

Results of Proficiency Test
Metals in Plastics
September 2007

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1	INTRODUCTION	3
2	SET UP.....	3
2.1	QUALITY SYSTEM.....	3
2.2	PROTOCOL	4
2.3	SAMPLES	4
2.4	ANALYSES	5
3	RESULTS.....	5
3.1	STATISTICS.....	5
3.2	GRAPHICS.....	6
3.3	Z-SCORES.....	6
4	EVALUATION	7
4.1	PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES	7
4.2	EVALUATION PER SAMPLE.....	8
4.3	EVALUATION OF THE METHODS USED.....	9
4.4	COMPARISON WITH PAST PROFICIENCY TESTS	10
5	CONCLUSIONS.....	10

Appendices:

1.	Data, statistical results and graphic results	12
2.	Method information	28
3.	List of participants	36
4.	Abbreviations and literature.....	37

1 INTRODUCTION

World-wide many consumer products with plastic parts are produced and transported. These plastic products / parts are produced under strict regulations. Most of these restrictions are presented by governments like the European Union (EU). 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, which are causing problems with the import of the products.

The determination of Metals in several types of plastics is known to give problems with the comparability of laboratory results due to the loss of for instant volatile Cadmiumchloride. However, still only few plastic reference materials are available (VDA 001/004 and BCR680/681) and these materials are all polyethylene based. As alternative, participation in a proficiency test may enable the laboratories to check this comparability. Therefore, a proficiency test (laboratory-evaluating interlaboratory study) for the determination of metals in plastics was organised by the Institute for Interlaboratory Studies.

Since 1998 every year a proficiency test for Metals in plastic was organised. Starting with only total Cadmium, over the years the scope was extended with total Lead, total Chromium and Chromium (VI). During the annual proficiency testing program 2007/2008, it was decided to continue the proficiency test for the analysis of metals in plastics. In this international interlaboratory study of September 2007 participated 101 (!) laboratories in 29 different countries (See appendix 3).

In this report the results of the proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (i.i.s.) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. It was decided to send 3 different samples (approximately 3 gram each), labelled 0750, 0751 and 0752. Participants were also requested to report 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 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of November 2003 (iis-protocol, version 3.0).

2.3 SAMPLES

Three different samples were prepared for sampling. The first bulk material (#0750) obtained from a extruding facility, was a white coloured polyethylene. The second bulk material (#0751), obtained from a participating laboratory, was a bright yellow coloured pvc and the third bulk material (#0752), obtained also from a participating laboratory, was a light brown coloured polypropylene. The materials were divided over plastic bags, about 5 grams for each sample. The homogeneity of the subsamples 0750 was checked by determination of the total Cadmium content on 4 stratified random selected subsamples using EN 1122:01. The homogeneities of the subsamples 0751 and 0752 were checked by determination of the total Lead content on 4 stratified random selected subsamples using an in-house test method.

<i>Total Cadmium in mg/kg Subsample of #0750 (pe)</i>	<i>Total Lead in mg/kg Subsample of #0751 (pvc)</i>	<i>Total Lead in mg/kg Subsample of #0752 (pp)</i>
82	139	80
77	136	83
76	137	85
80	138	82

Table 1: results of the homogeneity test on the subsamples 0750, 0751 and 0752

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 0750</i>	<i>Subsample 0751</i>	<i>Subsample 0752</i>
Observed r in mg/kg	7.7	3.6	5.8
r (Horwitz) in mg/kg	--	11.7	7.6
r (EN1122:01) in mg/kg	7.8	--	--

Table 2: repeatabilities of total Cadmium and total Lead of the subsamples 0750, 0751 and 0752

The repeatability calculated for the samples 0750 is in good agreement with the repeatability required by EN 1122:2001.

The observed repeatabilities of the sample 0751 and 0752 are in agreement with the estimated repeatability calculated using the Horwitz equation.

Therefore, homogeneity of subsamples was assumed.

To each of the participating laboratories one set of samples, (1* sample 0750, 1* sample 0751 and 1 * sample 0752) were sent on August 22, 2007.

2.4 ANALYSIS

The participants were requested to determine on sample 0750: the total Cadmium content, total Chromium content and the hexavalent Chromium (VI) content. On sample 0751 was requested to determine total Lead content, total Chromium content and the hexavalent Chromium (VI) content. On sample 0752 was requested to determine total Cadmium content and total Lead content. They were explicitly asked to treat the samples as if they 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. They were also asked 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 asked to complete the report form with the requested 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. Their code numbers presents the laboratories.

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 'i.i.s. 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:

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

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 $Z_{(target)}$ 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 one problem was encountered with despatch of the samples. One laboratory did not receive the samples. Five participants reported the results after the final reporting date and eight participants did not report any results at all. Not all laboratories were able to report all analyses requested.

Finally, 92 of the 101 participants submitted analysis results. The 92 reporting laboratories sent in 500 numerical results. Observed were 26 outlying results, which is 4.9 %. 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)
Cadmium #0750 (*)	mg/kg	90	66.9 (*)	15.4 (*)	16.7
Chromium #0750	mg/kg	45	2.7	2.8	Unknown
Hexavalent Chromium (VI) #0750	mg/kg	12	0.5	1.3	Unknown

Table 3: performance overview for pe sample #0750

(*) #0750 was used before as #0557; the cadmium results of #0557 were: average 67.5 mg/kg, 2.8*sd = 14.4 mg/kg

Parameter	unit	n	Average	2.8 * sd	R (lit)
Lead #0751	mg/kg	84	141.2	41.8	37.1
Chromium #0751	mg/kg	83	33.8	14.6	8.9
Hexavalent Chromium (VI) #0751	mg/kg	21	1.3	2.9	Unknown

Table 4: performance overview for pvc sample #0751

Parameter	unit	n	Average	2.8 * sd	R (lit)
Cadmium #0752	mg/kg	44	1.0	1.3	Unknown
Lead #0752	mg/kg	86	92.7	20.8	24.4

Table 5: performance overview for the pp sample #0752

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 and total Lead. The tests that are problematic have been discussed in paragraph 4.2.

4.2 EVALUATION PER SAMPLE

In this section the results are discussed per sample and per element.

Sample #0750: The observed reproducibility for Cadmium is, after rejection of the statistical outliers, in full agreement with the requirements of EN1122:01. When the EN1122 and EPA3052 data for Cadmium were evaluated separately, the calculated reproducibility of the EPA3052 results is much smaller. When results of the detection technique were evaluated separately, the spread of the ICP-group did meet the requirements of EN1122:01, where $R(\text{ICP})$ is smaller than $R(\text{AAS})$. The assigned value found for total Chromium (2.7 mg/kg) and hexavalent chromium (VI) (0.5 mg/kg) is near or below the detection limit of the test methods used. Therefore no significant conclusion can be drawn.

Sample #0751: The observed reproducibility of total Lead is, after rejection of the statistical outliers, not in agreement with the requirements of ASTM D4004:02. When the EPA3052 data was evaluated separately, the calculated reproducibility of the groups is much smaller than the calculated reproducibility of the whole group. When results of the detection technique were evaluated separately, only the spread of the ICP-group did meet the requirements of ASTM D4004 for lead. Where $R(\text{ICP})$ is much smaller than $R(\text{AAS})$.
The observed reproducibility of total Chromium is, after rejection of the statistical outliers, not in agreement with the strict estimated reproducibility calculated using the Horwitz equation. When the EPA3052 data was evaluated separately, the calculated reproducibility is smaller than the calculated reproducibility of the whole group. When results of the detection technique were evaluated separately, both spreads of the ICP-group and AAS-group did not meet the estimated requirements calculated using the Horwitz equation. Where $R(\text{ICP})$ is smaller than $R(\text{AAS})$.
Only one laboratory was able to detect the Chromium VI present in the sample. Another 64 laboratories reported only very low amounts of Chromium VI. Therefore drawing a conclusion is difficult, see also paragraphs 4.3 and 5.

Sample #0752: The observed reproducibility of total Lead is, after rejection of the statistical outliers, in good agreement with the requirements of D4004:02. When the EPA3052 data were evaluated separately, the calculated reproducibility is smaller than the calculated reproducibility of the whole group. When results of the detection technique were evaluated separately, both the spreads of the AAS-group and ICP-group did meet the requirements of ASTM D4004:02, where $R(\text{ICP})$ is much smaller than $R(\text{AAS})$.
The assigned value found for total cadmium (1.0 mg/kg) is near or below the detection limit of the test methods used. Therefore no significant conclusion can be drawn.

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 total Cadmium, the EN1122 method is considered as the official EC test method for Cadmium determination. In this proficiency test 24 participants used a version of EN1122. Most other participants used in-house or EPA test methods based on acid digestion (wet ashing or microwave destruction) and four participants used XRF as method. No correlation between method used and quality of the results could be found. Only four 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. Twelve participants did correct for recovery.

It may be concluded that the detection of Cadmium in pe and pp materials in this proficiency test is not problematic.

For total Lead, there is not a specific EN/ISO or ASTM method available for total Lead determination in plastics. In this interlaboratory study, four participants reported EN 1122 as method, which is only applicable for Cadmium. Fifty participants used the EPA 3052, 3051 or 3050-B methods, which are designed for environmental samples and the applicability for plastics is unknown. Three participants used XRF as method and most other participants used inhouse 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. Twelve participants did correct for recovery. It may be concluded that the detection of total Lead in pp and pvc materials in this proficiency test is not problematic.

For total Chromium, there is not a specific EN/ISO or ASTM method available for total Chromium determination in plastics. In this interlaboratory study, four participants reported EN 1122 as method, which is only applicable for Cadmium. Forty-eight participants used the EPA 3050, 3051 and 3052 methods, which are designed for environmental samples and the applicability for plastics is unknown. Three participants used XRF as method 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. Nine participants used AAS as detection technique, while 73 participants used ICP. Twelve participants did correct for recovery. One may conclude that the total Chromium determination in plastics in this proficiency test is not problematic.

For hexavalent Chromium, a draft method is available for determination of Chromium VI in polymers: 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 only 6 laboratories used this draft method and most participants reported EPA 3060A as method.

The test method for Chromium VI does contain several details that may influence the test results strongly. Using a deviating sample size reduction, a deviating digestion temperature or a less strong alkaline extraction solution may be used. In this proficiency test some vital details as particle size of reduced sample, extraction technique and extraction conditions were requested (see appendix 2). A very large variation is seen in the sample reduction (column 3). It is remarkable to see that several laboratories that used CD/IEC62321 did not reduce the particle size to 500 µm or even not at all.

4.4 COMPARISON WITH PAST PROFICIENCY TESTS

The number of participants increased from 66 in 2005 to 101 in this round.

The absolute number of statistical outliers was 27 in 2005 and is 26 in 2007. The percentage of outliers decreased from 10.3% to 4.9 % of the numerical results.

The overall Cadmium, Lead and Chromium results of this proficiency test (observed reproducibilities) were compared with the previous proficiency test 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 %
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 round robin is in full agreement with the requirements of the standard EN1122:01. Only three laboratories of all 93 did have problems with the determination of total Cadmium content. All other laboratories proved to be able to test total Cadmium with sufficient reliability.

