

Results of Proficiency Test  
Metals in Plastics  
September 2008

Organised by: Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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Report: iis08P02X

October 2008

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## 1 INTRODUCTION

World-wide, many consumer products with plastic parts are produced and transported. These plastic parts are produced under strict regulations. For instant in the European Directive 2002/95/EC maximum concentrations are specified for metals in plastic: the sum of Lead (Pb), mercury (Hg), Cadmium (Cd) and hexavalent Chromium (VI) may not exceed 0.1 %M/M, while the maximum concentration for Cadmium may not exceed 100 mg/kg.

Products are tested for metals contents in the countries of origin as well in EU-countries. Sometimes significantly different results are found, thus causing problems with the import of the products.

The determination of metals in plastics is known to give problems with the comparability of laboratory results. However, still only few plastic reference materials are available. As alternative, participation in a proficiency test may enable the laboratories to check their performance and thus to increase this comparability. Therefore, a proficiency testing scheme (laboratory-evaluating interlaboratory study) for the determination of metals in plastics is organised by the Institute for Interlaboratory Studies since 1998. Starting with only total Cadmium, over the years the scope was extended with total Lead, total Chromium and Chromium (VI).

In the international interlaboratory study of September 2008 99 laboratories from 32 different countries participated (See appendix 3). In this report the results of the proficiency test are presented and discussed.

## 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. It was decided to send 2 different samples (approximately 5 gram each), labelled #0841 and #0842. These two samples were also used in a previous proficiency test. Participants were also requested to report some details of the methods used.

### 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43 and ILAC-G13:2000. This ensures 100% confidentiality of participant's data. Also customer's satisfaction is measured on a regular basis by sending out questionnaires.

### 2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of November 2008 (iis-protocol, version 3.1). This protocol can be downloaded from the iis website <http://www.iisnl.com>.

## 2.3 CONFIDENTIALITY STATEMENT

All data present 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 of 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

Two different samples that were used in a previous proficiency test were selected. The first material (#0841) obtained from a participating laboratory, was a beige coloured polypropylene. The second material (#0842), also obtained from the same participating laboratory, was a light beige coloured polypropylene. The materials were divided over plastic bags, about 5 grams for each sample. The homogeneity of the subsamples #0841 was checked by determination of Chromium (VI) content on 4 stratified random selected subsamples. The homogeneity of the subsamples #0842 was checked by determination of Cadmium and Lead content on 4 stratified random selected subsamples.

<i>Chromium (VI) in mg/kg Subsample of #0841 (pp)</i>	<i>Total Cadmium in mg/kg Subsample of ##0842 (pp)</i>	<i>Total Lead in mg/kg Subsample of #0842 (pp)</i>
12	110	85
13	110	80
12	110	86
11	105	82

Table 1: results of the homogeneity test on the subsamples #0841 and #0842

From the results of the homogeneity tests, the repeatability was calculated and compared with the requirements of EN1122:2001 and Horwitz in the next table:

	<i>Subsample #0841 Chromium (VI) in mg/kg</i>	<i>Subsample #0842 Total Cadmium in mg/kg</i>	<i>Subsample #0842 Total Lead in mg/kg</i>
Observed r in mg/kg	2.3	7.0	7.7
r (Horwitz) in mg/kg	1.5	--	7.7
r (EN1122:01) in mg/kg	--	10.9	--

Table 2: repeatabilities of total Cadmium and total Lead of the subsamples #0841 and #0842

The repeatability calculated for the sample #0842 is in good agreement with the repeatability required by EN 1122 and the estimated repeatability calculated using the Horwitz equation. The observed repeatability of the sample #0841 is almost in agreement with the estimated repeatability calculated using the Horwitz equation. Therefore, homogeneity of all subsamples was assumed.

To each of the participating laboratories one set of samples, (1\* sample #0841 and 1\* sample #0842) were sent on August 21, 2008.

## 2.5 ANALYSIS

The participants were requested to determine on both samples: total Cadmium, total Lead, total Chromium and hexavalent Chromium (VI). It was explicitly requested to treat the samples as if it were routine samples and to report the analytical results using the indicated units on the report form and not to round the results, but report as much significant figures as possible. It was also requested not to report 'less than' results which are above the detection limit, because such results can not be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. Also a letter of instructions was added to the package.

The laboratories were requested to complete the report form with some details of the methods used.

## 3 RESULTS

During four weeks after sample despatch the results of the individual laboratories were received. The original data are tabulated per sample in the appendix 1 of this report. The laboratories are represented by their code numbers.

Directly after deadline, a reminder fax was sent to those laboratories that did not report results at that moment.

Shortly after the deadline the available results were screened for suspect data. A 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 results. Additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

### 3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of November 2003 (iis-protocol, version 3.0).

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers this check was repeated. All data sets proved to have a normal distribution.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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 analysis results are plotted. The corresponding laboratory numbers are under the X-axis. A straight line presents the average of the reported data. Two striped lines present the reproducibility limits of the selected standard, calculated as mean  $\pm$  target reproducibility, parallel to the average line. 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. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 4; nr.14 and 15).

### 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 spread of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

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

The literature requirements were taken from EN 1122:2001, "Determination of Cadmium in plastics with the method of the wet decomposition" for Cadmium and, for Lead from ASTM D4004:1998. For total Chromium and Chromium (VI) no literature requirements were available

The  $z_{(\text{target})}$  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. Therefore the usual interpretation of z-scores is as follows:

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

## 4 EVALUATION

In this interlaboratory study some problems were encountered with despatch of the samples. Several laboratories in Hungary, U.S.A. and United Kingdom received the samples near or after the final reporting date.

Eight other participants reported the results after the final reporting date and three participants did not report any results at all. Not all laboratories were able to report all analytes requested.

Finally, 96 of the 99 participants submitted analysis results. The 96 reporting laboratories sent in 418 numerical results. Observed were 5 outlying results, which is 1.2 %. In proficiency studies outlier percentages of 3 % - 7.5 % are quite normal.

### 4.1 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities derived from the literature standards are compared in the next tables.

Parameter	unit	n	Average	2.8 * sd	R (lit)
Total Cadmium #0841	mg/kg	19	<5	unknown	unknown
Total Lead #0841	mg/kg	20	<5	unknown	unknown
Total Chromium #0841 (*)	mg/kg	84	194.75	48.83	39.46
Hexavalent Chromium (VI) #0841	mg/kg	60	See page 27		

Table 3: performance overview for PP sample #0841

(\*) #0841 was used before as #0651. The Chromium results of #0651 were: average = 189.2 mg/kg, 2.8\*sd = 57.3 mg/kg

Parameter	unit	n	Average	2.8 * sd	R (lit)
Total Cadmium #0842 (**)	mg/kg	94	94.87	24.18	23.72
Total Lead #0842 (**)	mg/kg	90	97.74	22.77	24.92
Total Chromium #0842	mg/kg	27	<5	unknown	unknown
Hexavalent Chromium (VI) #0842	mg/kg	8	<2	unknown	unknown

Table 4: performance overview for PP sample #0842

(\*\*) #0842 was used before as #0652. The Cadmium results of #0652 were: average = 95.7 mg/kg, 2.8\*sd = 18.8 mg/kg. The Lead results for #0652 were: average = 95.9 mg/kg, 2.8\*sd = 24.5 mg/kg.

Without further statistical calculations it can be concluded that there is a good compliance of the group of participating laboratories with the relevant standards for total Cadmium, total Lead and total Chromium.

The analytes that were problematic are discussed in paragraph 4.2.

## 4.2 EVALUATION PER ELEMENT

In this section the results are discussed per analyte.

Total Cadmium: No analytical problems were observed. For sample #0841, the group agreed on a result of less than 5 mg/kg, which may be below or near the detection limit of the test method EN1122:01. For sample #0842, only two results are outside the reproducibility limits. The calculated reproducibility is in good agreement with the requirements of EN1122:01. When the EN1122 data is evaluated separately, the calculated reproducibility of the (only 17!) EN1122 results is somewhat larger than for the whole group. When the results are evaluated by detection technique, the spreads of both AAS & ICP-results do meet the requirements of EN1122:01. The spread of the XRF-results is larger. This may be caused by low number of XRF results.

Total Lead: No analytical problems were observed. For sample #0841, the group agreed on a result of less than 5 mg/kg, which may be below or near the detection limit. For sample #0842, only one result is outside the reproducibility limits. The calculated reproducibility is, after rejection of the statistical outlier, in full agreement with the requirements of ASTM D4004:02 and also with the estimated reproducibility calculated using the Horwitz equation. When the results are evaluated by detection technique, the spread of the ICP-results  $R(\text{ICP})$  is smaller than  $R(\text{AAS})$  and  $R(\text{XRF})$ . These differences may be explained by the very different numbers of results reported per technique.

Total Chromium: This determination is somewhat problematic. For sample #0842, the group agreed on a result of less than 5 mg/kg, which may be below or near the detection limit. For sample #0841, eight results are outside the reproducibility limits. The calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the estimated reproducibility limits calculated using the Horwitz equation. When the results are evaluated by detection technique, none of the spreads does meet the reproducibility limits, although  $R(\text{ICP})$  is much smaller than  $R(\text{AAS})$  or  $R(\text{XRF})$ . These differences may be explained by the very different numbers of results reported per technique.

Chromium VI: The determination for Hexavalent Chromium is very problematic. For sample #0842, the group agreed on a result of less than 5 mg/kg, which may be below or near the detection limit. Almost all laboratories did detect Chromium VI in sample #0841. However, the reported results can be divided into four groups, pointing to the presence of large systematic errors: Group 1 with a mean value of 13.4 mg/kg, group 2 with a mean value of 58.8 mg/kg, group 3 with a mean value of 102.3 mg/kg and the last group (only two results) with a mean value of 168.4 mg/kg.

The component that was used to spike Chromium VI to the plastic was pure Potassium Dichromate. Therefore the total Chromium content should in principle be equal to the hexavalent Chromium content. See also paragraphs 4.3 and 5.

#### 4.3 EVALUATION OF THE METHODS USED

The reported details of the methods that were used by the participants are listed in appendix 2.

For the determination of total Cadmium in plastics, the EN1122 method is considered to be the official EC test method. In this proficiency test only 17 participants used a version of EN1122. Most other participants used in-house, IEC62321 or EPA test methods based on acid digestion (wet ashing or microwave destruction) and six participants used XRF as method.

No correlation between the method used and quality of the results could be found.

Only six laboratories did use XRF for the determination of metals and thus unfortunately no significant conclusions could be drawn about the quality of the results of this technique.

Fifteen participants used AAS as detection technique, while 73 participants used ICP.

Only five participants did correct for recovery.

It can be concluded that the determination of Cadmium in the investigated polypropylene materials was not problematic.

For the determination of total Lead in plastics, no standardized test method is available.

In this interlaboratory study, four participants reported to have used EN 1122, only applicable for Cadmium. Thirty-three participants used the EPA 3052, 3051 or 3050-B methods, which are designed for environmental samples and the applicability for plastics is unknown.

Five participants used XRF and most other participants used in-house methods based on acid digestion (wet ashing, microwave destruction or combustion).

No correlation between method used and quality of the results could be found.

Eleven participants used AAS as detection technique, while 74 participants used ICP.

Only five participants did correct for recovery.

It can be concluded that the determination of total Lead in the investigated polypropylene materials was not problematic.

For the determination of total Chromium in plastics, no standardized test method is available.

In this interlaboratory study, five participants reported to have used EN 1122, only applicable for Cadmium. Thirty-three participants used the EPA 3050, 3051 or 3052 methods, which are designed for environmental samples and the applicability for plastics is unknown.

Four participants used XRF and most other participants used in-house methods based on acid digestion (dry ashing, microwave destruction or combustion).

No correlation between method used and quality of the results could be found.

Twelve participants used AAS as detection technique, while 73 participants used ICP.

Five participants did correct for recovery.

It can be concluded that the determination of total Chromium in the investigated polypropylene materials at a level of 194.8 mg/kg is somewhat problematic.

For determination of hexavalent Chromium in polymers, a draft method is available: CD/IEC62321 (111/24/CD). The method is based on EPA 3060A and EPA7196. Regretfully this draft method does not yet mention repeatability/reproducibility requirements. In this interlaboratory study thirty-two laboratories used this draft method and all other participants reported to have used EPA 3060A or an in house method.

