8,00	7,63
2658	Nitrogen	Nitrogen			
2666	Nitrogen	Nitrogen	180 min - 27°C	7,94	7,55
2674	Argon (oxygen-free)	not degassed	3 hours, RT (about 20 °C)	7.94	7.67
2682	---	---			
2695	Nitrogen	Nitrogen	180 minutes at 20°C	8.01	7,69
2701	Nitrogen	Nitrogen	3 hrs, room temperature		
2702	Nitrogen	Nitrogen	180 minutes and 22°C	8.01	7.75
2705	Argon	Argon	18 min / 25 °C	8.01	8.0
2706	Argon	Argon	180min / approx. 23°C	8.0	8.0
2711	Nitrogen	Nitrogen	180 minutes, 24 °C	8.0	7.5
2712	Nitrogen	Nitrogen	180 mins,23 °C	7.98	7.67
2713	Argon (oxygen-free)	Argon (oxygen-free)	Average 250 minutes 23 °C	8.00pH	7.52 pH
2716	Argon (oxygen-free)	not degassed	180 min / 22°C	8,0	7,7
2719	Argon (oxygen-free)	Argon (oxygen-free)	180 mins and 25oC	8.0	7.6
2725		not degassed	180 min/20-23ºC	8,0	7,6
2727	Nitrogen	not degassed	180 minutes at 23(+/-2)ºC	8.00	7.60
2730	Other	Other	190 minutes at RT	/	7.69
2737	Nitrogen	not degassed	180minutes ; 20.7°C	pH=8.01	pH-7.62
2741	Nitrogen	Nitrogen	180 minutes & 21°C	8.01	7.62
2743	Nitrogen	not degassed	3 hours 20°C	8.0	8.0
2749	Nitrogen	not degassed	180 min. @ 23°C	8.0	8.0
2756	Nitrogen	Nitrogen	4min and room temprature	7.9	7.76
2758	Other	Other	180 min at temperature ambient	8.0	Between 7.0&8.0
2766	Argon	Argon	3 hours at 30 deg C	7.8	7.8
2769	Nitrogen	Nitrogen	180 min. at 22ºC	8.05	7.65
2772	Argon (oxygen-free)	Argon (oxygen-free)	3hr and 25°C	before the extraction pH:7.99	after the extraction pH:7.89
2773	---	---			
2777	not degassed	---	3 hours and room temperature	9.80	7.62
2778	Argon	---	180 minutes and 25°C	pH=8	pH=7.9
2781	Nitrogen	Nitrogen			
2783	Nitrogen	Nitrogen	180 min at 22C (room temp)	8.00	7.84
2786	Nitrogen	Nitrogen	3 hours at room temp		
3100	Nitrogen	Nitrogen	180 minutes and 21.3°C	7.99	7.69

Lab	extraction solution degassed	analytical solution degassed	extraction time and temperature	pH extraction solution	pH analytical solution
3109	Nitrogen	not degassed	180min, 23°C	8.0	7.7
3116	Argon (oxygen-free)	Argon (oxygen-free)	180 mins, ambient (5-28°C)	pH 8.0	pH 7.8
3117	Nitrogen	Nitrogen	180 minutes; room temperature.	pH=8.0	pH=7.9
3118	Nitrogen	---	180 minutes, room temperature	7.92	7.65
3146	Nitrogen	Nitrogen	3 h RT (about 21-22°C)	8,0	7,6
3150	Other	not degassed	room temperature 3h	8,0	7,0 - 8,0
3151	Argon (oxygen-free)	Argon (oxygen-free)	180		
3154	Nitrogen	---	180 min		
3160	Nitrogen	Nitrogen	180 minutes at 23 ºC.	8,03	7,60
3172	not degassed	not degassed	3h - 25°C	8	7.5
3185	Nitrogen	Nitrogen	3 hours, normal temperature	8.00	7.88
3191	Nitrogen	Nitrogen	180 minutes, ambient:22 °C	7.92	7.78
3192	Argon (oxygen-free)	Argon (oxygen-free)	180 minutes at RT	8,0	7,59
3197	Argon (oxygen-free)	Argon (oxygen-free)	180 minutes and RT	8,0	7,8
3200	Argon (oxygen-free)	Argon (oxygen-free)	time: 180min temperature:21°C	8.0	7.6
3209	Nitrogen	Nitrogen	180 minutes at 23°C	7.6	7.5
3210	Nitrogen	Nitrogen	3h à 20°C	8.0	7.7
3214	Nitrogen	not degassed	180 mins, 25 degree	8.026	7.595
3216	Nitrogen	Nitrogen	180 min / Tª 22ºC	8.01	8.00
3218	Nitrogen	Nitrogen	180min,20°C	8.0	7.9
3220	Nitrogen	Nitrogen	3 hrs/ 25°C	8.0	7.99
3222	Nitrogen	Nitrogen	t=3 h -T=22°C	8.0	7.6
3228	Argon (oxygen-free)	Argon (oxygen-free)	180 minutes; room temperature	8.0	8.0
3233	Argon	Argon	3hours / Ambiant temperature	8.00	8.00
3237	Argon	Argon	180 minutes; room temperature	8.00	7.75
3243	Argon	Argon	180 minutes	7,95	7,62
3248	Argon (oxygen-free)	not degassed	180 minutes, 25°C	8.0	8.0

APPENDIX 3

Number of participants per country

6 labs in BANGLADESH
1 lab in BELGIUM
1 lab in BRAZIL
2 labs in BULGARIA
2 labs in CAMBODIA, Kingdom of
1 lab in ETHIOPIA
7 labs in FRANCE
17 labs in GERMANY
1 lab in GUATEMALA
8 labs in HONG KONG
12 labs in INDIA
3 labs in INDONESIA
13 labs in ITALY
6 labs in KOREA
1 lab in LUXEMBOURG
2 labs in MEXICO
2 labs in MOROCCO
1 lab in NETHERLANDS
29 labs in P.R. of CHINA
6 labs in PAKISTAN
1 lab in POLAND
2 labs in PORTUGAL
1 lab in SINGAPORE
5 labs in SPAIN
2 labs in SRI LANKA
5 labs in SWITZERLAND
4 labs in TAIWAN R.O.C.
2 labs in THAILAND
1 lab in TUNISIA
9 labs in TURKEY
3 labs in U.S.A.
1 lab in UNITED KINGDOM
7 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

Literature:

1 iis, Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation, March 2017
2 DIN 53316
3 ISO17075-1:17
4 ISO17075-2:17
5 ISO17234-1:15
6 9 XP G 08-014:97
7 P.L. Davies, Fr Z. Anal. Chem, 351, 513, (1988)
8 W.J. Conover, Practical; Nonparametric Statistics, J. Wiley&Sons, NY, p.302, (1971)
9 ISO 5725, (1986)
10 ISO 5725, parts 1-6, (1994)
11 ISO 13528:05
12 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
13 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
14 G. Rohm, J. Bohnen & H. Kruessmann, GIT Labor-Fachzeitschrift, p 1080, 11, (1997)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, T*echnometrics*, 25(2), 165-172, (1983)
16 Analytical Methods Committee Technical brief, No 4.January 2001
17 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
18 Horwitz, W and Albert, R, J. AOAC Int, 79, 3, 589, (1996)