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). Only four laboratories of all 90 did have problems with the determination of total Lead content, while five other laboratories had problems with only one sample. All the others laboratories proved to be able to test total Lead with sufficient reliability.

The observed spread in the results for the total Chromium determination is somewhat larger compared with the previous proficiency test (iis06P03). Five laboratories of all 86 reporting

laboratories did have some 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 pvc is very problematic in this proficiency test. During the sample preparation procedure only hexavalent Chromium was added as PbCrO_4 , hence the bright yellow colour of sample #0751. Therefore the hexavalent Chromium content was expected to be equal or close to the total Chromium content of 33.8 mg/kg.

Regretfully, no assigned value could be determined, because only one laboratory was able to detect the Chromium VI present in the sample. This laboratory 2202 did dissolve the pvc sample using THF and thus released the Chromium VI from the pvc matrix. The other 64 laboratories reported only very low amounts of Chromium VI.

The use of THF is not described in the draft test method is IEC62321. This (draft) test method prescribes the sample treatment before use, with one essential test detail. A sample must be grounded into a fine powder cable for passing through a 500 μm sieve (chapter 10.5). From appendix 2 can be seen that only few laboratories did reduce the particle size. Also the low solubility of the lead chromate used in the sample #0751 may explain for the very low chromium VI results as found in this proficiency test.

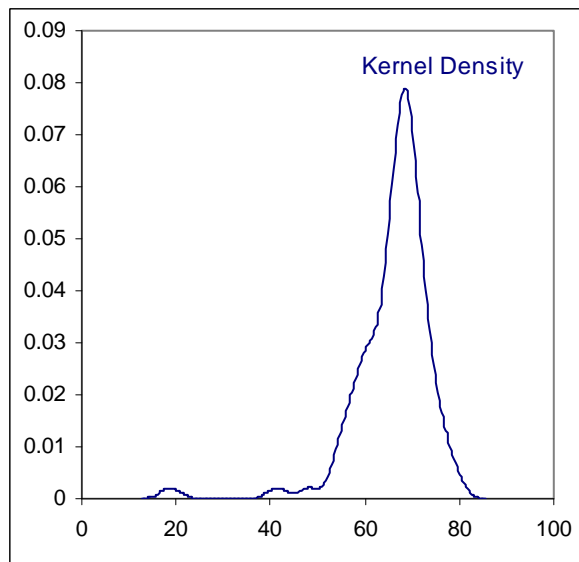
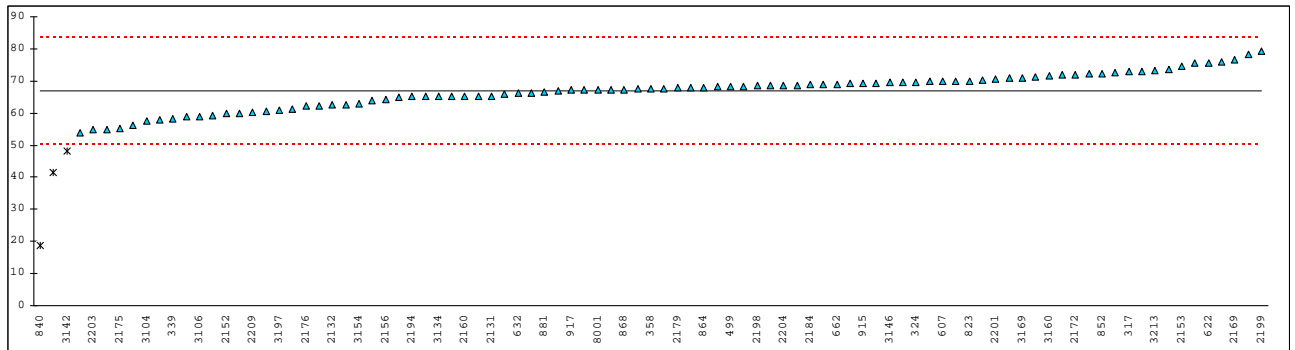
APPENDIX 1

Determination of total Cadmium as Cd on sample 0750; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
110		----		----	
310	in house	78.33		1.91	
317	in house	73		1.01	
324	EPA3052	69.64		0.45	
339	in house	58.3		-1.44	
358	in house	67.56		0.10	
390	EPA6020	67.29		0.06	
452	EN1122	57.74771		-1.54	
499	ICP-OES	68.2		0.21	
551	EPA3052	65.8		-0.19	
607	EPA3051-A	69.98		0.51	
622	EN1122	75.596		1.45	
632	EPA3052	66.20		-0.12	
662	EPA3051	69.0		0.35	
663		----		----	
823	EPA3052	70.05		0.52	
840	EPA3052	18.8	G(0.01)	-8.05	
852	EPA3052	72.3		0.90	
853	in house	66.8		-0.02	
854	EPA3051-A	73.1		1.03	
862	EPA3052	68.56		0.27	
864	EPA3052	68.0509		0.19	
868	EPA3052	67.3449		0.07	
869	EPA3052	71.2		0.71	
880	EPA3052	61.238		-0.95	
881	EPA3051	66.46		-0.08	
886	EPA3052	66.3		-0.11	
902	EPA3051-A	62.48772		-0.74	
915	EPA3052	69.32		0.40	
917	EPA3052	67.2		0.04	
1051	EN1122	72.2		0.88	
1126	XRF	56.3		-1.78	
2117	ISO11885	65.4		-0.26	
2129	ISO1729/EPA1620/EPA3015-A	68.57		0.27	
2131	in house	65.40		-0.26	
2132	in house	62.45		-0.75	
2146	EN1122	70.8		0.65	
2149	in house	71.956		0.84	
2152	ICP-AES	60		-1.16	
2153	EN1122	74.60		1.28	
2154		----		----	
2156	EN1122	64.09		-0.48	
2160	in house	65.4		-0.26	
2161	EN1122-B	72.5		0.93	
2169	EN1122	76.59183		1.62	
2172	EPA3051	71.9979		0.85	
2175	EPA3052	55.3	C	-1.95	First reported 49.3
2176	in house	62.1		-0.81	
2179	EPA3052	67.884		0.16	
2182		----		----	
2184	EN1122	68.94		0.34	
2185	EPA3052	68.39		0.24	
2190	in house	70	C	0.51	First reported 35
2192	EPA3052	70.39		0.58	
2194	in house	65.116		-0.30	
2196	EN1122-B	59.16		-1.30	
2197	EN1122	67.584		0.11	
2198	EN1122	68.4511		0.25	
2199	EPA3052	79.30		2.07	
2201	EPA3052	70.63		0.62	
2202	EN1122-B	68.0		0.18	
2203	in house	54.94		-2.01	
2204	EPA3052	68.56		0.27	
2205	EPA3052	69.96		0.51	
2206	EPA3052	65.14		-0.30	
2208		----		----	
2209	EN1122-B	60.351		-1.10	
3101	EN1122-B	54		-2.16	
3102	EN1122	75.5		1.43	
3104	EN1122	57.3916		-1.60	
3106	XRF Screening	59		-1.33	

3107	EPA3052	69		0.35	
3110	EN1122-B	60		-1.16	
3115	XRF	76		1.52	
3134	in house	65.25		-0.28	
3142	ICP	48.1	G(0.01)	-3.15	
3146	ISO11885	69.47		0.42	
3153	EPA3052	65.0		-0.32	
3154	EN1122	62.92		-0.67	
3159	EPA3051	65.266		-0.28	
3160	in house	71.75		0.81	
3162	EPA3052	58.88		-1.35	
3163	XRF	41.4	G(0.01)	-4.27	
3169	EN1122-B	71.09190		0.70	
3172		----		----	
3180	EN1122	55	C	-2.00	First reported 74
3182	EN1122	73.6		1.12	
3184	EPA3052	69.5		0.43	
3185	EPA3052	68.2		0.21	
3191	in house	67.53		0.10	
3192		----		----	
3197	IEC62321	60.8	C	-1.03	First reported 82.8
3199	EPA3051	63.974		-0.50	
3203	EN1122	62.12		-0.81	
3213	EPA3052	73.38		1.08	
3218	EPA3052	69.30		0.40	
3223		----		----	
3225	micr. dig.	60.65		-1.05	
3231	EPA3051	69.4		0.41	
4055	E29	67.34		0.07	
8001	EN1122-B	67.3		0.06	

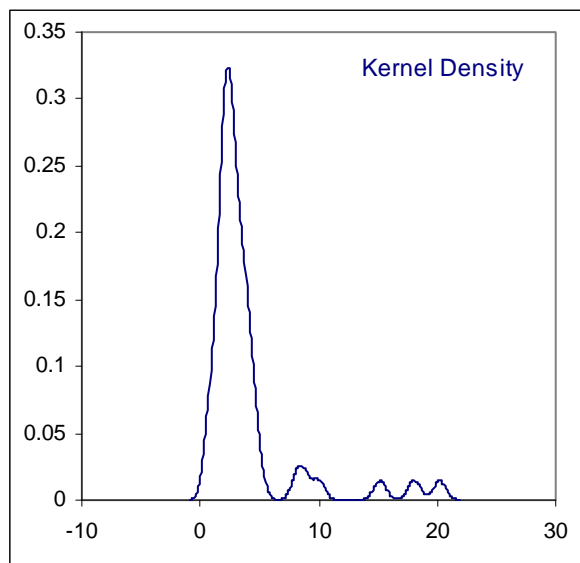
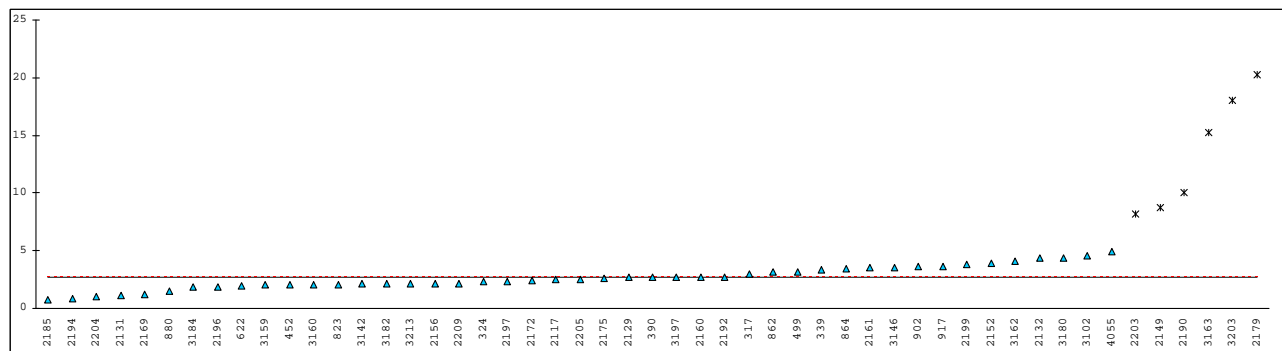
		Only EN1122 data:	Only EPA3052 data:	Only ICP data:	Only AAS data:
normality	not OK	OK	OK	not OK	OK
n	90	24	29	71	15
outliers	3	0	1	2	0
mean (n)	66.9	66.5	68.0	67.3	66.1
st.dev. (n)	5.51	6.99	4.37	5.22	6.12
R(calc.)	15.4	19.6	12.2	14.6	17.1
R(EN1122:01)	16.7	16.6	Unknown	16.8	16.5