The test method for Chromium VI does contain several parts that may influence the test results strongly, like: the particle size, the digestion temperature or the alkaline digestion solution.

Therefore some important details (particle size, extraction technique and extraction conditions) were requested to be reported (see appendix 2). One can see that a very large variation of details was reported by the participants.

Furthermore, it is remarkable to see that several laboratories that reported to have used CD/IEC62321 did not reduce the samples to a particle size of less than 500 µm or even not at all.

#### 4.4 COMPARISON WITH PAST PROFICIENCY TESTS

The number of participants increased from 66 in 2005 to 99 in this round. The absolute number of statistical outliers was 27 in 2005 and is only 5 in 2008. The percentage of outliers decreased from 10.3% to 1.2 % of the numerical results.

The relative reproducibilities per range for Cadmium, Lead and Chromium as observed in this proficiency test are compared with the findings in previous rounds in table 6.

Range	50-100 mg Cd/kg	±250 mg Cd/kg	50-100 mg Pb/kg	±13000 mg Pb/kg	50-250 mg Cr/kg
2002	51 %	35 %	81 %	47 %	--
2003	31 %	--	100 %	83 %	--
2004	33 %	25 %	--	41 %	--
2005	21 %	--	--	35 %	34 %
2006	20 %	--	26 %	--	30 %
2007	23 %	--	22-30 %	--	43 %
2008	25 %	--	24 %	--	25 %
EN1122:01	25 %	25 %	--	--	--
ASTM D4004:98	--	--	38 %	26 %	--

Table 6: comparison of the relative Cd, Pb and Cr reproducibilities (in %) in the previous rounds and in the present round

## 5 CONCLUSIONS

The observed spread in the results for the total Cadmium determination in this proficiency test is in full agreement with the requirements of the standard EN1122:01. All laboratories proved to be able to test total Cadmium with sufficient reliability. This is in agreement with our earlier findings (ref 16).

The observed spread in the results for the total Lead determination is still improving compared with the previous proficiency tests. The spread is in agreement with the requirements of D4004:02 (see

paragraph 4.4). Five laboratories (labs 310, 2132, 2197, 3180, 3243) did have problems with the determination of total Lead content. All the other laboratories proved to be able to test total Lead with sufficient reliability.

The relative spread in the results for the total Chromium determination is still improving compared with the previous proficiency tests. Thirteen laboratories (1051, 2132, 2160, 2199, 3106, 3107, 3154, 3163, 3166, 3169, 3180, 3236 and 3248) of all 88 reporting laboratories did have problems with the determination of total Chromium content. All other laboratories proved to be able to test total Chromium with sufficient reliability.

The determination of hexavalent Chromium in polypropylene is very problematic in this proficiency test. During the sample preparation procedure hexavalent Chromium was added as pure  $K_2Cr_2O_7$ , hence the brownish colour of the samples #0841 and #0842. Therefore the hexavalent Chromium content was expected to be equal or close to the total Chromium content of 195 mg/kg.

Regretfully, no unambiguous assigned value could be determined. Only two laboratories were able to find results that are near the theoretical value of 195 mg/kg.

The draft method IEC62321 prescribes the sample pre-treatment, with one very essential test detail. The sample must be grounded into a fine powder and passing through a 500  $\mu m$  sieve (chapter 10.5). From appendix 2 it is clear that many laboratories did not reduce the particle size sufficiently. Several laboratories reported a corrected Chromium VI result after an additional reduction step: Laboratory 2172 initially reported 8.6 mg/kg (particle size 1 mm) and 114 mg/kg after additional reduction to powder.

Laboratory 3166 initially reported 3.98 mg/kg (particle size 0.1-0.3  $cm^2$ ) and 67 mg/kg after additional reduction to powder.

**APPENDIX 1**

Determination of total Cadmium as Cd on sample #0841; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EPA3051	0.0669		----	
310	EN1122	1		----	
358	in house	<1		----	
390	EPA3052/6020A	<0.1		----	
452	in house	0.08		----	
453		----		----	
551	IEC	nd		----	
607	EPA3051A	nd		----	
622	EN1122	<1		----	
632	EPA3052	nd		----	
840	EPA3051/52	<2		----	
1051	EN1122	<5		----	
1121		----		----	
1126	in house	<20		----	
1800	in house	<5		----	
1842		----		----	
2117	DIN EN ISO17294	0.077		----	
2129	in house	0.067		----	
2131	in house	<0.01		----	
2132	in house	nd	C	----	First reported <3
2139	EN1122	<5.0		----	
2146	EN1122	nd		----	
2152	in house	<10		----	
2153	EPA3051	<12		----	
2156	IEC62321	0.1		----	
2160	in house	<12		----	
2161	EN1122B	0.2		----	
2167	IEC62321	0.14		----	
2169	IEC62321	<5		----	
2172	IEC62321	nd		----	
2179	EPA3052/6010C	<3		----	
2182	EN1122	<5		----	
2184	EN1122	<1		----	
2187	EPA3052/6010C	<2		----	
2189	in house	<5		----	
2190	in house	nd		----	
2192	EPA3052/6010C	nd		----	
2194	in house	0.158		----	
2196	EN1122B	<2		----	
2197	in house	<0.05		----	
2199	EPA3052	<2		----	
2202	IEC62321	nd		----	
2210	EN1122A	nd		----	
2211		----		----	
2212		----		----	
2214	in house	1		----	
2215	EPA3051	<2		----	
2217	IEC62321	0.07		----	
2310	EPA3052	<2		----	
2312	EPA3051A	<2.0		----	
2350	IEC62321	nd		----	
2361	EPA3052	<5		----	
2362	EPA3052	<5		----	
2363	IEC62321	<2		----	
2365	EPA3052	nd		----	
2366	EPA3052	nd		----	
2369	EPA3052	<2		----	
2370	EPA3052	nd		----	
2372	EPA3052	nd		----	
2375	IEC62321	<5		----	
2379	IEC62321	nd		----	
2380		----		----	
2385	in house	<2		----	
3100	EPA3052	nd		----	
3104	EN1122	<10		----	
3106	in house	nd		----	
3107	EPA3052	<5		----	
3110	EN1122B	<5		----	
3111	in house	0.06		----	
3113	in house	<5		----	
3124	EPA3052	0.0844		----	

3126		----	----
3127	DIN22022-5	<3	----
3134	in house	<2.4	----
3143	F2617	0.317	----
3153	EPA3052	<2	----
3154		----	----
3159	EPA3051	<2	----
3160	in house	<0.6	----
3163	in house	4.6	----
3166	in house	<10	----
3169	EPA3052	nd	----
3172	EN1122	<10	----
3180		----	----
3182	IEC62321	nd	----
3185	IEC62321	<2	----
3191	in house	<5	----
3192	EN1122	<5	----
3199	EPA3051	<2.60	----
3200	in house	<2.0	----
3209	in house	0.25	----
3213	IEC62321	0.08	----
3218	IEC111-95/CDV	<2	----
3225	in house	<2.0	----
3231	EPA3051	<10	----
3236	in house	0.0755	----
3239	in house	<2.0	----
3243	in house	2.05	----
3248	IEC62321	<5	----
	normality	n.a.	
	n	19	
	outliers	0	
	mean (n)	<5	
	st.dev. (n)	n.a.	
	R(calc.)	n.a.	
	R(EN1122:01)	n.a.	

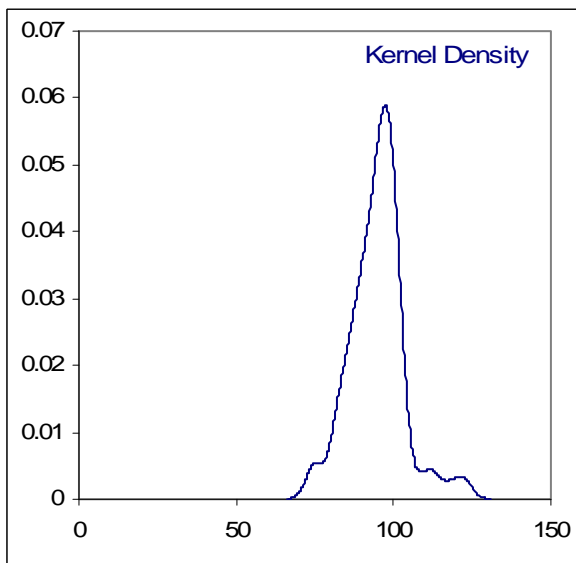
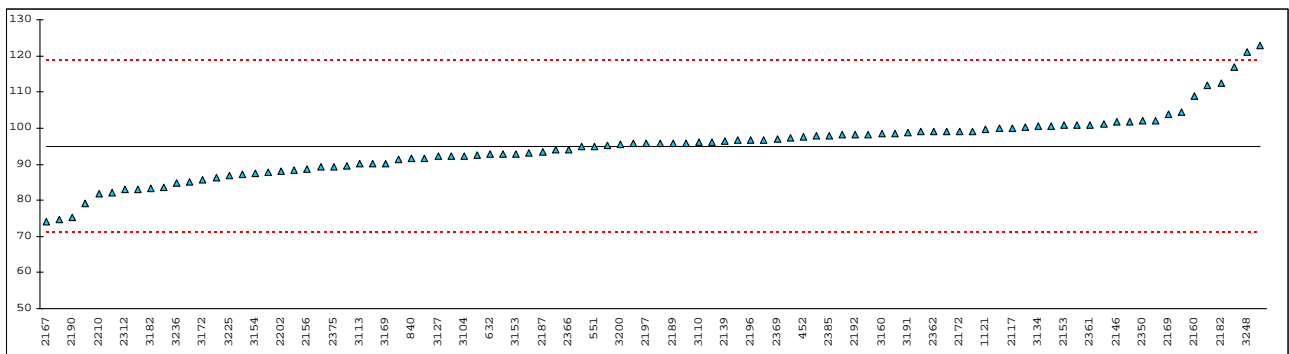
## Determination of total Cadmium as Cd on sample #0842; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EPA3051	92.5		-0.28	
310	EN1122	82		-1.52	
358	in house	87.2		-0.91	
390	EPA3052/6020A	74.67		-2.38	
452	in house	97.5		0.31	
453		-----		-----	
551	IEC	95.0		0.02	
607	EPA3051A	96.65		0.21	
622	EN1122	111.8206		2.00	
632	EPA3052	92.82		-0.24	
840	EPA3051/52	91.587		-0.39	
1051	EN1122	100.36		0.65	
1121	in house	99.6		0.56	
1126	in house	86.3		-1.01	
1800	in house	95.7		0.10	
1842		-----		-----	
2117	DIN EN ISO11885	100		0.61	
2129	in house	93		-0.22	
2131	in house	101.91		0.83	
2132	in house	92.85	C	-0.24	First reported 196.1
2139	EN1122	96.4		0.18	
2146	EN1122	101.6		0.79	
2152	in house	87.9		-0.82	
2153	EN1122	100.94		0.72	
2156	IEC62321	88.6		-0.74	
2160	in house	109.0		1.67	
2161	EN1122B	97.2		0.28	
2167	IEC62321	74.1		-2.45	
2169	IEC62321	103.9524		1.07	
2172	IEC62321	99.048		0.49	
2179	EPA3052/6010C	88.40		-0.76	
2182	EN1122	112.35		2.06	
2184	EN1122	95.86		0.12	
2187	EPA3052/6010C	93.28		-0.19	
2189	in house	95.8		0.11	
2190	in house	75.4		-2.30	
2192	EPA3052/6010C	98.20		0.39	
2194	in house	96.556		0.20	
2196	EN1122B	96.60		0.20	
2197	in house	95.7		0.10	
2199	EPA3052	91.3		-0.42	
2202	IEC62321	88.1		-0.80	
2210	EN1122A	81.9		-1.53	
2211		-----		-----	
2212		-----		-----	
2214	in house	83		-1.40	
2215	EPA3051	94.9916		0.01	
2217	IEC62321	95.3		0.05	
2310	EPA3052	92.2		-0.31	
2312	EPA3051A	82.99		-1.40	
2350	IEC62321	101.9		0.83	
2361	EPA3052	101.0		0.72	
2362	EPA3052	99		0.49	
2363	IEC62321	96.2		0.16	
2365	EPA3052	98.2		0.39	
2366	EPA3052	94.0		-0.10	
2369	EPA3052	96.9282		0.24	
2370	EPA3052	98.6		0.44	
2372	EPA3052	97.9		0.36	
2375	IEC62321	89.37		-0.65	
2379	IEC62321	94.0		-0.10	
2380		-----		-----	
2385	in house	98.0		0.37	
3100	EPA3052	99.11		0.50	
3104	EN1122	92.2531		-0.31	
3106	in house	116.9		2.60	
3107	EPA3052	123		3.32	
3110	EN1122B	96		0.13	
3111	in house	91.68		-0.38	
3113	in house	90.1		-0.56	
3124	EPA3052	89.3		-0.66	
3126	in house	99		0.49	