Determination of total Chromium as Cr on sample 0750; results in mg/kg

lab	method	Value	mark	Z(targ)	remarks
110		----		----	
310		----		----	
317	in house	3		----	
324	EPA3052	2.31		----	
339	in house	3.3		----	
358	in house	<10		----	
390	EPA6020	2.680		----	
452	in house	2.019029		----	
499	ICP-OES	3.2		----	
551	EPA3052	nd		----	
607	EPA3051-A	nd		----	
622	EN1122	1.924		----	
632	EPA3052	nd		----	
662	EPA3051	nd		----	
663		----		----	
823	EPA3052	2.077		----	
840	EPA3052	nd		----	
852	EPA3052	<5		----	
853	in house	<5		----	
854	EPA3051-A	<2		----	
862	EPA3052	3.17		----	
864	EPA3052	3.42772		----	
868	EPA3052	nd		----	
869	EPA3052	nd		----	
880	EPA3052	1.528		----	
881	EPA3051	nd		----	
886	EPA3052	nd		----	
902	EPA3051-A	3.6		----	
915	EPA3052	<2		----	
917	EPA3052	3.62		----	
1051	in house	<5		----	
1126		----		----	
2117	ISO11885	2.49		----	
2129	ISO1729/EPA1620/EPA3015-A	2.659		----	
2131	in house	1.10		----	
2132	in house	4.35		----	
2146		----		----	
2149	in house	8.7405	C,G(0.01)	----	First reported 9.794
2152	ICP-AES	3.9		----	
2153	EPA3051	<2		----	
2154		----		----	
2156	EPA3052	2.15		----	
2160	in house	2.7		----	
2161	in house	3.5		----	
2169	EPA3052	1.17990		----	
2172	EPA3051	2.4226		----	
2175	EPA3052	2.6		----	
2176	in house	nd		----	
2179	EPA3052	20.222	G(0.01)	----	
2182		----		----	
2184	in house	<1		----	
2185	EPA3052	0.73		----	
2190	in house	10	C,G(0.01)	----	First reported 8
2192	EPA3052	2.730		----	
2194	in house	0.842		----	
2196	EN1122-B	1.88		----	
2197	ISO11885	2.328		----	
2198		----		----	
2199	EPA3052	3.84		----	
2201	EPA3052	nd		----	
2202		----		----	
2203	in house	8.21	G(0.01)	----	
2204	EPA3052	1.018		----	
2205	EPA3052	2.533		----	
2206	EPA3052	<2		----	
2208		----		----	
2209	EPA3052	2.184		----	
3101	EPA3050-B	<5		----	
3102	EN1122	4.53		----	
3104	EPA3051	<10		----	
3106	XRF Screening	nd		----	
3107	EPA3052	nd		----	
3110	EPA3052	<5		----	

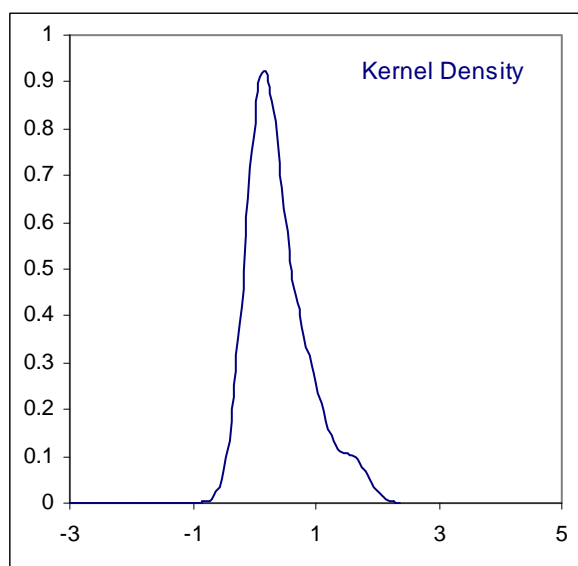
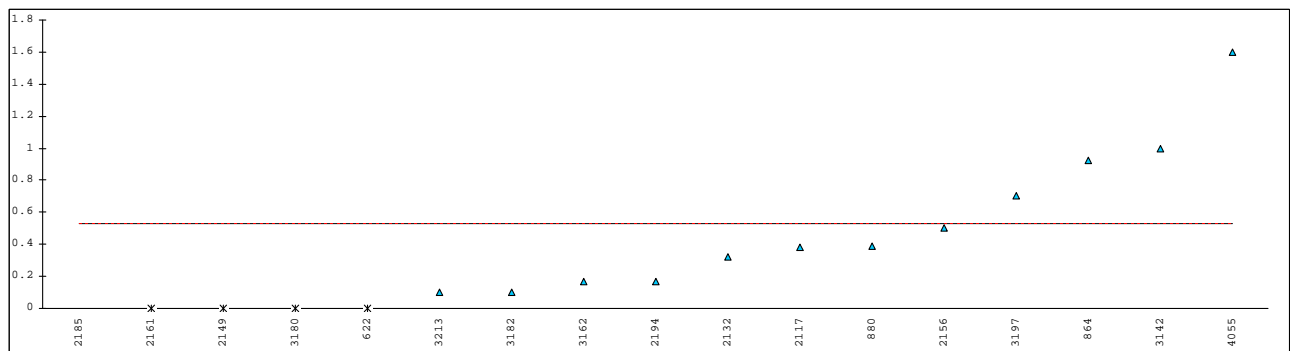
3115	XRF	<5		----
3134		----		----
3142	ICP	2.1		----
3146	ISO11885	3.5422		----
3153		----		----
3154	EN1122	<2		----
3159	EPA3051	2.007		----
3160	in house	2.07		----
3162	EPA3052	4.11		----
3163	XRF	15.2	G(0.01)	----
3169	in house	<5		----
3172		----		----
3180	EN1122	4.4	C	---- First reported 89
3182	EN1122	2.1		----
3184	EPA3052	1.83		----
3185	EPA3052	nd		----
3191	in house	<10		----
3192		----		----
3197	IEC62321	2.7		----
3199	EPA3051	<2.488		----
3203	EPA3052	18.07	C,G(0.01)	---- First reported 7.58
3213	EPA3052	2.12		----
3218	EPA3052	nd		----
3223		----		----
3225	micr. dig.	<2.00		----
3231	EPA3051	<10		----
4055	E29	4.914		----
8001	in house	<5		----
normality		OK		
n		45		
outliers		6		
mean (n)		2.7		
st.dev. (n)		1.01		
R(calc.)		2.8		
R(lit.)		Unknown		



Determination of Hexavalent Chromium as Cr⁶⁺ on sample 0750; results in mg/kg

lab	method	value	Mark	Z(targ)	remarks
110		----		----	
310		----		----	
317		----		----	
324	EPA3060-A	<2		----	
339		----		----	
358	EPA3060-A	<5		----	
390	EPA3060-A	<1		----	
452		----		----	
499	Photometry	<1		----	
551	EPA3052	nd		----	
607	EPA3060-A	nd		----	
622	in house	0	ex	----	Not a true value
632	EPA3060	nd		----	
662	EPA3060-A	nd		----	
663		----		----	
823	EPA3060-A	nd		----	
840	EPA3060-A	nd		----	
852	EPA3060-A	<5		----	
853	EPA3060-A	<2		----	
854	EPA3060-A	<2		----	
862	EPA3060	<2		----	
864	EPA3060-A	0.9250		----	
868		----		----	
869	EPA3060-A	nd		----	
880	EPA3060-A	0.3887		----	
881	EPA3060	nd		----	
886	EPA3060-A	nd		----	
902	EPA3060-A	nd		----	
915	EPA3060-A	<2		----	
917	EPA3060-A	<3		----	
1051		----		----	
1126		----		----	
2117	DIN53314	0.38		----	
2129		----		----	
2131		----		----	
2132	EPA3060-A	0.32		----	
2146		----		----	
2149	in house	0.0000	ex	----	Not a true value
2152	UV spec	<0.1		----	
2153	EPA3060-A	<2		----	
2154		----		----	
2156	EPA3060-A	0.5		----	
2160		----		----	
2161	in house	0.0	ex	----	Not a true value
2169		----		----	
2172	EPA3060	nd		----	
2175	EPA3060-A	<1		----	
2176	in house	nd		----	
2179	EPA3060-A	<1		----	
2182		----		----	
2184	EPA3060-A	<1		----	
2185	IEC62321	-6.97	ex	----	Not a true value
2190		----		----	
2192		----		----	
2194	in house	0.1706		----	
2196	EPA3060-A	<1.0		----	
2197	ISO17075	<10		----	
2198	EPA3060-A	nd		----	
2199	EPA3060-A	<2.0		----	
2201	EPA3060-A	nd		----	
2202		----		----	
2203		----		----	
2204		----		----	
2205		----		----	
2206	EPA3060-A	<2		----	
2208		----		----	
2209	EPA3060-A	<1		----	
3101	IEC62321	<5		----	
3102		----		----	
3104	IEC62321	<10		----	
3106		----		----	
3107	IEC-draft	nd		----	
3110	EPA3060-A	<5		----	

3115	----	----			
3134	----	----			
3142	UV	1.0			
3146	----	----			
3153	EPA3060-A	<1			
3154	----	----			
3159	EPA3060	<2			
3160	----	----			
3162	EPA3060-A	0.17			
3163	----	----			
3169	IEC62321	<2			
3172	----	----			
3180	DIN17075	0	ex	----	Not a true value
3182	EN1122	0.1		----	
3184	EPA3060-A	<1.0		----	
3185	EPA3060-A	nd		----	
3191	EPA3060-A	<2		----	
3192	----	----		----	
3197	IEC62321	0.7		----	
3199	----	----		----	
3203	EPA3060-A	<2		----	
3213	EPA3060-A	0.1		----	
3218	EPA3060-A	nd		----	
3223	----	----		----	
3225	EPA3060-A	<2.00		----	
3231	EPA3060	<10		----	
4055	EPA3060-A	1.60		----	
8001	EPA3060-A	<5		----	
normality	OK				
n	12				
outliers	0				
mean (n)	0.5				
st.dev. (n)	0.45				
R(calc.)	1.3				
R(lit.)	Unknown				

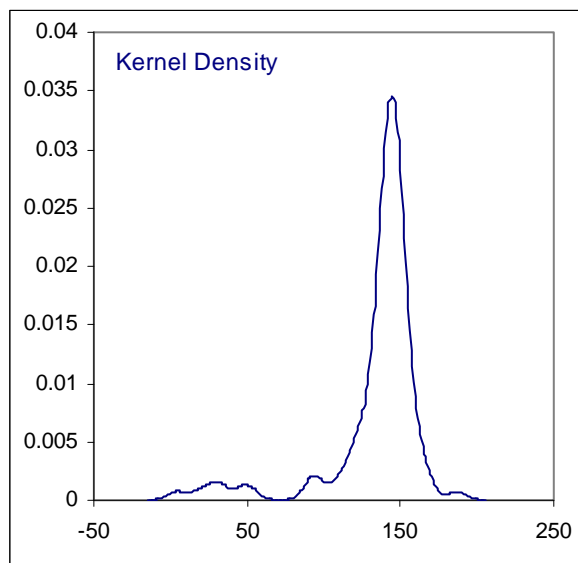
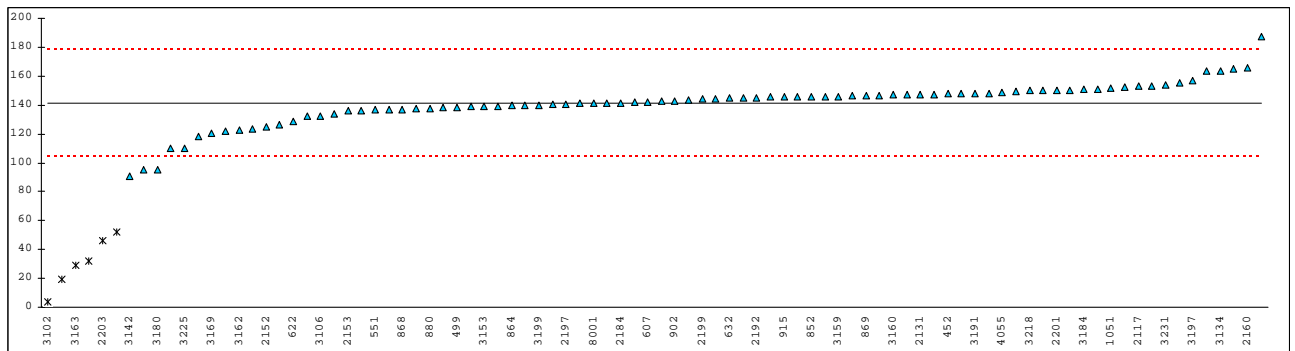


Determination of total Lead as Pb on sample 0751; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
110		----		----	
310		----		----	
317	in house	95		-3.48	
324	EPA3052	147.85		0.50	
339	in house	134		-0.54	
358	in house	163.5		1.68	
390	EPA6020	153.5		0.93	
452	in house	147.8473		0.50	
499	ICP-OES	138.4		-0.21	
551	EPA3052	136.9		-0.32	
607	EPA3051-A	141.91		0.06	
622	in house	128.512		-0.95	
632	EPA3052	144.63		0.26	
662	EPA3051	137.0		-0.31	
663		----		----	
823	EPA3052	143.7		0.19	
840	EPA3052	52.4	G(0.01)	-6.69	
852	EPA3052	145.8		0.35	
853	in house	151.1		0.75	
854	EPA3051-A	150.5		0.70	
862	EPA3052	144.71		0.27	
864	EPA3052	139.660		-0.11	
868	EPA3052	137.038		-0.31	
869	EPA3052	146.6		0.41	
880	EPA3052	137.878		-0.25	
881	EPA3051	139.73		-0.11	
886	EPA3052	136		-0.39	
902	EPA3051-A	142.921		0.13	
915	EPA3052	145.53		0.33	
917	EPA3052	140.64		-0.04	
1051	in house	152		0.82	
1126		----		----	
2117	ISO11885	153.2		0.91	
2129	ISO1729/EPA1620/EPA3015-A	145.87		0.35	
2131	in house	147.00		0.44	
2132	in house	126.40		-1.11	
2146		----		----	
2149	in house	146.90		0.43	
2152	ICP-AES	125		-1.22	
2153	EPA3051	135.98		-0.39	
2154		----		----	
2156	EPA3052	123.6		-1.32	
2160	in house	166.1		1.88	
2161	in house	145.6		0.33	
2169	EPA3052	149.32350		0.62	
2172	EPA3051	140.9173		-0.02	
2175	EPA3052	122.0		-1.45	
2176	in house	144.5		0.25	
2179	EPA3052	32.135	G(0.01)	-8.22	
2182		----		----	
2184	in house	141.54		0.03	
2185	EPA3052	138.12		-0.23	
2190	in house	132		-0.69	
2192	EPA3052	145.0		0.29	
2194	in house	145.437		0.32	
2196	in house	141.91		0.06	
2197	ISO11885	140.759		-0.03	
2198	EPA3050-B	165.3204		1.82	
2199	EPA3052	144.47		0.25	
2201	EPA3052	150.2		0.68	
2202	EN1122-B	148.2		0.53	
2203	in house	46.36	G(0.01)	-7.15	
2204	EPA3052	146.4		0.39	
2205	EPA3052	147.3		0.46	
2206	EPA3052	146.63		0.41	
2208		----		----	
2209	EPA3052	118.211		-1.73	
3101	EPA3050-B	19	G(0.01)	-9.21	
3102	Ashing-ICP	3.73	G(0.01)	-10.36	
3104	in house	139.3147		-0.14	
3106	XRF Screening	132	C	-0.69	First reported 103
3107	EPA3052	110	C	-2.35	First reported 107
3110	EPA3052	139		-0.16	

3115	XRF	187		3.46	
3134	in house	163.7	C	1.70	First reported 172.7
3142	ICP	90.5		-3.82	
3146	ISO11885	142.44		0.10	
3153	EPA3052	139.1		-0.16	
3154	EN1122	137.7	C	-0.26	First reported 110.67
3159	EPA3051	145.971		0.36	
3160	in house	146.85		0.43	
3162	EPA3052	122.75		-1.39	
3163	XRF	29.0	G(0.01)	-8.46	
3169	in house	120.5826		-1.55	
3172		-----		-----	
3180	EN1122	95	C	-3.48	First reported 85
3182	EPA3052	141.3		0.01	
3184	EPA3052	151.0		0.74	
3185	EPA3052	150		0.67	
3191	in house	148.0	C	0.52	First reported 184.7
3192		-----		-----	
3197	IEC62321	157.2		1.21	
3199	EPA3051	139.85		-0.10	
3203	EPA3052	152.26		0.84	
3213	EPA3052	155.24		1.06	
3218	EPA3052	150.00		0.67	
3223		-----		-----	
3225	micr. dig.	110.15		-2.34	
3231	EPA3051	154		0.97	
4055	E29	149.05		0.59	
8001	in house	141.0		-0.01	

		Only EPA3052 data:	Only ICP data:	Only AAS data:
Normality	not OK	not OK	not OK	OK
n	84	34	70	10
Outliers	6	2	4	1
mean (n)	141.2	140.8	141.2	139.5
st.dev. (n)	14.92	10.43	12.81	15.58
R(calc.)	41.8	29.2	35.9	43.6
R(D4004:02)	37.1	unknown	37.1	36.7

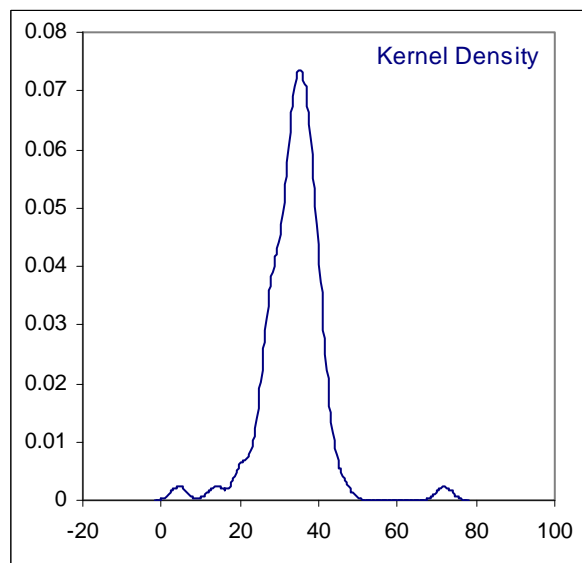
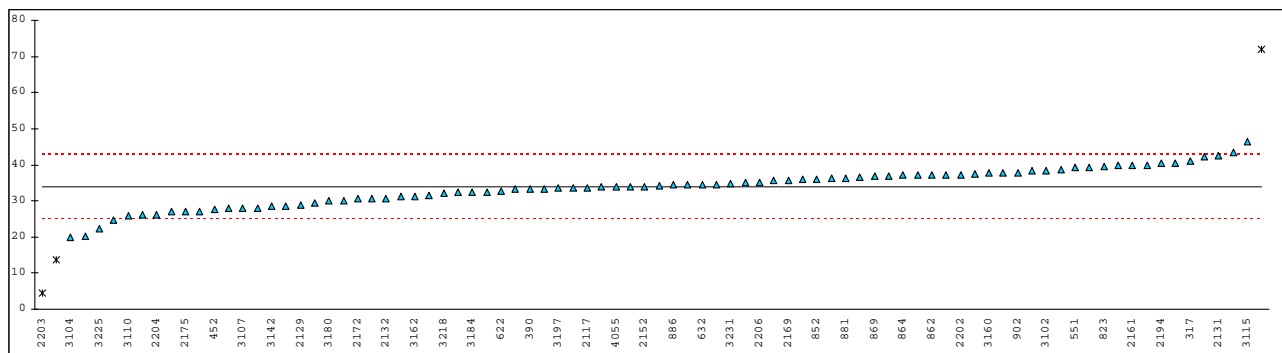


Determination of total Chromium as Cr on sample 0751; results in mg/kg

lab	method	value	mark	Z(targ)	Remarks
110		----		----	
310		----		----	
317	in house	41		2.25	
324	EPA3052	34.28		0.14	
339	in house	32.4		-0.45	
358	in house	42.12		2.61	
390	EPA6020	33.20		-0.20	
452	in house	27.74979		-1.92	
499	ICP-OES	35.7		0.59	
551	EPA3052	39.2		1.69	
607	EPA3051-A	33.36		-0.15	
622	in house	32.707		-0.36	
632	EPA3052	34.6		0.24	
662	EN1122	34.0		0.05	
663		----		----	
823	EPA3052	39.7		1.85	
840	EPA3052	13.75	G(0.05)	-6.33	
852	EPA3052	36.1		0.71	
853	in house	38.6		1.50	
854	EPA3051-A	38.3		1.40	
862	EPA3052	37.16		1.05	
864	EPA3052	37.0740		1.02	
868	EPA3052	36.62		0.88	
869	EPA3052	36.8		0.93	
880	EPA3052	36.328		0.78	
881	EPA3051	36.42		0.81	
886	EPA3052	34.4		0.18	
902	EPA3051-A	37.914		1.28	
915	EPA3052	37.47		1.14	
917	EPA3052	35.93		0.66	
1051	in house	28		-1.84	
1126		----		----	
2117	ISO11885	33.7		-0.04	
2129	ISO1729/EPA1620/EPA3015-A	28.90		-1.56	
2131	in house	42.40		2.70	
2132	in house	30.70		-0.99	
2146		----		----	
2149	in house	40.398		2.06	
2152	ICP-AES	34		0.05	
2153	EPA3051	29.48		-1.37	
2154		----		----	
2156	EPA3052	31.17		-0.84	
2160	in house	71.9	G(0.01)	11.98	
2161	in house	39.8		1.88	
2169	EPA3052	35.70200		0.59	
2172	EPA3051	30.5743		-1.03	
2175	EPA3052	27.0		-2.15	
2176	in house	37.2	C	1.06	First reported 50.0
2179	EPA3052	30.671		-1.00	
2182		----		----	
2184	in house	30.03		-1.20	
2185	EPA3052	27.96		-1.85	
2190	in house	40		1.94	
2192	EPA3052	32.55		-0.41	
2194	in house	40.342		2.05	
2196	in house	34.59		0.24	
2197	ISO11885	39.40		1.75	
2198		----		----	
2199	EPA3052	37.91		1.28	
2201	EPA3052	33.82		-0.01	
2202	EN1122-B	37.2		1.06	
2203	in house	4.38	G(0.01)	-9.28	
2204	EPA3052	26.29		-2.38	
2205	EPA3052	33.62		-0.07	
2206	EPA3052	35.19		0.43	
2208		----		----	
2209	EPA3052	31.621		-0.70	
3101	EPA3050-B	<5		----	False negative
3102	EN1122	38.5		1.47	
3104	EPA3051	19.92205		-4.38	
3106	XRF Screening	27	C	-2.15	First reported 51
3107	EPA3052	28	C	-1.84	First reported 17
3110	EPA3052	26		-2.47	