3127	DIN22022-3	92.16	-0.32
3134	in house	100.5	0.66
3143	F2617	89.6	-0.62
3153	EPA3052	92.9	-0.23
3154	DIN1122	87.48	-0.87
3159	EPA3051	90.14	-0.56
3160	in house	98.40	0.42
3163	in house	104.4	1.13
3166	in house	79.20	-1.85
3169	EPA3052	90.228	-0.55
3172	EN1122	85.8	-1.07
3180	EN1122	83.7	-1.32
3182	EPA3052	83.19	-1.38
3185	IEC62321	98.97	0.48
3191	in house	98.8	0.46
3192	EN1122	99.97	0.60
3199	EPA3051	101	0.72
3200	in house	95.62	0.09
3209	in house	95.7	0.10
3213	IEC62321	100.53	0.67
3218	IEC111-95/CDV	98.1	0.38
3225	in house	87.0	-0.93
3231	EPA3051	101.2	0.75
3236	in house	84.705	-1.20
3239	in house	101.69	0.81
3243	in house	85.01	-1.16
3248	IEC62321	121	3.09

normality	not OK
n	94
outliers	0
mean (n)	94.87
st.dev. (n)	8.636
R(calc.)	24.18
R(EN1122:01)	23.72

	Only EN1122 data:	Only AAS	Only ICP	Only XRF
OK	OK	OK	not OK	OK
	17	15	68	6
	0	0	0	0
	95.43	96.51	94.95	98.98
	9.156	8.406	7.782	14.168
	25.64	23.54	21.79	39.67
	23.86	24.13	23.74	24.75



## Determination of total Lead as Pb on sample #0841; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EPA3051	1.3800		----	
310	subcontracted within comp.	<10		----	
358	in house	<5		----	
390	EPA3052/6020A	<1		----	
452	in house	-0.18		----	
453		----		----	
551	IEC	2.5		----	
607	EPA3051A	nd		----	
622	in house	<2		----	
632	EPA3052	nd		----	
840	EPA3051/52	<2		----	
1051	in house	<5		----	
1121	in house	1.8		----	
1126		----		----	
1800	in house	<20		----	
1842		----		----	
2117	DIN EN ISO17294	<0.050		----	
2129	in house	0.091		----	
2131	in house	0.05		----	
2132	in house	nd	C	----	First reported 19.9
2139	EPA3052	<5.0		----	
2146		----		----	
2152	in house	<10		----	
2153	EPA3051	<12		----	
2156	IEC62321	0.5		----	
2160	in house	<12		----	
2161	EN1122B	1.4		----	
2167	IEC62321	0.83		----	
2169	IEC62321	<10		----	
2172	IEC62321	nd		----	
2179	EPA3052/6010C	<30		----	
2182	EPA3050B	<5		----	
2184	in house	2.28		----	
2187	EPA3052/6010C	<2		----	
2189	in house	<10		----	
2190	in house	nd		----	
2192	EPA3052/6010C	nd		----	
2194	in house	0.105		----	
2196	in house	<2		----	
2197	in house	<1		----	
2199	EPA3052	<2		----	
2202	IEC62321	nd		----	
2210	in house	nd		----	
2211	in house	<21.8		----	
2212	in house	<LOD		----	
2214	in house	6		----	
2215	EPA3051	<2		----	
2217	IEC62321	<0.3		----	
2310	EPA3052	<2		----	
2312	EPA3051A	<2.0		----	
2350	IEC62321	nd		----	
2361	EPA3052	<5		----	
2362	EPA3052	<5		----	
2363	IEC62321	<2		----	
2365	EPA3052	nd		----	
2366	EPA3052	nd		----	
2369	EPA3052	<2		----	
2370	EPA3052	nd		----	
2372	EPA3052	nd		----	
2375	IEC62321	<5		----	
2379	IEC62321	nd		----	
2380		----		----	
2385	in house	<2		----	
3100	EPA3052	nd		----	
3104	EPA3051	<50		----	
3106	in house	nd		----	
3107	EPA3052	<5		----	
3110	EPA3052	<10		----	
3111	in house	0.03		----	
3113	in house	<5		----	
3124	EPA3052	0.176		----	
3126		----		----	

3127	DIN22022-5	<3		----	
3134		----		----	
3143	F2617	1.2		----	
3153	EPA3052	<2		----	
3154		----		----	
3159	EPA3051	<4		----	
3160	in house	<4		----	
3163	in house	0.7		----	
3166	in house	<10		----	
3169	EPA3052	nd		----	
3172	EN1122	<10		----	
3180	EN1122	2.3	C	----	First reported 16.4
3182	IEC62321	nd		----	
3185	IEC62321	<2		----	
3191	in house	<5		----	
3192	in house	<20		----	
3199	EPA3051	<7.90		----	
3200	in house	<5.0		----	
3209	in house	2.5		----	
3213	EPA3052/6020A/IEC62321	0.14		----	
3218	IEC111-95/CDV	<2		----	
3225	in house	<10.0		----	
3231	EPA3051	<10		----	
3236	in house	0.126		----	
3239	in house	<10.0		----	
3243	in house	21.48		----	Result excluded , false positive
3248	IEC62321	<5		----	
	normality	n.a.			
	n	20			
	outliers	0			
	mean (n)	<5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit.)	n.a.			

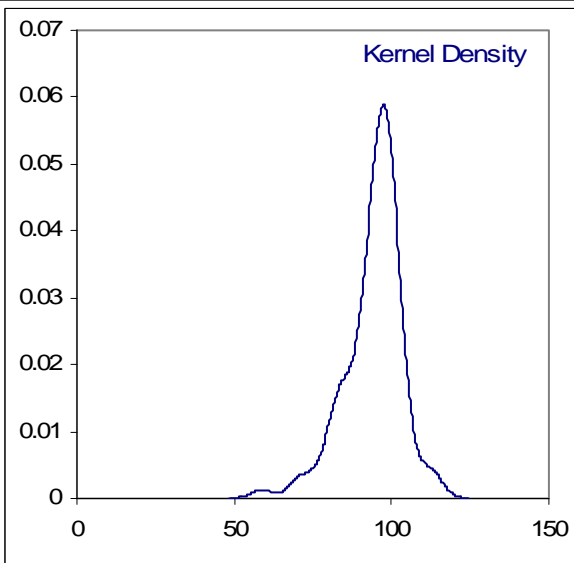
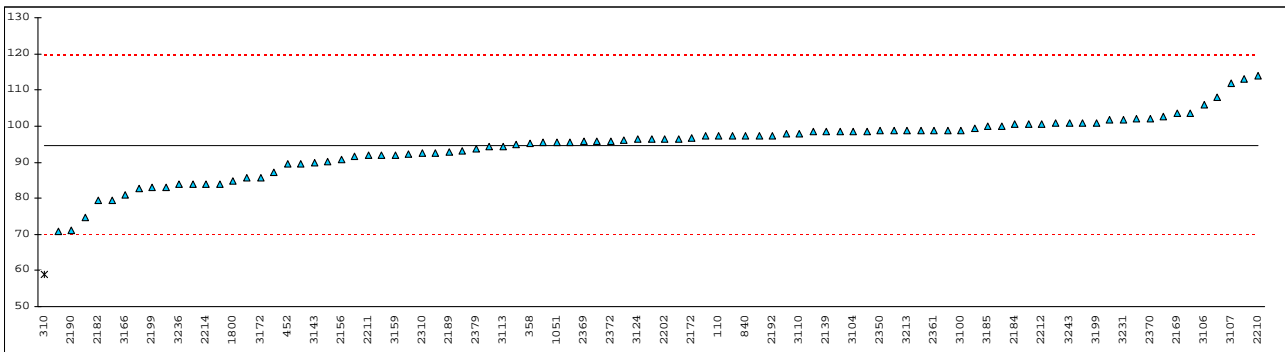
## Determination of total Lead as Pb on sample #0842; results in mg/kg

lab	method	value	Mark	z(targ)	remarks
110	EPA3051	97.2		0.28	
310	subcontracted within comp.	59	G(0.01)	-4.02	
358	In house	95.3		0.06	
390	EPA3052/6020A	97.33		0.29	
452	in house	89.6		-0.58	
453		-----		-----	
551	IEC	93.0		-0.20	
607	EPA3051A	95.58		0.09	
622	in house	100.5476		0.65	
632	EPA3052	100.905		0.69	
840	EPA3051/52	97.224		0.28	
1051	in house	95.55		0.09	
1121	in house	84		-1.21	
1126		-----		-----	
1800	in house	84.9		-1.11	
1842		-----		-----	
2117	DIN EN ISO11885	102		0.82	
2129	in house	113		2.05	
2131	in house	100.78		0.68	
2132	in house	91.65	C	-0.35	First reported 137.3
2139	EPA3052	98.4		0.41	
2146		-----		-----	
2152	in house	85.7		-1.02	
2153	EPA3051	101.64		0.78	
2156	IEC62321	90.6		-0.46	
2160	in house	108.0		1.49	
2161	EN1122B	97.9		0.36	
2167	IEC62321	74.6		-2.26	
2169	IEC62321	103.4078		0.97	
2172	IEC62321	96.836		0.24	
2179	EPA3052/6010C	83.91		-1.22	
2182	EPA3050B	79.39		-1.72	
2184	in house	100.44		0.64	
2187	EPA3052/6010C	97.16		0.27	
2189	in house	92.7		-0.23	
2190	in house	71.0		-2.67	
2192	EPA3052/6010C	97.33		0.29	
2194	in house	96.325		0.18	
2196	in house	102.60		0.88	
2197	in house	<0.3		-----	False negative
2199	EPA3052	83.0	C	-1.32	First reported 68.5
2202	IEC62321	96.4		0.19	
2210	in house	113.8		2.14	
2211	in house	91.9		-0.32	
2212	in house	100.6		0.66	
2214	in house	84		-1.21	
2215	EPA3051	92.1703		-0.29	
2217	IEC62321	89.7		-0.57	
2310	EPA3052	92.5		-0.25	
2312	EPA3051A	83.01		-1.32	
2350	IEC62321	98.63		0.44	
2361	EPA3052	98.8		0.46	
2362	EPA3052	96		0.14	
2363	IEC62321	98.8		0.46	
2365	EPA3052	95.9		0.13	
2366	EPA3052	95.5		0.09	
2369	EPA3052	95.8534		0.13	
2370	EPA3052	102		0.82	
2372	EPA3052	95.9		0.13	
2375	IEC62321	79.56		-1.71	
2379	IEC62321	93.8		-0.11	
2380		-----		-----	
2385	in house	98.7		0.45	
3100	EPA3052	98.83		0.46	
3104	EPA3051	98.5178		0.42	
3106	in house	105.8		1.24	
3107	EPA3052	112		1.94	
3110	EPA3052	98		0.37	
3111	in house	90.07		-0.52	
3113	in house	94.3		-0.05	
3124	EPA3052	96.3		0.18	
3126		-----		-----	

3127	DIN22022-3	92.00	-0.31
3134		-----	-----
3143	F2617	89.8	-0.55
3153	EPA3052	95.0	0.03
3154	DIN1122	70.69	-2.70
3159	EPA3051	92.04	-0.30
3160	in house	98.52	0.42
3163	in house	96.5	0.20
3166	in house	80.8	-1.57
3169	EPA3052	92.629	-0.24
3172	EN1122	85.8	-1.00
3180	EN1122	82.6	-1.36
3182	EPA3052	87.06	-0.86
3185	IEC62321	99.97	0.59
3191	in house	98.81	0.46
3192	in house	98.36	0.41
3199	EPA3051	101	0.70
3200	in house	94.28	-0.05
3209	in house	100.0	0.59
3213	EPA3052/6020A/IEC62321	98.71	0.45
3218	IEC111-95/CDV	99.4	0.52
3225	in house	97.2	0.28
3231	EPA3051	101.8	0.79
3236	in house	83.89	-1.22
3239	in house	103.44	0.98
3243	in house	100.8	0.68
3248	IEC62321	98.5	0.42

normality not OK  
n 90  
outliers 1  
mean (n) 94.74  
st.dev. (n) 8.132  
R(calc.) 22.77  
R(D4004:02) 24.92

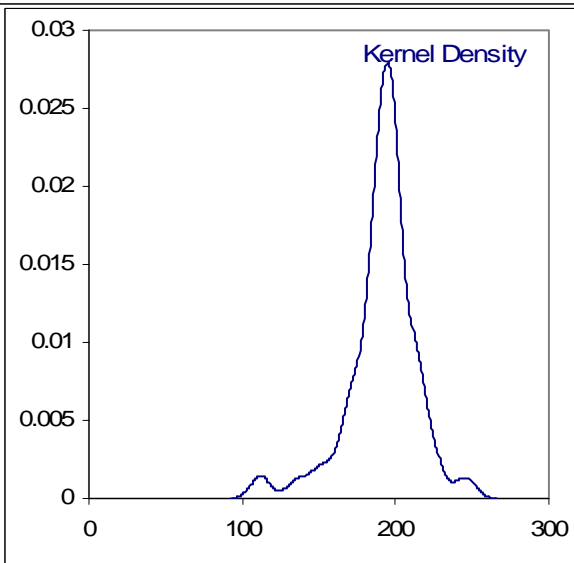
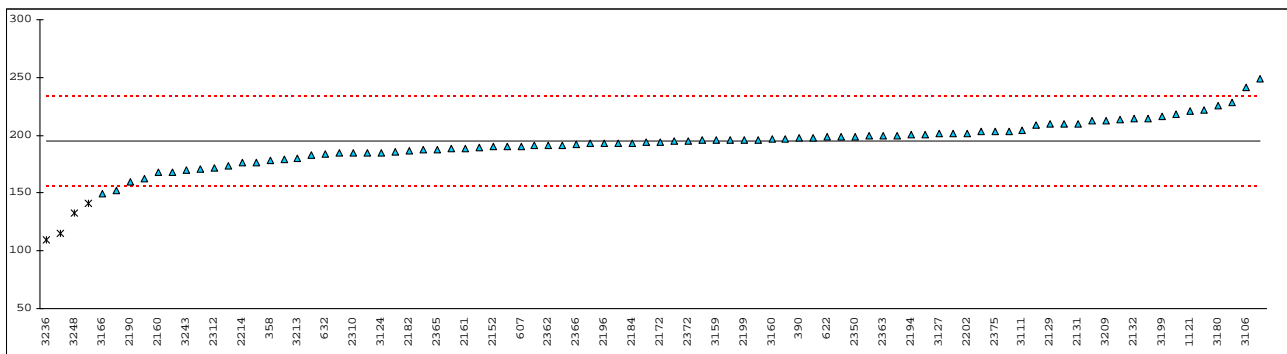
	<u>Only AAS</u>	<u>Only ICP</u>	<u>Only XRF</u>
OK	11	74	5
not OK	0	0	0
Compare	92.99	94.77	98.10
R(Horwitz) = 21.40	9.269	7.830	10.734
	25.95	21.92	30.05
	24.46	24.929	25.80



## Determination of total Chromium as Cr on sample #0841; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EPA3051	203.05		0.59	
310		-----		-----	
358	in house	178.6		-1.15	
390	EPA3052/6020A	197.67		0.21	
452	in house	197.8		0.22	
453		-----		-----	
551	IEC	188.1		-0.47	
607	EPA3051A	190.41		-0.31	
622	EN1122	198.7161		0.28	
632	EPA3052	183.75		-0.78	
840	EPA3051/52	190.913		-0.27	
1051	in house	114.87	G(0.05)	-5.67	
1121	in house	221.2		1.88	
1126		-----		-----	
1800	in house	202.9		0.58	
1842		-----		-----	
2117	DIN EN ISO 11885	200		0.37	
2129	in house	210		1.08	
2131	in house	210.21		1.10	
2132	in house	214.75	C	1.42	First reported 215.5
2139	EPA3052	213.4		1.32	
2146		-----		-----	
2152	in house	190		-0.34	
2153	EPA3051	197.22		0.18	
2156	IEC62321	170.8		-1.70	
2160	in house	168.4	C	-1.87	First reported 534.3
2161	EN1122B	188.6		-0.44	
2167	IEC62321	201.9		0.51	
2169	IEC62321	179.6464		-1.07	
2172	IEC62321	194.114		-0.05	
2179	EPA3052/6010C	168.40		-1.87	
2182	EPA3050B	186.6		-0.58	
2184	in house	193.34		-0.10	
2187	EPA3052/6010C	212.5		1.26	
2189	in house	185.7		-0.64	
2190	in house	160		-2.47	
2192	EPA3052/6010C	222.1		1.94	
2194	in house	200.234		0.39	
2196	in house	193.16		-0.11	
2197		-----		-----	
2199	EPA3052	196		0.09	
2202	IEC62321	201.9		0.51	
2210	in house	182.7		-0.86	
2211		-----		-----	
2212		-----		-----	
2214	in house	176		-1.33	
2215	EPA3051	193.696		-0.07	
2217	IEC62321	174		-1.47	
2310	EPA3052	184.9		-0.70	
2312	EPA3051A	171.47		-1.65	
2350	IEC62321	199.0		0.30	
2361	EPA3052	189.3		-0.39	
2362	EPA3052	191		-0.27	
2363	IEC62321	199.97		0.37	
2365	EPA3052	188.0		-0.48	
2366	EPA3052	192.4		-0.17	
2369	EPA3052	196.073		0.09	
2370	EPA3052	193		-0.12	
2372	EPA3052	195.2		0.03	
2375	IEC62321	202.98		0.58	
2379	IEC62321	191.1		-0.26	
2380		-----		-----	
2385	in house	201		0.44	
3100	EPA3052	195.0		0.02	
3104	EPA3051	193.2707		-0.10	
3106	in house	241.5		3.32	
3107	EPA3052	249		3.85	
3110	EPA3052	185		-0.69	
3111	in house	204.2		0.67	
3113		-----		-----	
3124	EPA3052	185		-0.69	
3126	in house	196		0.09	

3127	DIN22022-3	201.53		0.48			
3134	in house	228.9		2.42			
3143	F2617	162.0		-2.32			
3153	EPA3052	199.0		0.30			
3154	DIN1122	152.3	C	-3.01	First reported 139.1		
3159	EPA3051	195.9		0.08			
3160	in house	197.16		0.17			
3163	in house	141.4	DG(0.05)	-3.79			
3166	in house	149.6		-3.20			
3169	EPA3052	210.15		1.09			
3172	EN1122	176.1		-1.32			
3180	EN1122	225.3		2.17			
3182	IEC62321	217.90		1.64			
3185	IEC62321	195.6		0.06			
3191	in house	187.4		-0.52			
3192	-----	-----		-----			
3199	EPA3051	216		1.51			
3200	in house	209.03		1.01			
3209	in house	212.5		1.26			
3213	EPA3052/6020A/IEC62321	180.44		-1.02			
3218	IEC111-95/CDV	190.4		-0.31			
3225	in house	184.6		-0.72			
3231	EPA3051	214.9		1.43			
3236	in house	109.26	G(0.05)	-6.07			
3239	in house	199.62		0.35			
3243	in house	169.8		-1.77			
3248	IEC62321	133	DG(0.05)	-4.38			
	normality	not OK			<u>Only AAS</u>	<u>Only ICP</u>	<u>Only XRF</u>
	n	84			OK	OK	OK
	outliers	4			10	71	4
	mean (n)	194.75			194.28	195.16	162.45
	st.dev. (n)	17.439			22.309	15.039	21.793
	R(calc.)	48.83			62.46	42.11	61.02
	R(Horwitz)	39.46			39.38	39.53	33.83



## Determination of total Chromium as Cr on sample #0842; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EPA3051	1.0765		----	
310		----		----	
358	in house	1.47		----	
390		----		----	
452	in house	1.87		----	
453		----		----	
551	IEC	4.7		----	
607	EPA3051A	nd		----	
622	EN1122	1.1331		----	
632	EPA3052	nd		----	
840	EPA3051/52	<2		----	
1051	in house	<5		----	
1121	in house	0.83		----	
1126		----		----	
1800	in house	<10		----	
1842		----		----	
2117	DIN EN ISO17254	0.90		----	
2129	in house	0.85		----	
2131	in house	1.53		----	
2132	in house	8.45	C	----	First reported 9.8, false positive
2139	EPA3052	<5.0		----	
2146		----		----	
2152	in house	<5		----	
2153	EPA3051	<12		----	
2156	IEC62321	2.2		----	
2160	in house	<24		----	
2161	EN1122B	0.4		----	
2167	IEC62321	1.21		----	
2169	IEC62321	<10		----	
2172	IEC62321	nd		----	
2179	EPA3052/6010C	<3		----	
2182	EPA3050B	<5		----	
2184	in house	1.00		----	
2187	EPA3052/6010C	<2		----	
2189	in house	<10		----	
2190	in house	nd		----	
2192	EPA3052/6010C	nd		----	
2194	in house	1.073		----	
2196	in house	<2		----	
2197		----		----	
2199	EPA3052	6.6		----	False positive
2202	IEC62321	nd		----	
2210	in house	nd		----	
2211		----		----	
2212		----		----	
2214	in house	5		----	
2215	EPA3051	<2		----	
2217	IEC62321	3.4		----	
2310	EPA3052	<2		----	
2312	EPA3051A	<2.0		----	
2350	IEC62321	nd		----	
2361	EPA3052	<5		----	
2362	EPA3052	<3		----	
2363	IEC62321	2.35		----	
2365	EPA3052	nd		----	
2366	EPA3052	nd		----	
2369	EPA3052	<2		----	
2370	EPA3052	nd		----	
2372	EPA3052	nd		----	
2375	IEC62321	<5		----	
2379	IEC62321	nd		----	
2380		----		----	
2385	in house	<2		----	
3100	EPA3052	nd		----	
3104	EPA3051	<50		----	
3106	in house	nd		----	
3107	EPA3052	<5		----	
3110	EPA3052	<5		----	
3111	in house	0.62		----	
3113		----		----	
3124	EPA3052	2.28		----	
3126	in house	0.77		----	