3115	XRF	46.5		3.99	
3134		-----		-----	
3142	ICP	28.5		-1.68	
3146	ISO11885	34.62		0.25	
3153		-----		-----	
3154	EN1122	24.6	C	-2.91	First reported 22.1
3159	EPA3051	37.152		1.04	
3160	in house	37.8		1.25	
3162	EPA3052	31.25		-0.82	
3163	XRF	20.2		-4.29	
3169	in house	28.630487		-1.64	
3172		-----		-----	
3180	EN1122	30	C	-1.21	First reported 16
3182	EPA3052	26.1		-2.44	
3184	EPA3052	32.5		-0.42	
3185	EPA3052	33.2		-0.20	
3191	in house	43.43		3.02	
3192		-----		-----	
3197	IEC62321	33.6		-0.08	
3199	EPA3051	26.935		-2.17	
3203	EPA3052	39.76		1.86	
3213	EPA3052	36.85		0.95	
3218	EPA3052	32.15		-0.53	
3223		-----		-----	
3225	micr. dig.	22.27		-3.64	
3231	EPA3051	34.8		0.30	
4055	E29	33.98		0.04	
8001	in house	35.1		0.40	

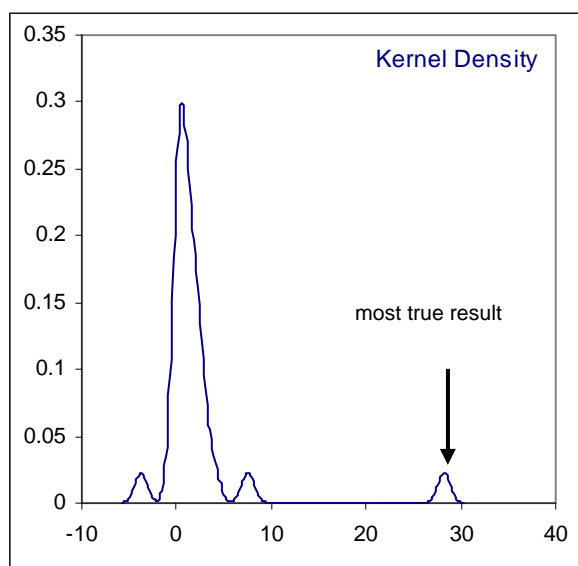
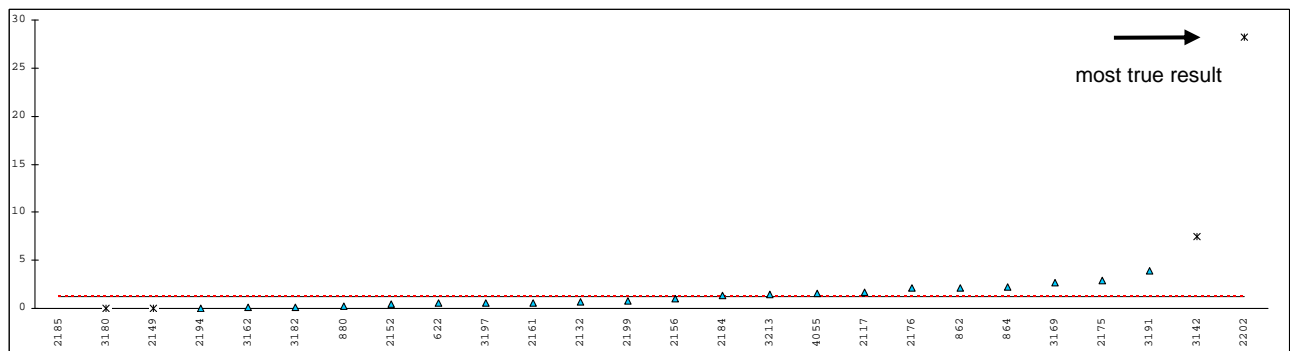
		Only EPA3052 data:	Only ICP data:	Only AAS data:
normality	OK	OK	OK	OK
n	83	34	72	8
outliers	3	1	1	1
mean (n)	33.8	33.7	34.5	29.1
st.dev. (n)	5.21	3.99	4.44	5.66
R(calc.)	14.6	11.2	12.4	15.8
R(Horwitz)	8.9	unknown	9.1	7.9



Determination of Hexavalent Chromium as Cr⁶⁺ on sample 0751; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
110		----		----	
310		----		----	
317		----		----	
324	EPA3060-A	<2		----	
339		----		----	
358	EPA3060-A	<5		----	
390	EPA3060-A	<1		----	
452		----		----	
499	Photometry	<1		----	
551	EPA3060-A	nd		----	
607	EPA3060-A	nd		----	
622	in house	0.54		----	
632	EPA3060	nd		----	
662	EPA3060-A	nd		----	
663		----		----	
823	EPA3060-A	nd		----	
840	EPA3060-A	nd		----	
852	EPA3060-A	<5		----	
853	EPA3060-A	<2		----	
854	EPA3060-A	<2		----	
862	EPA3060	2.14		----	
864	EPA3060-A	2.2100		----	
868	EPA3060	nd		----	
869	EPA3060-A	nd		----	
880	EPA3060-A	0.2726		----	
881	EPA3060	nd		----	
886	EPA3060-A	nd		----	
902	EPA3060-A	nd		----	
915	EPA3060-A	<2		----	
917	EPA3060-A	<3		----	
1051		----		----	
1126		----		----	
2117	DIN53314	1.66		----	
2129		----		----	
2131		----		----	
2132	EPA3060-A	0.62		----	
2146		----		----	
2149	in house	0.0000	ex	----	Not a true value
2152	UV spec	0.42		----	
2153	EPA3060-A	<2		----	
2154		----		----	
2156	EPA3060-A	1.0		----	
2160		----		----	
2161	in house	0.6		----	
2169		----		----	
2172	EPA3060	nd		----	
2175	EPA3060-A	2.9		----	
2176	in house	2.1		----	
2179	EPA3060-A	<1		----	
2182		----		----	
2184	EPA3060-A	1.31		----	
2185	IEC62321	-3.76	ex	----	Not a true value
2190		----		----	
2192		----		----	
2194	in house	0.04596		----	
2196	EPA3060-A	<1.0		----	
2197	ISO17075	<10		----	
2198		----		----	
2199	EPA3060-A	0.8	C	----	First reported 8.30
2201	EPA3060-A	nd		----	
2202	in house	28.2	C,G(0.01)	----	First reported 18.2
2203		----		----	
2204		----		----	
2205		----		----	
2206	EPA3060-A	<2		----	
2208		----		----	
2209	EPA3060-A	<1		----	
3101	IEC62321	<5		----	
3102		----		----	
3104	IEC62321	<10		----	
3106		----		----	
3107	IEC-draft	nd		----	
3110	EPA3060-A	<10		----	

3115	----		----	
3134	----		----	
3142	UV	7.5	G(0.01)	----
3146	----		----	
3153	EPA3060-A	<1		----
3154	----		----	
3159	EPA3060	<2		----
3160	----		----	
3162	EPA3060-A	0.07		----
3163	----		----	
3169	IEC62321	2.688		----
3172	----		----	
3180	DIN17075	0	ex	---- Not a true value
3182	EPA3052	0.1		----
3184	EPA3060-A	<1.0		----
3185	EPA3060-A	nd		----
3191	EPA3060-A	3.86		----
3192	----		----	
3197	IEC62321	0.6		----
3199	----		----	
3203	EPA3060-A	<2		----
3213	EPA3060-A	1.4		----
3218	EPA3060-A	nd		----
3223	----		----	
3225	EPA3060-A	<2.00		----
3231	EPA3060	<10		----
4055	EPA3060-A	1.60		----
8001	EPA3060-A	<5		----
normality	OK			
n	21			
outliers	2			
mean (n)	1.3			
st.dev. (n)	1.05			
R(calc.)	2.9			
R(lit.)	Unknown			