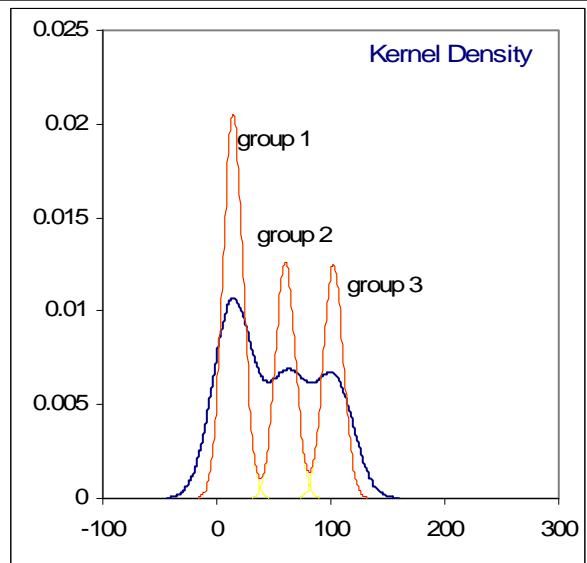
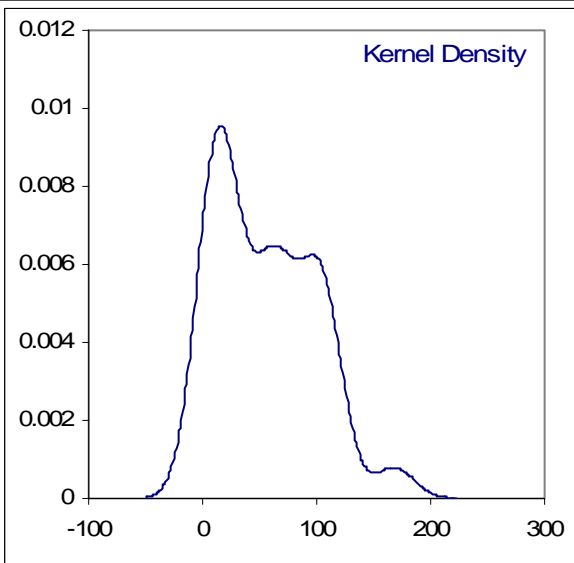
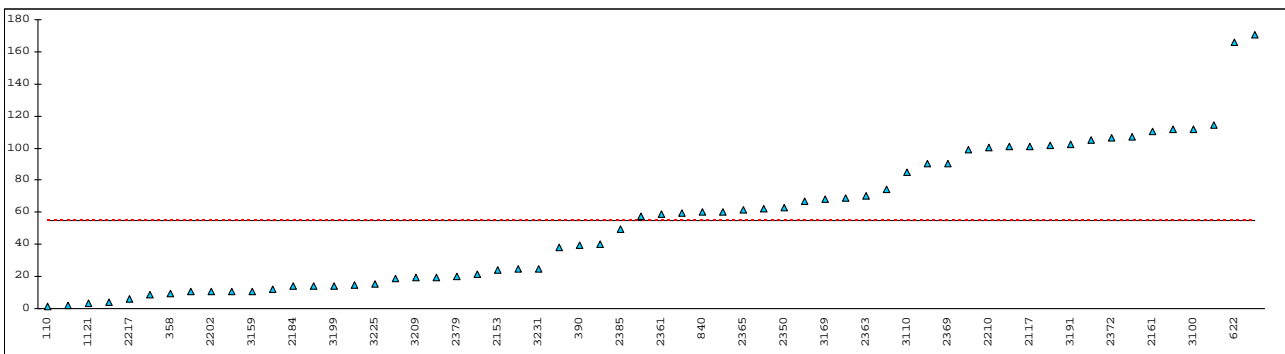
3127	DIN22022-3	<15		----	
3134	in house	<7		----	
3143	F2617	2.1		----	
3153	EPA3052	<2		----	
3154		----		----	
3159	EPA3051	<2		----	
3160	in house	<3		----	
3163	in house	1.6		----	
3166	in house	<10		----	
3169	EPA3052	nd	C	----	First reported 23.157
3172	EN1122	<10		----	
3180	EN1122	1.5	C	----	First reported 13
3182	EPA3052	nd		----	
3185	IEC62321	<2		----	
3191	in house	<5		----	
3192		----		----	
3199	EPA3051	<2.47		----	
3200	in house	<2.0		----	
3209	in house	2.5		----	
3213	EPA3052/6020A	0.94		----	
3218	IEC111-95/CDV	<2		----	
3225	in house	<2.0		----	
3231	EPA3051	<10		----	
3236	in house	0.408		----	
3239	in house	<2.0		----	
3243	in house	3.75		----	
3248	IEC62321	<5		----	
	normality	n.a.			
	n	23			
	outliers	0			
	mean (n)	<5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	unknown			

Determination of Hexavalent Chromium as Cr<sup>6+</sup> on sample #0841; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	IEC62321C	1.6615		----	
310		----		----	
358	EPA3060A/7196A	9.12		----	
390	EPA3060A/7196A	39.5		----	
452		----		----	
453		----		----	
551	IEC	38.0		----	
607	EPA3060A/7196A	60.54		----	
622	in house	165.93		----	
632		----		----	
840	EPA3060A/7196A	60.076		----	
1051		----		----	
1121	in house	3.08		----	
1126		----		----	
1800		----		----	
1842		----		----	
2117	IEC62321C	101		----	
2129		----		----	
2131	in house	1.77		----	
2132		----		----	
2139	EPA3060A	99.3		----	
2146		----		----	
2152		----		----	
2153	in house	24.42		----	
2156	IEC62321	10.6		----	
2160		----		----	
2161	EPA3060A	110.3		----	
2167	IEC62321	68.9		----	
2169		----		----	
2172	IEC62321	114.379	C	----	First reported 8.602; corrected result after reduction into powder
2179	in house	24.51	C	----	First reported 7.30
2182	EPA3060UV	40.2		----	
2184	EPA3060A	13.76		----	
2187	EPA3060A/7196	59.78		----	
2189	IEC62321	61.9		----	
2190	EPA3060A	14.5		----	
2192		----		----	
2194		----		----	
2196	EPA3060A/7196A	19.28		----	
2197		----		----	
2199	EPA3060A	66.8		----	
2202	in house	10.7	C	----	First reported 5.6
2210	in house	100.2		----	
2211		----		----	
2212		----		----	
2214		----		----	
2215	EPA3060	74.192		----	
2217	IEC62321	5.7		----	
2310		----		----	
2312	EPA3060A/7196A	8.68		----	
2350	IEC62321	63.2163		----	
2361	EPA3060A	59.0		----	
2362	IEC62321	101		----	
2363	EPA3060	70.5		----	
2365	EPA3060	61.8		----	
2366	EPA3060	57.6		----	
2369	EPA3060A	90.1002		----	
2370	EPA3060	105		----	
2372	EPA3060	106.6		----	
2375	IEC62321	10.82		----	
2379	IEC62321	20.4		----	
2380		----		----	
2385	in house	49.5		----	
3100	IEC62321	112.0		----	
3104		----		----	
3106		----		----	
3107		----		----	
3110	EPA3060A/7196A	85		----	
3111		----		----	
3113		----		----	
3124		----		----	
3126		----		----	

3127		----		----	
3134		----		----	
3143		----		----	
3153	IEC62321	112.0		----	
3154		----		----	
3159	EPA3060	10.997		----	
3160		----		----	
3163		----		----	
3166	EPA3060A/7199	67	C	----	First reported 3.9843; corrected result after pulverizing into fine powder
3169	IEC62321	68.386		----	
3172		----		----	
3180	EPA3060A/7196A	12	C	----	First reported 7.5
3182	IEC62321	21.53		----	
3185	IEC62321	106.9		----	
3191	IEC62321	102.069		----	
3192		----		----	
3199	EPA7196A	14.37		----	
3200		----		----	
3209	in house	19.14		----	
3213	EPA3060A	90.02		----	
3218	IEC111-95CDV	101.6		----	
3225	EPA3060A	15.71		----	
3231	IEC62321	24.92		----	
3236		----		----	
3239	in house	170.76		----	
3243	EPA3060A	14.25		----	
3248	IEC62321	18.7		----	

		<u>Group 1</u>	<u>Group 2</u>	<u>Group 3</u>	<u>Group 4</u>
normality	(not OK)	OK	not OK	OK	n.a.
n	(60)	25	17	16	2
outliers	(0)	0	0	0	0
mean (n)	(56.19)	14.74	60.52	102.34	168.35
st.dev. (n)	(42.533)	8.331	9.678	8.411	n.a.
R(calc.)	(119.09)	23.33	27.10	23.55	n.a.
R(Horwitz)	(unknown)	4.40	14.62	22.84	n.a.



Determination of Hexavalent Chromium as Cr<sup>6+</sup> on sample #0842; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
110	IEC62321C	0.046		----	
310		----		----	
358	EPA3060A/7196A	<1		----	
390	EPA3060A/7196A	<1		----	
452		----		----	
453		----		----	
551	IEC	nd		----	
607	EPA3060A/7196A	nd		----	
622	in house	<1		----	
632	EPA3052	nd		----	
840	EPA3060A/7196A	<2		----	
1051		----		----	
1121		----		----	
1126		----		----	
1800		----		----	
1842		----		----	
2117	IEC62321C	0.43		----	
2129		----		----	
2131	in house	nd		----	
2132		----		----	
2139	EPA3060A	<5.0		----	
2146		----		----	
2152		----		----	
2153	in house	<2		----	
2156	IEC62321	0.5		----	
2160		----		----	
2161	EPA3060A	0.4		----	
2167	IEC62321	1.33		----	
2169		----		----	
2172	IEC62321	nd		----	
2179	in house	<0.3		----	
2182	EPA3060	<1		----	
2184	EPA3060A	<1		----	
2187	EPA3060A/7196	<2		----	
2189	IEC62321	<5		----	
2190	EPA3060A	nd		----	
2192		----		----	
2194		----		----	
2196	EPA3060A/7196A	<1		----	
2197		----		----	
2199	EPA3060A	<2		----	
2202	in house	nd		----	
2210	in house	nd		----	
2211		----		----	
2212		----		----	
2214		----		----	
2215	EPA3051	<2		----	
2217	IEC62321	<1		----	
2310		----		----	
2312	EPA3060A/7196A	<2.0		----	
2350	IEC62321	nd		----	
2361	EPA3060A	<5		----	
2362	IEC62321	<3		----	
2363	EPA3060	<2		----	
2365	EPA3060	nd		----	
2366	EPA3060	nd		----	
2369	EPA3060A	<2		----	
2370	EPA3060	nd		----	
2372	EPA3060	nd		----	
2375	IEC62321	<5		----	
2379	IEC62321	nd		----	
2380		----		----	
2385	in house	<2		----	
3100	IEC62321	nd		----	
3104		----		----	
3106		----		----	
3107		----		----	
3110	EPA3060A/7196A	<5		----	
3111		----		----	
3113		----		----	
3124		----		----	
3126		----		----	

3127		----	----
3134		----	----
3143		----	----
3153	IEC62321	<1	----
3154		----	----
3159	EPA3060	<5	----
3160		----	----
3163		----	----
3166	in house	<0.03	----
3169	IEC62321	nd	----
3172		----	----
3180		----	----
3182	EPA3052	nd	----
3185	IEC62321	<1	----
3191	IEC62321	<5	----
3192		----	----
3199		----	----
3200		----	----
3209	in house	0.08	----
3213	EPA3060A	0.36	----
3218	IEC111-95CDV	<1	----
3225	EPA3060A	<2.0	----
3231	IEC62321	<10	----
3236		----	----
3239	in house	<2.0	----
3243	EPA3060A	0.34	----
3248	IEC62321	<1	----
	normality	n.a.	
	n	8	
	outliers	0	
	mean (n)	<2	
	st.dev. (n)	n.a.	
	R(calc.)	n.a.	
	R(Horwitz)	Unknown	

**APPENDIX 2****Analytical details regarding total Cadmium determination**

Lab	Analysis method	Technique to release the metals	Acid / acid mixture was used	Solid/liquid ratio was used	Technique to detect & quantify	Corrected for rec.
110	EPA3051	microwave	HNO3	0.25/10	ICP	yes
310	EN1122	---	---	---	---	---
358	in house	microwave	HNO3	0.3/10	ICP	no
390	EPA3052/6020A	---	---	---	---	---
452	in house	microwave	---	1/100	ICP	no
453	---	---	---	---	---	---
551	IEC	acid digestion with microwave	HNO3	0.25/10	ICP	no
607	EPA3051A	microwave	HNO3	1/40	ICP	no
622	EN1122	acid digestion	HNO3,H2SO4,H2O2	0.5/50	ICP	no
632	EPA3052	microwave digestion	HNO3,HF	0.25/12	ICP-OES	no
840	EPA3051/3052A	microwave	HNO3,H2O2	---	ICP-OES	no
1051	EN1122	acid digestion	H2SO4	0.05	AAS	no
1121	in house	microwave digestion	HNO3,H2SO4,HCL	0.5/50	ICP-MS	no
1126	in house	---	---	---	XRF	---
1800	in house	microwave digestion	HNO3	0.15/3	ICP-AES	no
1842	---	---	---	---	---	---
2117	DIN EN ISO17294	microwave digestion	HNO3	0.2/10	ICP-MS	no
2129	in house	acid digestion by microwave	HNO3	0.2/6	ICP-MS,ICP-OES	no
2131	in house	microwave	HNO3,H2O2	0.2/7	ICP-MS	no
2132	in house	microwave digestion	8 ml HNO3+2 ml H2O2	0.1/10	ICP-OES	no
2139	EN1122	acid digestion	H2SO4, HNO3	1/100	ICP	no
2146	EN1122	acid digestion	H2SO4,H2O2	---	AAS	no
2152	in house	microwave	HNO3	10 ml	ICP-OES	no
2153	EN1122	hotplate digestion	H2SO4,H2O2	0.5/10	ICP-OES	no
2156	IEC62321	---	---	---	---	---
2160	in house	other	---	---	edXRF	no
2161	Base on EN1122B	acid decomposition	HNO3,H2SO4,HCl	2.5/100	ICP-AES	no
2167	IEC62321/11/54/CDV	acid digestion using microwave	HNO3	0.2/3	ICP-MS	no
2169	IEC62321	microwave	HNO3	0.05/5	ICP-MS	no
2172	IEC62321	microwave digestion	HNO3	---	ICP-AES	no
2179	EPA3052/6010C	acid digestion with microwave	HNO3(6ml),H2O2(2ml),HF(1ml)	---	ICP-AES	no
2182	EN1122	acid digestion	H2SO4,H2O2	0.5/10	ICP	no
2184	EN1122	acid digestion	H2SO4,H2O2	0.5/100	AAS	no
2187	EPA3052/6010C	microwave	HNO3 (65-68%)	0.1/8	ICP	no
2189	in house	acid digestion	HNO3,HClO4	1/20	ICP	no
2190	in house	microwave	HNO3,H2O2,HF	0.2/50	ICP-AES	no
2192	EPA30526010C	microwave digestion	HNO3	---	ICP-OES	no
2194	in house	microwave acid digestion	HNO3	0.1/10	ICP-OES	no
2196	EN1122B	acid digestion	HNO3	0.5/100	AAS, ICP	no
2197	in house	---	---	---	AAS	---
2199	EPA3052	microwave	HNO3,HCl	1/5	ICP	no
2202	IEC62321	acid digestion	H2SO4,HNO3, H2O2	0.25/2.5	ICP-OES	no
2210	EN1122A	acid digestion	HNO3,H2SO4,H2O2	0.5/20	ICP-OES	no
2211	---	---	---	---	---	---
2212	---	---	---	---	---	---
2214	in house	microwave	---	---	ICP	yes
2215	EPA3051	microwave	HNO3,H2O2	1/125	ICP	no
2217	IEC62321	microwave	HNO3,H2O2	6/100	AAS ETA	no
2310	EPA3052	acid digestion with microwave	HNO3,H2O2	1/100	ICP	no
2312	EPA3051A	microwave digestion	HNO3	1/1000	ICP-MS	no
2350	IEC62321	microwave	HNO3	0.2/25	ICP-OES	no
2361	EPA3052	microwave	HNO3	0.4/100	ICP	no
2362	EPA3052	microwave	HNO3,H2O2	2.5/100	ICP-OES	no
2363	IEC62321	microwave digestion	HNO3 (9ml),HCl (1ml)	0.25/25	ICP-OES	no
2365	EPA3052	microwave	HNO3(6.5ml),HF(0.25ml),HClO4(0.5ml)	1/125	ICP-OES	no
2366	EPA3052	microwave	HNO3	0.1/10	ICP	no
2369	EPA3052	microwave	HNO3	0.25/25	ICP	no
2370	EPA3052	acid digestion with microwave	HNO3 (10ml),HF (1ml)	2/100	ICP-AES	no
2372	EPA3052	microwave	HNO3,HF,HCl	0.2/13.5	ICP	no
2375	IEC62321/111/116/FDIS	microwave digestion	HNO3,H2O2, HBF4,H2O	1/100	ICP-OES	no
2379	IEC62321/111/116/FDIS	microwave	HNO3,H2O2	0.1/10	ICP	no
2380	in house	microwave	HNO3,H2O2	(0.1-0.2)/6	ICP-OES	no
2385	in house	microwave	HNO3,H2O2	0.1-0.2/6	ICP-OES	no
3100	EPA3052	microwave acid digestion	HNO3	0.1/25	ICP	no
3104	EN1122	acid digestion	H2SO4,H2O2	0.5/10	AAS	no
3106	in house	---	---	---	XRF	---

3107	EPA3052	microwave digestion	HNO3 (9M)	0.2/15	ICP-OES	no
3110	EN1122B	acid digestion	H2SO4,H2O2	---	AAS, ICP	no
3111	in house	microwave combustion	Nitric	0.2/50	ICP	no
3113	in house	acid digestion	HNO3(10ml),H2SO4(2ml)	1gr/12 ml	AAS	no
3124	EPA3052	microwave acid digestion	HNO3 (8 ml),HF (1 ml),HCL (2 ml)	2.27/100	ICP-MS	no
3126	in house	---	---	---	INAA	---
3127	DIN22022-5	microwave acid digestion	HNO3	4/100	AAS	no
3134	in house	microwave acid digestion	HNO3,H2SO4	---	AAS	no
3143	---	---	---	---	---	---
3153	EPA3052	microwave acid digestion	HNO3	2/100	ICP	no
3154	DIN1122	microwave digestion	HNO3 (6 ml),H2O2 (2 ml)	0.2/8	AAS	no
3159	EPA3051	microwave acid digestion	HNO3	0.1/25	ICP-AES	no
3160	in house	microwave digestion	HNO3 (7ml),H2O2 (1ml),HCL(2ml)	0.25/10	ICP-AES	no
3163	in house	---	---	---	XRF	no
3166	in house	---	---	---	XRF	no
3169	EPA3052	microwave	HNO3	0.1/5	ICP	no
3172	EN1122	microwave acid digestion	HNO3,H2O2	100mg/10ml	ICP	no
3180	EN1122	microwave	HNO3	200mg/10ml	AAS	no
3182	IEC62321EPA3052	microwave	HNO3,H2O2,HCL	0.1/25	ICP-OES	no
3185	IEC62321/111/95/CDV	microwave acid digestion	HNO3	0.15/25	ICP-OES	no
3191	in house	microwave	HNO3, HBF4	0.2/50	ICP	no
3192	EN1122	microwave	HNO3	---	ICP-OES	no
3199	EPA3051	microwave	HNO3,H2O2	0.4/100	ICP	no
3200	in house	microwave digestion	HNO3	2/100	ICP	no
3209	in house	microwave	HNO3	2/100	ICP-OES	no
3213	EPA3052/6020A/IEC62321	microwave	HNO3,H2O2	0.2/8	ICP-MS	no
3218	IEC111-95CDV	microwave	HNO3,H2O2,HF	0.2/50	ICP-XRF	yes
3225	in house	microwave	HNO3	0.8/100	ICP	yes
3231	EPA3051	microwave	HNO3,H2O2	1.875/100	ICP	yes
3236	in house	ashing & acid digestion	HNO3	2gr for ashing	---	no
3239	in house	microwave acid digestion	HNO3,HCL	0.1/25	ICP-OES	no
3243	in house	microwave	HNO3,HCL	0.1/25	ICP-OES	no
3248	IEC62321	microwave	HNO3	2.5/100	AAS	no

## Analytical details regarding total Lead determination

Lab	Analysis method	Technique to release the metals	Acid / acid mixture was used	Solid/liquid ratio was used	Techn. to detect & quantify	Corrected for rec.
110	EPA3051	microwave	HNO3	0.25/10	ICP	yes
310	subcontracted within company	---	---	---	---	---
358	in house PBBBB	microwave	HNO3	0.3/10	ICP	no
390	EPA3052/6020A	---	---	---	---	---
452	in house	microwave	---	1/100	ICP	no
453	---	---	---	---	---	---
551	IEC	acid digestion with microwave	HNO3	0.25/10	ICP	no
607	EPA3051A	microwave	HNO3	1/40	ICP	no
622	in house	combustion	HNO3,H2SO4,H2O2	0.5/50	ICP	no
632	EPA3052	microwave digestion	HNO3,HF	0.25/12	ICP-OES	no
840	EPA3051/3052A	microwave	HNO3,H2O2	---	ICP-OES	no
1051	in house	dry ashing	HNO3	0.03	ICP	no
1121	in house	microwave digestion	HNO3,H2SO4,HCL	0.5/50	ICP-MS	no
1126	---	---	---	---	---	---
1800	in house	microwave digestion	HNO3	0.15/3	ICP-AES	no
1842	---	---	---	---	---	---
2117	DIN EN ISO17294	microwave digestion	HNO3	0.2/10	ICP-MS	no
2129	in house	acid digestion by microwave	HNO3	0.2/6	ICP-MS,ICP-OES	no
2131	in house	microwave	HNO3,H2O2	0.2/7	ICP-MS	no
2132	in house	microwave digestion	HNO3(8ml),H2O2(2ml)	0.1/10	ICP-OES	no
2139	EPA3052	microwave	---	1/200	ICP	no
2146	---	---	---	---	---	---
2152	in house	microwave	HNO3	10 ml	ICP-OES	no
2153	EPA3051	microwave digestion	HNO3	0.1/10	ICP-OES	no
2156	IEC62321	---	---	---	---	---
2160	in house	other	---	---	edXRF	no
2161	Base on EN1122B	acid decomposition	HNO3,H2SO4,HCl	2.5/100	ICP-AES, ET-AAS	no
2167	IEC62321/11/54/CDV	acid digestion using microwave	HNO3	0.2/3	ICP-MS	no
2169	IEC62321	microwave	HNO3	0.05/5	ICP-MS	no
2172	IEC62321	microwave digestion	HNO3	---	ICP-AES	no
2179	EPA3052	acid digestion with microwave	HNO3 (6 ml),H2O2 (2 ml),HF (1 ml)	---	ICP-AES	no
2182	EPA3050B	acid digestion + ashing	HNO3	0.25/10	ICP	no
2184	in house	microwave	HNO3, H2O2	1/100	ICP-OES	no
2187	EPA3052/6010C	microwave	HNO3 (65-68%)	0.1/8	ICP	no
2189	in house	acid digestion	HNO3,HClO4	1/20	ICP	no
2190	in house	microwave	HNO3,H2O2,HF	0.2/50	ICP-AES	no
2192	EPA30526010C	microwave digestion	HNO3	---	ICP-OES	no
2194	ICP/OES	microwave acid digestion	HNO3	0.1/10	ICP-OES	no
2196	in house	acid digestion	HNO3	0.2/25	AAS	no
2197	in house	---	---	---	ICP-OES	---
2199	EPA3052	microwave	HNO3,HCl	1/5	ICP	no
2202	IEC62321	acid digestion	H2So4,HNO3,H2O2	0.25/2.5	ICP-OES	no
2210	in house	acid digestion	HNO3,H2SO4	0.5/16	ICP-OES	no
2211	in house	microwave	HNO3	0.2/7.5	AAS	no
2212	in house	microwave	nit. Acid	4/100	AAS	no
2214	in house	microwave	---	---	ICP	yes
2215	EPA3051	microwave	HNO3, H2O2 (30%)	1/125	ICP	no
2217	IEC62321	microwave	HNO3,H2O2	6/100	AAS ETA	no
2310	EPA3052	acid digestion with microwave	HNO3,H2O2	1/100	ICP	no
2312	EPA3051A	microwave digestion	HNO3	1/1000	ICP-MS	no
2350	IEC62321	microwave	HNO3	0.2/25	ICP-OES	no
2361	EPA3052	microwave	HNO3	0.4/100	ICP	no
2362	EPA3052	microwave	HNO3,H2O2	2.5/100	ICP-OES	no
2363	IEC62321	microwave digestion	HNO3 (9ml),HCl (1ml)	0.25/25	ICP-OES	no
2365	EPA3052	microwave	HNO3(6.5ml),HF(0.25ml),HClO4(0.5ml)	1/125	ICP-OES	no
2366	EPA3052	microwave	HNO3	0.1/10	ICP	no
2369	EPA3052	microwave	HNO3	0.25/25	ICP	no
2370	EPA3052	acid digestion with microwave	HNO3 (10ml),HF (1ml)	2/100	ICP-AES	no
2372	EPA3052	microwave	HNO3,HF,HCl	0.2/13.5	ICP	no
2375	IEC62321/111/116/FDIS	microwave digestion	HNO3,H2O2,HBF4,H2O	1/100	ICP-OES	no
2379	IEC62321	microwave	HNO3,H2O2	0.1/10	ICP	no
2380	in house	microwave	HNO3,H2O2	(0.1-0.2)/6	ICP-OES	no
2385	ICP-OES	microwave	HNO3,H2O2	0.1-0.2/6	ICP-OES	no
3100	EPA3052	microwave acid digestion	HNO3	0.1/25	ICP	no
3104	EPA3051	microwave digestion	HNO3, H2O2	0.2/8	AAS	no
3106	in house	---	---	---	XRF	---
3107	EPA3052	microwave digestion	HNO3 (9M)	0.2/15	ICP-OES	no