Determination of total Cadmium as Cd on sample 0752; results in mg/kg

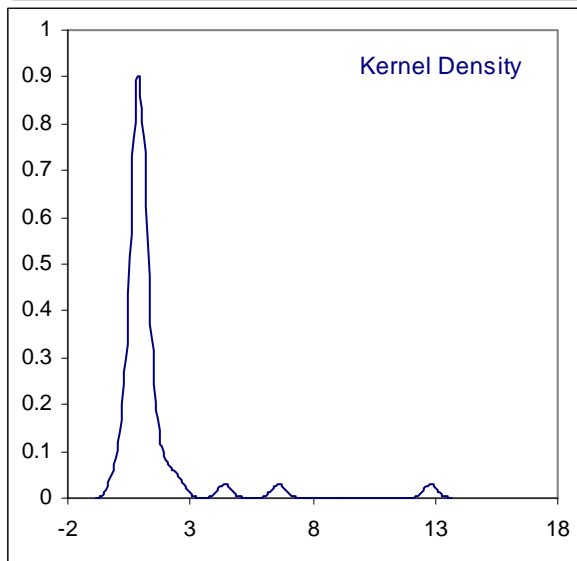
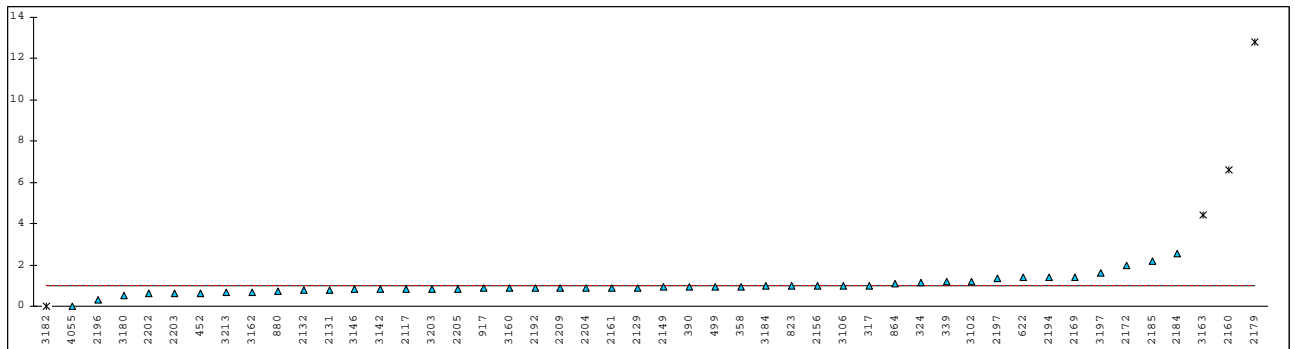
lab	method	value	mark	Z(targ)	remarks
110		----		----	
310	in house	<10		----	
317	in house	1		----	
324	EPA3052	1.14		----	
339	in house	1.2		----	
358	in house	0.953		----	
390	EPA6020	0.9199		----	
452	EN1122	0.634639		----	
499	ICP-OES	0.94		----	
551	EPA3052	nd		----	
607	EPA3051-A	nd		----	
622	EN1122	1.389		----	
632	EPA3052	nd		----	
662	EPA3051	nd		----	
663		----		----	
823	EPA3052	0.99		----	
840	EPA3052	nd		----	
852	EPA3052	<5		----	
853	in house	<5		----	
854	EPA3051-A	<2		----	
862	EPA3052	<2		----	
864	EPA3052	1.09069		----	
868	EPA3052	nd		----	
869	EPA3052	nd		----	
880	EPA3052	0.717		----	
881	EPA3051	nd		----	
886	EPA3052	nd		----	
902	EPA3051-A	nd		----	
915	EPA3052	<2		----	
917	EPA3052	0.86		----	
1051	EN1122	<5		----	
1126	XRF	<20		----	
2117	ISO11885	0.84		----	
2129	ISO1729/EPA1620/EPA3015-A	0.906		----	
2131	in house	0.77		----	
2132	in house	0.76		----	
2146	EN1122	nd		----	
2149	in house	0.9143		----	
2152	ICP-AES	<2		----	
2153	EN1122	<2		----	
2154		----		----	
2156	EN1122	0.995		----	
2160	in house	6.6	G(0.01)	----	
2161	EN1122-B	0.9		----	
2169	EN1122	1.42537		----	
2172	EPA3051	1.9529		----	
2175	EPA3052	<1		----	
2176	in house	nd		----	
2179	EPA3052	12.804	G(0.01)	----	
2182		----		----	
2184	EN1122	2.57		----	
2185	EPA3052	2.16		----	
2190	in house	<10		----	
2192	EPA3052	0.8794		----	
2194	in house	1.408		----	
2196	EN1122-B	0.31		----	
2197	EN1122	1.335		----	
2198	EN1122	nd		----	
2199	EPA3052	<2.00		----	
2201	EPA3052	nd		----	
2202	EN1122-B	0.6		----	
2203	in house	0.63		----	
2204	EPA3052	0.884		----	
2205	EPA3052	0.8530		----	
2206	EPA3052	<2		----	
2208		----		----	
2209	EN1122-B	0.882		----	
3101	EN1122-B	<5		----	
3102	EN1122	1.2		----	
3104	in house	<5		----	
3106	XRF Screening	1		----	
3107	EPA3052	<2		----	
3110	EN1122-B	<5		----	

3115	XRF	<10		----
3134	in house	<2.4		----
3142	ICP	0.84		----
3146	ISO11885	0.8307		----
3153	EPA3052	<2		----
3154	EN1122	<1		----
3159	EPA3051	<2		----
3160	in house	0.866		----
3162	EPA3052	0.68		----
3163	XRF	4.4	G(0.01)	----
3169	EN1122-B	<5		----
3172		----		----
3180	EN1122	0.5	C	----
3182	EPA3052	0	ex	----
3184	EPA3052	0.97		----
3185	EPA3052	nd		----
3191	in house	<10		----
3192		----		----
3197	IEC62321	1.6		----
3199	EPA3051	<2.339		----
3203	EN1122	0.852		----
3213	EPA3052	0.67		----
3218	EN1122	nd		----
3223		----		----
3225	micr. dig.	<2.00		----
3231	EPA3051	<10		----
4055	E29	0.01		----
8001	EN1122-B	<5		----

First reported 0
Not a true value

normality not OK
n 44
outliers 3
mean (n) 1.0
st.dev. (n) 0.45
R(calc.) 1.3
R(EN1122:01) Unknown

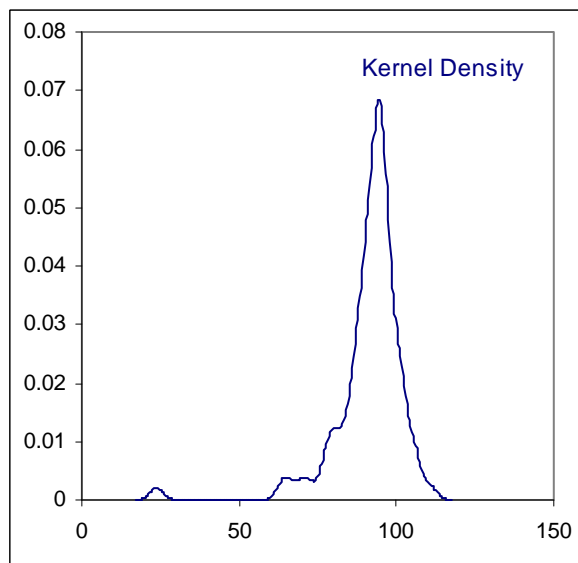
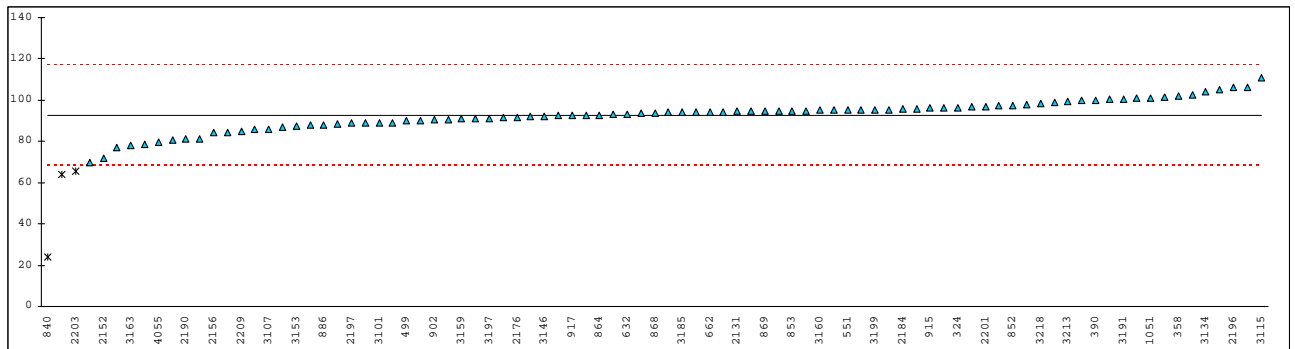
Range: 10 - 3000 mg Cd/kg



Determination of total Lead as Pb on sample 0752; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
110		----		----	
310		----		----	
317	in house	86		-0.77	
324	EPA3052	96.43		0.42	
339	in house	87.7		-0.58	
358	in house	101.8		1.04	
390	EPA6020	100.1		0.84	
452	in house	76.89226		-1.82	
499	ICP-OES	89.8		-0.34	
551	EPA3052	95.2		0.28	
607	EPA3051-A	96.04		0.38	
622	in house	88.390		-0.50	
632	EPA3052	93.35		0.07	
662	EPA3051	94.3		0.18	
663		----		----	
823	EPA3052	95.48		0.31	
840	EPA3052	23.8	G(0.01)	-7.91	
852	EPA3052	97.2		0.51	
853	in house	94.9		0.25	
854	EPA3051-A	98.7		0.68	
862	EPA3052	94.66		0.22	
864	EPA3052	92.5199		-0.03	
868	EPA3052	93.92		0.13	
869	EPA3052	94.8		0.24	
880	EPA3052	97.068		0.50	
881	EPA3051	91.01		-0.20	
886	EPA3052	87.8		-0.57	
902	EPA3051-A	90.301		-0.28	
915	EPA3052	96.04		0.38	
917	EPA3052	92.4		-0.04	
1051	in house	101		0.95	
1126		----		----	
2117	ISO11885	100		0.83	
2129	ISO1729/EPA1620/EPA3015-A	104.90		1.39	
2131	in house	94.50		0.20	
2132	in house	84.40		-0.96	
2146		----		----	
2149	in house	89.050		-0.42	
2152	ICP-AES	72		-2.38	
2153	EPA3051	91.36		-0.16	
2154		----		----	
2156	EPA3052	84.18		-0.98	
2160	in house	80.8		-1.37	
2161	in house	93.0		0.03	
2169	EPA3052	88.97650		-0.43	
2172	EPA3051	97.5980		0.56	
2175	EPA3052	78.7		-1.61	
2176	in house	91.7		-0.12	
2179	EPA3052	<30		----	False negative
2182		----		----	
2184	in house	95.56		0.32	
2185	EPA3052	95.22		0.28	
2190	in house	81		-1.35	
2192	EPA3052	94.31		0.18	
2194	in house	92.420		-0.04	
2196	EN1122-B	106.29		1.55	
2197	ISO11885	88.885		-0.44	
2198	EPA3050-B	102.5120		1.12	
2199	EPA3052	100.54		0.89	
2201	EPA3052	96.99		0.49	
2202	EN1122-B	94.0		0.14	
2203	in house	65.79	G(0.05)	-3.09	
2204	EPA3052	96.58		0.44	
2205	EPA3052	94.89		0.25	
2206	EPA3052	93.77		0.12	
2208		----		----	
2209	EPA3052	84.623	C	-0.93	First reported 74.482
3101	EPA3050-B	89		-0.43	
3102	Ashing-ICP	86.9		-0.67	
3104	in house	91.9014		-0.10	
3106	XRF Screening	95		0.26	
3107	EPA3052	86	C	-0.77	First reported 73
3110	EPA3052	90		-0.32	

3115	XRF	111		2.09	
3134	in house	104.3		1.33	
3142	ICP	63.9	G(0.05)	-3.31	
3146	ISO11885	91.97		-0.09	
3153	EPA3052	87.3		-0.63	
3154	EN1122	90.47		-0.26	
3159	EPA3051	90.895		-0.21	
3160	in house	95.00		0.26	
3162	EPA3052	81.20	C	-1.33	First reported 75.35
3163	XRF	78.0		-1.69	
3169	in house	94.9663		0.25	
3172		-----		-----	
3180	EN1122	70	C	-2.61	First reported 150
3182	EPA3052	106.4		1.57	
3184	EPA3052	101.4		0.99	
3185	EPA3052	94.2		0.17	
3191	in house	100.6		0.90	
3192		-----		-----	
3197	IEC62321	91.2		-0.18	
3199	EPA3051	95.275		0.29	
3203	EPA3052	95.84		0.35	
3213	EPA3052	99.51		0.78	
3218	EPA3052	98.20		0.63	
3223		-----		-----	
3225	micr. dig.	94.28		0.18	
3231	EPA3051	101		0.95	
4055	E29	79.53		-1.52	
8001	in house	92.4		-0.04	
	normality	OK			
	n	86			
	outliers	3			
	mean (n)	92.7			
	st.dev. (n)	7.41			
	R(calc.)	20.8			
	R(D4004:02)	24.4			
			Only EPA3052 data:	Only ICP data:	Only AAS data:
			not OK	not OK	OK
			34	72	10
			1	2	1
			93.4	92.6	94.1
			5.82	6.85	8.31
			16.3	19.2	23.3
			unknown	24.4	24.8