3110	EPA3052	microwave	HNO3, H2O2	---	AAS, ICP	no
3111	in house	microwave combustion	HNO3	0.2/50	ICP	no
3113	in house	acid digestion	HNO3(10ml),H2SO4(2ml)	1gr/12 ml	AAS	no
3124	EPA3052	microwave acid digestion	HNO3 (8 ml),HF (1 ml),HCL (2 ml)	2.27/100	ICP-MS	no
3126	in house	---	---	---	INAA	---
3127	DIN22022-5	microwave acid digestion	HNO3	4/100	AAS	no
3134	---	---	---	---	---	---
3143	---	---	---	---	---	---
3153	EPA3052	microwave acid digestion	HNO3	2/100	ICP	no
3154	DIN1122	microwave digestion	HNO3 (6 ml),H2O2 (2 ml)	0.2/8	ICP	no
3159	EPA3051	microwave acid digestion	HNO3	0.1/25	ICP-AES	no
3160	in house	microwave digestion	HNO3 (7ml),H2O2 (1ml),HCL(2ml)	0.25/10	ICP-AES	no
3163	in house	---	---	---	XRF	no
3166	in house	---	---	---	XRF	no
3169	EPA3052	microwave	HNO3	0.1/5	ICP	no
3172	EN1122	microwave acid digestion	HNO3,H2O2	100mg/10ml	ICP	no
3180	EN1122	microwave	HNO3	200mg/10ml	AAS	no
3182	IEC62321/EPA3052	microwave digestion	HNO3,HCL,H2O2	0.1/25	ICP-OES	no
3185	IEC62321/111/95/CDV	microwave acid digestion	HNO3	0.15/25	ICP-OES	no
3191	in house	microwave	HNO3,HBF4	0.2/50	ICP	no
3192	in house	microwave	HNO3	---	ICP-OES	no
3199	EPA3051	microwave	HNO3,H2O2	0.4/100	ICP	no
3200	in house	microwave digestion	HNO3	2/100	ICP	no
3209	in house	microwave	HNO3	2/100	ICP-OES	no
3213	EPA3052/6020A/IEC62321	microwave	HNO3,H2O2	0.2/8	ICP-MS	no
3218	IEC111-95CDV	microwave	HNO3,H2O2,HF	0.2/50	ICP-XRF	yes
3225	in house	microwave	HNO3	0.8/100	ICP	yes
3231	EPA3051	microwave	HNO3,H2O2	1.875/100	ICP	yes
3236	in house	ashing & acid digestion	HNO3	2gr for ashing	---	no
3239	in house	microwave acid digestion	HNO3,HCL	0.1/25	ICP-OES	no
3243	in house	microwave	HNO3,HCL	0.1/25	ICP-OES	no
3248	IEC62321	microwave	HNO3	2.5/100	AAS	no

## Analytical details regarding total Chromium determination

Lab	Analysis method	Technique to release the metals	Acid / acid mixture was used	Solid/liquid ratio was used	Techn. to detect & quantify	Corrected for rec.
110	EPA3051	microwave	HNO3	0.25/10	ICP	yes
310	subcontracted within company	---	---	---	---	---
358	in house	microwave	HNO3	0.3/10	ICP	no
390	EPA3052/6020A	---	---	---	---	---
452	in house	microwave	---	1/100	ICP	no
453	---	---	---	---	---	---
551	IEC	acid digestion with microwave	HNO3	0.25/10	ICP	no
607	EPA3051A	microwave	HNO3	1/40	ICP	no
622	EN1122	combustion	HNO3,H2SO4,H2O2	0.5/50	ICP	no
632	EPA3052	microwave digestion	HNO3,HF	0.25/12	ICP-OES	no
840	EPA3051/3052A	microwave	HNO3,H2O2	---	ICP-OES	no
1051	in house	dry ashing	HNO3	0.03	ICP	no
1121	in house	microwave digestion	HNO3,H2SO4,HCL	0.5/50	ICP-MS	no
1126	---	---	---	---	---	---
1800	in house	microwave digestion	HNO3	0.15/3	ICP-AES	no
1842	---	---	---	---	---	---
2117	DIN EN ISO11885	microwave digestion	HNO3	0.2/10	ICP-MS	no
2129	in house	acid digestion by microwave	HNO3	0.2/6	ICP-MS,ICP-OES	no
2131	in house	microwave	HNO3,H2O2	0.2/7	ICP-MS	no
2132	in house	microwave digestion	HNO3(8ml),H2O2(2ml)	0.1/10	ICP-OES	no
2139	EPA3052	microwave	---	1/200	ICP	no
2146	---	---	---	---	---	---
2152	in house	microwave	HNO3	10 ml	ICP-OES	no
2153	EPA3051	microwave digestion	nitric acid	0.1/10	ICP-OES	no
2156	IEC62321	---	---	---	---	---
2160	in house	other	---	---	edXRF	no
2161	Base on EN1122B	acid decomposition	HNO3,H2SO4,HCl	2.5/100	ICP-AES, ET-AAS	no
2167	IEC62321/11/54/CDV	acid digestion using microwave	HNO3	0.2/3	ICP-MS	no
2169	IEC62321	microwave	HNO3	0.05/5	ICP-MS	no
2172	IEC62321	microwave digestion	HNO3	---	ICP-AES	no
2179	EPA3052	acid digestion with microwave	HNO3 (6 ml),H2O2 (2 ml),HF (1 ml)	---	ICP-AES	no
2182	EPA3050B	acid digestion + ashing	HNO3	0.25/10	ICP	no
2184	in house	microwave	HNO3, H2O2	1/100	ICP-OES	no
2187	EPA3052/6010C	microwave	HNO3 (65-68%)	0.1/8	ICP	no
2189	in house	acid digestion	HNO3,HClO4	1/20	ICP	no
2190	in house	microwave	HNO3,H2O2,HF	0.2/50	ICP-AES	no
2192	EPA30526010C	microwave digestion	HNO3	---	ICP-OES	no
2194	ICP/OES	microwave acid digestion	HNO3	0.1/10	ICP-OES	no
2196	in house	acid digestion	HNO3	0.2/25	AAS	no
2197	in house	---	---	---	ICP-OES	---
2199	EPA3052	microwave	HNO3,HCl	1/5	ICP	no
2202	IEC62321	acid digestion	H2SO4,HNO3,H2O2	0.25/2.5	ICP-OES	no
2210	in house	acid digestion	HNO3,H2SO4	0.5/16	ICP-OES	no
2211	---	---	---	---	---	---
2212	---	---	---	---	---	---
2214	in house	microwave	---	---	ICP	yes
2215	EPA3051	microwave	HNO3,H2O2 (30%)	1/125	ICP	no
2217	IEC62321	microwave	HNO3,H2O2	6/100	AAS ETA	no
2310	EPA3052	acid digestion with microwave	HNO3,H2O2	1/100	ICP	no
2312	EPA3051A	microwave digestion	HNO3	1/1000	ICP-MS	no
2350	IEC62321	microwave	HNO3	0.2/25	ICP-OES	no
2361	EPA3052	microwave	HNO3	0.4/100	ICP	no
2362	EPA3052	microwave	HNO3,H2O2	2.5/100	ICP-OES	no
2363	IEC62321	microwave digestion	HNO3 (9ml),HCl (1ml)	0.25/25	ICP-OES	no
2365	EPA3052	microwave	HNO3(6.5ml),HF(0.25ml),HClO4(0.5ml)	1/125	ICP-OES	no
2366	EPA3052	microwave	HNO3	0.1/10	ICP	no
2369	EPA3052	microwave	HNO3	0.25/25	ICP	no
2370	EPA3052	acid digestion with microwave	HNO3 (10ml),HF (1ml)	2/100	ICP-AES	no
2372	EPA3052	microwave	HNO3,HF,HCl	0.2/13.5	ICP	no
2375	IEC62321/111/116/FDIS	microwave digestion	HNO3,H2O2,HBf4,H2O	1/100	ICP-OES	no
2379	IEC62321/111/116/FDIS	microwave	HNO3,H2O2	0.1/10	ICP	no
2380	in house	microwave	HNO3,H2O2	(0.1-0.2)/6	ICP-OES	no
2385	ICP-OES	microwave	HNO3,H2O2	0.1-0.2/6	ICP-OES	no
3100	EPA3052	microwave acid digestion	HNO3	0.1/25	ICP	no
3104	EPA3051	microwave digestion	HNO3, H2O2	0.2/8	AAS	no
3106	in house	---	---	---	XRF	---
3107	EPA3052	microwave digestion	HNO3 (9M)	0.2/15	ICP-OES	no

3110	EPA3052	microwave	HNO3, H2O2	---	AAS, ICP	no
3111	in house	microwave combustion	HNO3	0.2/50	ICP	no
3113	---	---	---	---	---	---
3124	EPA3052	microwave acid digestion	HNO3 (8 ml),HF (1 ml),HCL (2 ml)	2.27/100	ICP-MS	no
3126	---	---	---	---	---	---
3127	DIN22022-3	microwave acid digestion	HNO3 (65%)	4/100	AAS	no
3134	in house	microwave acid digestion	HNO3,H2SO4	---	AAS	no
3143	---	---	---	---	---	---
3153	EPA3052	microwave acid digestion	HNO3	2/100	ICP	no
3154	DIN1122	microwave digestion	HNO3 (6 ml),H2O2 (2 ml)	0.2/8	ICP	no
3159	EPA3051	microwave acid digestion	HNO3	0.1/25	ICP-AES	no
3160	in house	microwave digestion	HNO3 (7ml),H2O2 (1ml),HCL(2ml)	0.25/10	ICP-AES	no
3163	in house	---	---	---	XRF	no
3166	in house	---	---	---	XRF	no
3169	EPA3052	microwave	HNO3	0.1/5	ICP	no
3172	EN1122	microwave acid digestion	HNO3,H2O2	100mg/10ml	ICP	no
3180	EN1122	microwave	HNO3	200mg/10ml	AAS	no
3182	IEC62321/EPA3052	microwave digestion	HNO3,HCL,H2O2	0.1/25	ICP-OES	no
3185	IEC62321/111/95/CDV	microwave acid digestion	HNO3	0.15/25	ICP-OES	no
3191	in house	microwave	HNO3, HBF4	0.2/50	ICP	no
3192	---	---	---	---	---	---
3199	EPA3051	microwave	HNO3,H2O2	0.4/100	ICP	no
3200	in house	microwave digestion	HNO3	2/100	ICP	no
3209	in house	microwave	HNO3	2/100	ICP-OES	no
3213	EPA3052/6020A	microwave	HNO3,H2O2	0.2/8	ICP-MS	no
3218	IEC111-95CDV	microwave	HNO3,H2O2, HF	0.2/50	ICP-XRF	yes
3225	in house	microwave	HNO3	0.8/100	ICP	yes
3231	EPA3051	microwave	HNO3,H2O2	1.875/100	ICP	yes
3236	in house	ashing & acid digestion	HNO3	2gr for ashing	---	no
3239	in house	microwave acid digestion	HNO3,HCL	0.1/25	ICP-OES	no
3243	in house	microwave	HNO3,HCL	0.1/25	ICP-OES	no
3248	IEC62321	microwave	HNO3	2.5/100	AAS	no