APPENDIX 2

Method information regarding total Cadmium determination

Lab	Analysis method	Technique to release / extract	Acid / acid mixture was used	Technique to detect and quantify	Corrected for rec.
110	--				
310	EN 1122	Acid digestion, microwave	--	AAS	No
317	in house	Acid digestion (only for 0751)	--	ICP (0751), XRF (0750+0752)	No
324	EPA 3052	Acid digestion, microwave	7 mL HNO ₃ + 3 mL HF	ICP-OES-MS	No
339	in house	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-OES	No
358	in house	Microwave	HNO ₃	ICP	No
390	EPA 6020	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-MS	No
452	EN 1122	Acid digestion	HNO ₃ + H ₂ SO ₄	ICP-AES	No
499	ICP-OES	Microwave digestion	HNO ₃ + H ₂ O ₂	ICP-OES	No
551	EPA 3052	Microwave	HNO ₃	ICP	No
607	EPA 3051-A	Microwave	Nitric acid	ICP	No
622	EN 1122	Acid digestion	HNO ₃ + H ₂ SO ₄ + H ₂ O ₂	ICP	No
632	EPA 3052	Acid digestion	HNO ₃ + HF	AAS	Yes
662	EPA 3051	Acid digestion, microwave	HNO ₃	ICP	No
663	--				
823	EPA 3052	Microwave	Nitric acid	ICP	No
840	EPA 3052	Microwave digestion	Nitric+Sulfuric+Hydrofluoric acid	ICP-OES	No
852	EPA 3052	Microwave digestion	Nitric acid	ICP	No
853	in house	Microwave digestion	HNO ₃ + H ₂ O ₂	ICP-OES	No
854	EPA 3051-A	Microwave digestion	HNO ₃	ICP	No
862	EPA 3052	Acid digestion	7mL HNO ₃ + 2mL HF + 2mL HCl	ICP-OES	Yes
864	EPA 3052	Microwave	10mL HNO ₃ + 0.75 mL HF	ICP	No
868	EPA 3052	Acid digestion, microwave	HNO ₃	ICP-AES	No
869	EPA 3052	Microwave	HNO ₃ + HF	ICP	No
880	EPA 3052	Acid digestion, microwave	10 mL HNO ₃ + 2 mL H ₂ O ₂	ICP-OES	No
881	EN 1122-B	Microwave	HNO ₃ + HCl	ICP-AES	No
886	EN 1122	Acid digestion, microwave	10 mL HNO ₃ + 1 mL HF	ICP-AES	No
902	EPA 3051-A	Microwave digestion	Conc. HNO ₃	ICP-MS	No
915	EPA 3052	Acid digestion, microwave	Conc. HNO ₃	ICP-MS	No
917	in house	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-MS-OES	No
1051	EN 1122	Acid digestion	H ₂ SO ₄	AAS	No
1126	XRF	--	--	XRF	--
2117	ISO 11885	Microwave digestion	HNO ₃	ICP-AAS	No
2129	ISO 1729/EPA 1620/EPA 3015-A	Acid digestion, microwave	HNO ₃	ICP-MS-OES	No
2131	Microwave digestion	Microwave	Nitric acid	ICP-MS	No
2132	in house	Microwave digestion	Conc.nitric acid+conc. H ₂ O ₂	ICP-OES	No
2146	EN 1122	Acid digestion	H ₂ SO ₄	AAS	No
2149	in house	Microwave digestion	HNO ₃ + H ₂ O ₂ + HCl	ICP	No
2152	ICP-AES	--	--	--	--
2153	EN1122	Hotplate digestion	Sulphuric acid + H ₂ O ₂	ICP-OES	No
2154	--				
2156	EN 1122-B	Acid digestion	Sulphuric acid + H ₂ O ₂	ICP-AES	No
2160	in house	--	Other	EDXRF	Yes
2161	EN 1122-B	Acid decomposition	H ₂ SO ₄ + H ₂ O ₂	ICP-AES	No
2169	EN 1122	Acid digestion	HNO ₃ + H ₂ O ₂ + H ₂ SO ₄	ICP-MS	No
2172	EPA 3051	Microwave	HNO ₃ + H ₂ O ₂	ICP-AES	Yes
2175	EPA 3052	Microwave	5mL HNO ₃ +2mL HCl+1mL H ₂ O ₂	ICP-OES	No
2176	in house	Acid digestion, microwave	HNO ₃ +HF+Masking by boric acid	ICP-AES	Yes
2179	EPA 3052/6010-B	Microwave	--	ICP	No
2182	--				
2184	EN1122	Acid digestion	H ₂ SO ₄ + H ₂ O ₂	AAS	No
2185	EPA 3052	Microwave digestion	Nitric acid	AAS	No
2190	in house	Acid digestion	H ₂ SO ₄ + H ₂ O ₂	ICP	No
2192	EPA 3052/6010-C	Microwave	Nitric acid	ICP-OES	No
2194	in house	Acid digestion, microwave	HNO ₃	ICP-OES	No
2196	EN 1122	Acid digestion	HNO ₃	AAS-ICP	No
2197	EN 1122	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-OES	No
2198	--				
2199	EPA 3052/EPA 6010-B	Microwave	HNO ₃ + HF + HCl	ICP	Yes
2201	EPA 3052	Microwave	Nitric acid	ICP	Yes
2202	EN 1122	Acid digestion	HNO ₃ + H ₂ O ₂ + H ₂ SO ₄	ICP	No
2203	in house	Acid digestion, Ash 450 °C	HCl 10% V/V	AAS-Flame	No
2204	EPA 3052/EPA 6010-B	Microwave	Nitric acid	ICP	No
2205	EPA 3052/EPA 6010-C	Microwave	Nitric acid	ICP-OES	No
2206	EPA 3052	Microwave	Nitric acid	ICP-OES	No

2208	--				
2209	EN 1122	Acid digestion	HNO ₃ + H ₂ O ₂ + H ₂ SO ₄	AAS	No
3101	EN 1122 (XRF)	Acid digestion	Mixture of H ₂ SO ₄ + H ₂ O ₂	ICP-OES	Yes
3102	EN 1122	Acid digestion, microwave	H ₂ SO ₄ + H ₂ O ₂	ICP	No
3104	EN 1122	0750: Acid digestion 0752: Acid digestion, ashing at 430 °C	0750+0752: H ₂ SO ₄	AAS	No
3106	XRF Sreening	NA	NA	XRF	NA
3107	EPA 3052	Microwave digestion	HNO ₃ approx. 9 M	ICP-OES	No
3110	EN 1122	Acid digestion	H ₂ SO ₄ + H ₂ O ₂	AAS-ICP	No
3115	XRF-uniqueant	---	--	XRF-uniqueant	Yes
3134	in house	Acid digestion	HNO ₃ + H ₂ SO ₄	AAS-Flame	No
3142	ICP	Acid digestion	HNO ₃ + H ₂ SO ₄	ICP	No
3146	ISO 11885	Acid digestion, microwave	Conc. HNO ₃ + H ₂ O ₂	ICP-OES	Yes
3153	EN 1122-B	Acid digestion, microwave	Nitric acid	ICP	No
3154	EN 1122	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	AAS	No
3159	EPA 3051	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP	No
3160	in house	microwave	HNO ₃	ICP-OES	No
3162	EPA 3052	Microwave	HNO ₃	ICP	No
3163	XRF	--	--	XRF	No
3169	EN 1122-B	Acid digestion	H ₂ SO ₄	AAS	No
3172	--				
3180	EN 1122	Microwave digestion	9mL HNO ₃ +1mL HCl+2mL H ₂ SO ₄	ICP-MS	No
3182	EN 1122	Hotplate digestion	H ₂ SO ₄ + H ₂ O ₂	ICP-OES	No
3184	EPA 3052	Microwave	HNO ₃	ICP-MS	No
3185	EPA 3052	Microwave	HNO ₃	ICP-OES	No
3191	in house	--	--	--	--
3192	--				
3197	IEC 62321	Acid digestion, microwave		ICP-OES	Yes
3199	EPA 3051	Acid digestion, microwave		ICP-AES	No
3203	EN 1122	Acid digestion		ICP	No
3213	EPA 3052/EPA 6020-A	Microwave		ICP	No
3218	EN 1122, EPA 3052	Acid digestion, microwave		ICP-XRF	Yes
3223	--				
3225	Microwave digestion	Microwave digestion		AAS	No
3231	EPA 3051	Microwave		ICP-AES	No
4055	Compendium of methods for food analysis, first ed. 2003	Microwave digestion		AAS-Flame	No
8001	EN 1122-B	Acid digestion		AAS	No