## Analytical details regarding Hexavalent Chromium determination

Lab	Analysis method	Size of reduced sample	Solution	ratio g/mL	Extraction conditions	Corr for recov.	Other details
110	IEC62321C	<250µm	Follow IEC62321 Annex C 2nd ed	2.5/50	95°C @3hrs	yes	IEC62321C CR <sup>+</sup>
310		---	---	---	---	---	subcontracted within company
358	EPA3060A/7196A	manually cut	alkaline digestion sol.	1/50	1hr,95°C,water bath	no	extraction procedure inefficient, results too low
390	EPA3060A/7196A	---	---	---	---	---	---
452	in house	---	---	---	---	---	---
453	---	---	---	---	---	---	---
551	IEC	1.10mm	mgCl <sub>2</sub> /phosphate buffer	2.5/100	90-95°C @3hrs	no	---
607	EPA3060A/7196A	<1mm <sup>3</sup>	alkaline sol.,MgCl <sub>2</sub> & buffer phosphate	1/20	90-95°C @60min. Continuous stirring	no	---
622	in house	0.300mm	alkaline sol.,MgCl <sub>2</sub> & buffer phosphate	0.3/50	90-95°C @60min. Shaking waterbath	no	---
632	EPA3060A/7196A	<2x<2x<2	Na <sub>2</sub> CO <sub>3</sub> , NaOH, pH>12	2.5/50	90-95°C @60min.	no	rec.92.8%; LR=0.9990
840	EPA3060A/7196A	2x2x2	alkaline sol.	20g NaOH/30g Na <sub>2</sub> CO <sub>3</sub> /1L	90-95°C @60±5min. Continious stirring	no	ext. std., Analysis by UV/VIS
1051	---	---	---	---	---	---	---
1121	in house	<4mm	0.1m HCl	3/100	37°C @1hr	no	---
1126	---	---	---	---	---	---	---
1800	---	---	---	---	---	---	---
1842	---	---	---	---	---	---	---
2117	IEC62321C	1mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	2.5/50	90-95°C @3hrs	no	---
2129	---	---	---	---	---	---	---
2131	in house	---	---	2/40	40°C @1hr Ultra sonic	no	---
2132	---	---	---	---	---	---	---
2139	EPA3060A	Fine powder (<250µm)	NaOH, Na <sub>2</sub> CO <sub>3</sub> , MgCl <sub>2</sub> , Phosphate buffer	1/100	90-95°C @1hr, hot plate	no	no
2146	---	---	---	---	---	---	---
2152	---	---	---	---	---	---	---
2153	in house	5x5mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	0.5/50	90-95°C @3hrs waterbath	yes	---
2156	IEC62321	---	---	---	---	---	---
2160	in house	---	---	---	---	---	---
2161	Base on EPA SW-846 3060A	powder by frost shattering	alkali extraction	2.0/100	95°C @1hr	no	---
2167	IEC62321/11/54/CDV	1-2mm	---	1/50	90°C @1hr	no	---
2169	---	---	---	---	---	---	---
2172	IEC62321	1mm	alkaline digestion sol.	1/100	95°C @1hr waterbath	no	sample size effect Cr <sup>6+</sup> result
2179	in house	<500µm	NaOH, Na <sub>2</sub> CO <sub>3</sub> , buffer K <sub>2</sub> HPO <sub>4</sub> + KH <sub>2</sub> PO <sub>4</sub>	---	---	no	---
2182	EPA3060	<2x<2x<2mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	2.5/100	90-95°C @60min. Continuous stirring	no	UV/VIS spectrometer
2184	EPA3060A	2x2mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	5/100	95°C @60min.	no	Cr <sup>6+</sup> content measured by UV-VIS
2187	EPA3060A/7196	1mm diameter	NaOH (0.5M), Na <sub>2</sub> CO <sub>3</sub> (0.28M)	1/30	90°C @1hr shaking table	no	method EPA7196, buffer sol.: 0.5M K <sub>2</sub> HPO <sub>4</sub> +0.5M KH <sub>2</sub> PO <sub>4</sub> +0.4gr MgCl
2189	IEC62321/ICD chapter10	<30µm	NaOH (0.5M), Na <sub>2</sub> CO <sub>3</sub> (0.28M), MgCl <sub>2</sub>	1/10	90-95°C @60min. waterbath	no	UV-VIS measure @540nm
2190	EPA3060A	2x2x2mm	alkaline digestion	1.5/100	90-95°C @60min.	no	---
2192	---	---	---	---	---	---	---
2194	---	---	---	---	---	---	---
2196	EPA3060A/7196A	<500µm	alkaline digestion sol. (0.2M Na <sub>2</sub> CO <sub>3</sub> /0.5M NaOH)	1/100	90-95°C @60min.	no	no
2197	in house	---	---	---	ICP-OES	---	in house
2199	EPA3060A	cut to thin film	NaOH, Na <sub>2</sub> CO <sub>3</sub>	1/100	95°C @1hr	no	---
2202	in house	samples were not powdered	chlorobenzene, toluene, 1-mehtyl-2-pyrrolidinone	0.2/12	150°C @30min.	no	---
2210	in house	0.5mm	NaOH (0.5M), Na <sub>2</sub> CO <sub>3</sub> (0.28M), water, triton x100	0.5/50	90-95°C @90min	no	---
2211	---	---	---	---	---	---	---
2212	---	---	---	---	---	---	---
2214	---	---	---	---	---	---	---
2215	EPA3060	3x3mm	alkaline sol. (NaOH,NaCO <sub>3</sub> )	1/200	95°C @60min. waterbath	no	---
2217	IEC62321	3x3x2mm	NaOH (0.5M), NaCO <sub>3</sub> (0.28M)	10/100	90-95°C @60min.	no	spectrophotometer
2310	---	---	---	---	---	---	---
2312	EPA3060A/7196A	<5X5X5mm	NaOH, Na <sub>2</sub> CO <sub>3</sub> digestion sol.	1/100	90°C @1hr	no	---
2350	IEC62321	80-600µm freezing	NaOH, Na <sub>2</sub> CO <sub>3</sub>	2.5/50	90°C-95°C @3hrs	no	---

		mill			waterbath shaker		
2361	EPA3060A	2x2x2mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	1/100	95°C @1hr shaker	no	---
2362	IEC62321	250µm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	1/20	90°C @3hrs	no	---
2363	EPA3060	1x1mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	2.5/100	92.5°C @1hr	no	EPA3060
2365	EPA3060	1x1x1mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	2.5/100	90°C-95°C @60min.	no	no
2366	EPA3060	<1mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	2.5/50	90°C-95°C @60min.	no	---
2369	EPA3060A	<250x250x250µm	NaOH, Na <sub>2</sub> CO <sub>3</sub> , MgCl <sub>2</sub> , Triton x100	1.25/250	92.5°C @1hr (EPA3060A), 3hrs (IEC62321), continuous stirring in water bath	no	---
2370	EPA3060	<2x<2x<2mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	5/100	90°C-95°C @60min., continuous stirring	no	no
2372	EPA3060	<0.5mm	NaOH, Na <sub>2</sub> CO <sub>3</sub> , MgCl <sub>2</sub> , Phosphate buffer	2.5/50	90°C-95°C @60min.	no	no
2375	IEC62321/111/116/FDIS	0.25x0.25mm	NaOH, Na <sub>2</sub> CO <sub>3</sub> in H <sub>2</sub> O, Triton x	5/100	90°C-95°C @3hrs on hotplate with continuous stirring	no	---
2379	IEC62321/111/116/FDIS	1x1cm	alkaline extraction	1.5/50	90°C @3hrs waterbath	no	---
2380	in house	<0.5mm	NaOH (20g/l), Na <sub>2</sub> CO <sub>3</sub> (30g/l)	1/50	90°C-95°C @60min.	no	---
2385	UV-VIS	<0.5mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	1/50	90°C-95°C @1hr	no	
3100	IEC62321	<250µm	digestion sol. Surfactant decon-90	25/100	93°C @3hrs	no	no
3104	---	---	---	---	---	---	---
3106	---	---	---	---	---	---	---
3107	---	---	---	---	---	---	---
3110	EPA3060A/7196A	<(2X2X2)mm3	NaOH (0.5M), Na <sub>2</sub> CO <sub>3</sub> (0.28M), MgCl <sub>2</sub> phosphate buffer	---	90°C-95°C @60min. waterbath	no	---
3111	---	---	---	---	---	---	---
3113	---	---	---	---	---	---	---
3124	---	---	---	---	---	---	---
3126	---	---	---	---	---	---	---
3127	---	---	---	---	---	---	---
3134	---	---	---	---	---	---	---
3143	---	---	---	---	---	---	---
3153	IEC62321/111/95/CDV	250µm	alkaline digestion sol.	5/100	90°C-95°C @3hrs	no	no
3154	---	---	---	---	---	---	---
3159	EPA3060	5x5mm	NaOH, Na <sub>2</sub> CO <sub>3</sub> phosphate buffer	0.5gr/50ml dig. sol./1ml phosphate buff.	90°C-95°C @1hr shaking water bath	no	---
3160	---	---	---	---	---	---	---
3163	---	---	---	---	---	---	---
3166	EPA3060A/7199	0.1-0.3cm3	NaOH (2%), Na <sub>2</sub> CO <sub>3</sub> (3%), MgCl <sub>2</sub>	#0841 5/100, #0842 3/100	on hotplate refluxing and stirring for 1hr	no	---
3169	IEC62321/111/95/CDV	0.2mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	1/50	90°C-95°C @3hrs shaking water bath	no	the sample can't be group into powder
3172	---	---	---	---	---	---	---
3180	EPA3060A/7196A	2x2mm	ref. EPA3060A	2/50	90°C-95°C @60min.	no	---
3182	IEC62321/EPA3052	0.2x0.2mm	buffer sol.	2.5/50	90°C-95°C @1hr	no	---
3185	IEC62321/111/95/CDV	250µm	alkaline extraction sol.	1.25/50	90°C-95°C @3hrs continuous stirring	no	---
3191	IEC62321/111/95/CDV	<250µm	NaOH (0.5M), Na <sub>2</sub> CO <sub>3</sub> (0.28M)	0.4/50	90°C @3hrs waterbath	no	no
3192	---	---	---	---	---	---	---
3199	EPA7196A	10x2x2mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	1/100	94.0°C @1hr shake in water bath	no	0.3 g of MgCl <sub>2</sub> added to all samples/std prior to extraction
3200	---	---	---	---	---	---	---
3209	in house	1x1mm	NaOH (0.5M), Na <sub>2</sub> CO <sub>3</sub> (0.28M)	5/100	90°C-95°C @60min. shake in water bath	no	EPA3060A/7196A
3213	EPA3052/6020A/IEC62321ed1/FDIS	freezer mill	NaOH, Na <sub>2</sub> CO <sub>3</sub>	0.7/50	95°C @1hr heating and stirring	no	---
3218	IEC111-95CDV	250µm	alkaline dig.sol.:phosphate buff.;Triton x-100	2.5/50	95°C @3hrs	no	uv-vis spectrom. W.ith 2 cm cell and wavelength set at 540nm
3225	EPA3060A	500µm	NaOH (0.5M), Na <sub>2</sub> CO <sub>3</sub> (0.28M)	2.5/100	90°C @1hr hot plate	yes	nil
3231	IEC62321	5x5mm	NaOH, Na <sub>2</sub> CO <sub>3</sub> ,KH <sub>2</sub> PO <sub>4</sub>	0.99/100	90-95°C @60min. waterbath	yes	---
3236	---	---	---	---	---	---	---
3239	in house	---	NaOH (0.5M), Na <sub>2</sub> CO <sub>3</sub> (0.28M)	0.3/100	95°C @1hr heating	no	---
3243	EPA3060A	1-2mm	NaOH, Na <sub>2</sub> CO <sub>3</sub>	0.5/20	95°C @1hr	no	---
3248	IEC62321	1x1x1mm	dig.sol. (pH>11.5) (NaOH, Na <sub>2</sub> CO <sub>3</sub> , water)	2/100	90-95°C @60min.	no	---

**APPENDIX 3****List of number of participating laboratories per country**

<b>Number of laboratories</b>	<b>Country</b>
1 laboratory in	BANGLADESH
1 laboratory in	BRASIL
1 laboratory in	CANADA
2 laboratories in	DENMARK
2 laboratories in	FINLAND
1 laboratory in	FRANCE
8 laboratories in	GERMANY
1 laboratory in	GREECE
20 laboratories in	HONG KONG
1 laboratory in	HUNGARY
2 laboratories in	INDIA
1 laboratory in	INDONESIA
1 laboratory in	ISRAEL
2 laboratories in	ITALY
2 laboratories in	JAPAN
3 laboratories in	KOREA
2 laboratories in	MALAYSIA
16 laboratories in	P.R. of CHINA
2 laboratories in	PHILIPPINES
1 laboratory in	PORTUGAL
3 laboratories in	SINGAPORE
1 laboratory in	SOUTH KOREA
1 laboratory in	SPAIN
2 laboratories in	SWITZERLAND
2 laboratories in	TAIWAN R.O.C.
2 laboratories in	THAILAND
5 laboratories in	THE NETHERLANDS
2 laboratories in	TURKEY
4 laboratories in	U.S.A.
6 laboratories in	UNITED KINGDOM
1 laboratory in	VIETNAM

## APPENDIX 4

### Abbreviations:

C	= final result after checking of first reported suspect 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
n.a.	= not applicable

### Literature:

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- 8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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- 10 IP 367/96
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- 13 J.N. Miller, Analyst, 118, 455, (1993)
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