Method information regarding total Lead determination

Lab	Analysis method	Technique to release / extract	Acid / acid mixture was used	Technique to detect and quantify	Corrected for rec.
110	--				
310	in house	Acid digestion, microwave	--	AAS	No
317	in house	Acid digestion (only for 0751)	Ash at 500°C, HCl for 0751	ICP (0751), XRF (0750+0752)	No
324	EPA 3052	Acid digestion, microwave	7 mL HNO ₃ + 3 mL HF	ICP-OES-MS	No
339	in house	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-OES	No
358	in house	Microwave	HNO ₃	ICP	No
390	EPA 6020	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-MS	No
452	EN 1122	Acid digestion	HNO ₃ + H ₂ SO ₄	ICP-AES	No
499	ICP-OES	Microwave digestion	HNO ₃ + H ₂ O ₂	ICP-OES	No
551	EPA 3052	Microwave	HNO ₃	ICP	No
607	EPA 3051-A	Microwave	Nitric acid	ICP	No
622	EN 1122	Acid digestion	HNO ₃ + H ₂ SO ₄ + H ₂ O ₂	ICP	No
632	EPA 3052	Acid digestion	HNO ₃ + HF	AAS	Yes
662	in house	Combustion	HNO ₃	ICP	No
663	--				
823	EPA 3052	Microwave	Nitric acid	ICP	No
840	EPA 3052	Microwave digestion	Nitric+Sulfuric+Hydrofluoric acid	ICP-OES	No
852	EPA 3052	Microwave digestion	Nitric acid	ICP	No
853	in house	Microwave digestion	HNO ₃ + H ₂ O ₂	ICP-OES	No
854	EPA 3051-A	Microwave digestion	HNO ₃	ICP	No
862	EPA 3052	Acid digestion	7mL HNO ₃ + 2mL HF + 2mL HCl	ICP-OES	Yes
864	EPA 3052	Microwave	10mL HNO ₃ + 0.75 mL HF	ICP	No
868	EPA 3052	Acid digestion, microwave	HNO ₃	ICP-AES	No
869	EPA 3052	Microwave	HNO ₃ + HF	ICP	No
880	EPA 3052	Acid digestion, microwave	10 mL HNO ₃ + 2 mL H ₂ O ₂	ICP-OES	No
881	EN 1122-B	Microwave	HNO ₃ + HCl	ICP-AES	No
886	EN 1122	Acid digestion, microwave	10 mL HNO ₃ + 1 mL HF	ICP-AES	No
902	EPA 3051-A	Microwave digestion	Conc. HNO ₃	ICP-MS	No
915	EPA 3052	Acid digestion, microwave	Conc. HNO ₃	ICP-MS	No
917	in house	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-MS-OES	No
1051	EN 1122	Acid digestion	H ₂ SO ₄	AAS	No
1126	--	--	--	--	--
2117	ISO 11885	Microwave digestion	HNO ₃	ICP-AAS	No
2129	ISO 1729/EPA 1620/EPA 3015-A	Acid digestion, microwave	HNO ₃	ICP-MS-OES	No
2131	Microwave digestion	Microwave	Nitric acid	ICP-MS	No
2132	in house	Microwave digestion	Conc.nitric acid+conc. H ₂ O ₂	ICP-OES	No
2146	EN 1122	Acid digestion	H ₂ SO ₄	AAS	No
2149	in house	Microwave digestion	HNO ₃ + H ₂ O ₂ + HCl	ICP	No
2152	ICP-AES	--	--	--	--
2153	EPA 3051	Hotplate digestion	Sulphuric acid + H ₂ O ₂	ICP-OES	No
2154	--				
2156	EPA 3052	Acid digestion, microwave	HNO ₃ + HCl + H ₂ O ₂	ICP-AES	No
2160	in house	--	Other	EDXRF	Yes
2161	in house	Acid + Combustion decomposition	H ₂ SO ₄ + H ₂ O ₂	ICP-AES	No
2169	EPA 3052	Microwave	HNO ₃	ICP-MS	No
2172	EPA 3051	Microwave	HNO ₃ + H ₂ O ₂	ICP-AES	Yes
2175	EPA 3052	Microwave	5mL HNO ₃ +2mL HCl+1mL H ₂ O ₂	ICP-OES	No
2176	in house	Acid digestion, microwave	HNO ₃ +HF+Masking by boric acid	ICP-AES	Yes
2179	EPA 3052/6010-B	Microwave	--	ICP	No
2182	--				
2184	in house	Microwave	HNO ₃ + H ₂ O ₂	ICP-OES	No
2185	EPA 3052	Microwave digestion	Nitric acid	AAS	No
2190	in house	Acid digestion	H ₂ SO ₄ + H ₂ O ₂	ICP	No
2192	EPA 3052/6010-C	Microwave	Nitric acid	ICP-OES	No
2194	in house	Acid digestion, microwave	HNO ₃	ICP-OES	No
2196	in house	Acid digestion	HNO ₃	AAS	No
2197	ISO 11885	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-OES	No
2198	--				
2199	EPA 3052/EPA 6010-B	Microwave	HCl + HNO ₃ + HF	ICP	Yes
2201	EPA 3052	Microwave	Nitric acid	ICP	Yes
2202	EN 1122	Acid digestion	HNO ₃ + H ₂ O ₂ + H ₂ SO ₄	ICP	No
2203	in house	Acid digestion, Ash 450 °C	HCl 10% (V/V)	AAS-Flame	No
2204	EPA 3052/EPA 6010-B	Microwave	Nitric acid	ICP	No
2205	EPA 3052/EPA 6010-C	Microwave	Nitric acid	ICP-OES	No
2206	EPA 3052	Microwave	Nitric acid	ICP-OES	No

2208	--				
2209	EPA 3052	Microwave	HNO ₃ + H ₂ O ₂ + H ₂ SO ₄	AAS	No
3101	EPA 3050-B (XRF)	Acid digestion	Mixture of HNO ₃ + H ₂ O ₂ + HCl	ICP-OES	Yes
3102	Ashing-ICP	Ashing	HNO ₃	ICP	No
3104	in house	Acid digestion, ashing at 430 °C	H ₂ SO ₄	AAS	No
3106	XRF Sreening	NA	NA	XRF	NA
3107	EPA 3052	Microwave digestion	HNO ₃ approx. 9 M	ICP-OES	No
3110	EN 1122	Acid digestion	H ₂ SO ₄ + H ₂ O ₂	AAS-ICP	No
3115	XRF-uniqueant	--	--	XRF-uniqueant	Yes
3134	in house	Acid digestion	HNO ₃ + H ₂ SO ₄	AAS-Flame	No
3142	ICP	Acid digestion	HNO ₃ + H ₂ O ₂	ICP	No
3146	ISO 11885	Acid digestion, microwave	Conc. HNO ₃ + H ₂ O ₂	ICP-OES	Yes
3153	EN 1122-B	Acid digestion, microwave	Nitric acid	ICP	No
3154	EN 1122	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	AAS	No
3159	EPA 3051	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP	No
3160	in house	microwave	HNO ₃	ICP-OES	No
3162	EPA 3052	Microwave	HNO ₃	ICP	No
3163	XRF	--	--	XRF	No
3169	in house	Combustion	Nitric acid	ICP-OES	No
3172	--				
3180	EN 1122	Microwave digestion	9mL HNO ₃ +1mL HCl+2mL H ₂ SO ₄	ICP-MS	No
3182	EN 1122	Hotplate digestion	H ₂ SO ₄ + H ₂ O ₂	ICP-OES	No
3184	EPA 3052	Microwave	HNO ₃	ICP	No
3185	EPA 3052	Microwave	HNO ₃	ICP-OES	No
3191	in house	--	--	--	--
3192	--				
3197	IEC 62321	Acid digestion, microwave	Nitric acid	ICP-OES	Yes
3199	EPA 3051	Acid digestion, microwave	Nitric acid	ICP-AES	No
3203	EPA 3052	Microwave	Nitric acid	ICP	No
3213	EPA 3052/EPA 6020-B	Microwave	HNO ₃ + H ₂ O ₂	ICP	No
3218	EPA 3052	Microwave	HNO ₃ + H ₂ O ₂ + HF	ICP-XRF	Yes
3223	--				
3225	Microwave digestion	Microwave digestion	HNO ₃ + H ₂ O ₂	AAS	No
3231	EPA 3051	Microwave	HNO ₃ + H ₂ O ₂	ICP-AES	No
4055	Compendium of methods for food analysis, first ed. 2003	Microwave digestion	Conc. HNO ₃	AAS-Flame	No
8001	in house	Ashing	Nitric acid	ICP	No

Method information regarding total Chromium determination

Lab	Analysis method	Technique to release / extract	Acid / acid mixture was used	Technique to detect and quantify	Corrected for rec.
110	--				
310	in house	Acid digestion, microwave	--	AAS	No
317	in house	Acid digestion (only for 0751)	Digestion H ₂ O ₂ (only for 0751)	ICP (0751), XRF (0750+0752)	No
324	EPA 3052	Acid digestion, microwave	7 mL HNO ₃ + 3 mL HF	ICP-OES-MS	No
339	in house	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-OES	No
358	in house	Microwave	HNO ₃	ICP	No
390	EPA 6020	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-MS	No
452	EN 1122	Acid digestion	HNO ₃ + H ₂ SO ₄	ICP-AES	No
499	ICP-OES	Microwave digestion	HNO ₃ + H ₂ O ₂	ICP-OES	No
551	EPA 3052	Microwave	HNO ₃	ICP	No
607	EPA 3051-A	Microwave	Nitric acid	ICP	No
622	EN 1122	Acid digestion	HNO ₃ + H ₂ SO ₄ + H ₂ O ₂	ICP	No
632	EPA 3052	Acid digestion	HNO ₃ + HF	AAS	Yes
662	in house	Combustion	HNO ₃	ICP	No
663	--				
823	EPA 3052	Microwave	Nitric acid	ICP	No
840	EPA 3052	Microwave digestion	Nitric+Sulfuric+Hydrofluoric acid	ICP-OES	No
852	EPA 3052	Microwave digestion	Nitric acid	ICP	No
853	in house	Microwave digestion	HNO ₃ + H ₂ O ₂	ICP-OES	No
854	EPA 3051-A	Microwave digestion	HNO ₃	ICP	No
862	EPA 3052	Acid digestion	7mL HNO ₃ + 2mL HF + 2mL HCl	ICP-OES	Yes
864	EPA 3052	Microwave	10mL HNO ₃ + 0.75 mL HF	ICP	No
868	EPA 3052	Acid digestion, microwave	HNO ₃	ICP-AES	No
869	EPA 3052	Microwave	HNO ₃ + HF	ICP	No
880	EPA 3052	Acid digestion, microwave	10 mL HNO ₃ + 2 mL H ₂ O ₂	ICP-OES	No
881	EN 1122-B	Microwave	HNO ₃ + HCl	ICP-AES	No
886	EN 1122	Acid digestion, microwave	10 mL HNO ₃ + 1 mL HF	ICP-AES	No
902	EPA 3051-A	Microwave digestion	Conc. HNO ₃	ICP-MS	No
915	EPA 3052	Acid digestion, microwave	Conc. HNO ₃	ICP-MS	No
917	in house	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-MS-OES	No
1051	EN 1122	Acid digestion	H ₂ SO ₄	AAS	No
1126	--	--	--	--	--
2117	ISO 11885	Microwave digestion	HNO ₃	ICP-AAS	No
2129	ISO 1729/EPA 1620/EPA 3015-A	Acid digestion, microwave	HNO ₃	ICP-MS-OES	No
2131	Microwave digestion	Microwave	Nitric acid	ICP-MS	No
2132	in house	Microwave digestion	Conc.nitric acid+conc. H ₂ O ₂	ICP-OES	No
2146	EN 1122	Acid digestion	H ₂ SO ₄	AAS	No
2149	in house	Microwave digestion	HNO ₃ + H ₂ O ₂ + HCl	ICP	No
2152	ICP-AES	--	--	--	--
2153	EPA 3051	Hotplate digestion	H ₂ SO ₄ + H ₂ O ₂	ICP-OES	No
2154	--				
2156	EPA 3052	Acid digestion, microwave	HNO ₃ + HCl + H ₂ O ₂	ICP-AES	No
2160	in house	--	Other	EDXRF	Yes
2161	in house	Acid + Combustion decomposition	H ₂ SO ₄ + H ₂ O ₂	ICP-AES	No
2169	EPA 3052	Microwave	HNO ₃	ICP-MS	No
2172	EPA 3051	Microwave	HNO ₃ + H ₂ O ₂	ICP-AES	Yes
2175	EPA 3052	Microwave	5mL HNO ₃ +2mL HCl+1mL H ₂ O ₂	ICP-OES	No
2176	in house	Acid digestion, microwave	HNO ₃ +HF+Masking by boric acid	ICP-AES	Yes
2179	EPA 3052/6010-B	Microwave	--	ICP	No
2182	--				
2184	in house	Microwave	HNO ₃ + H ₂ O ₂	ICP-OES	No
2185	EPA 3052	Microwave digestion	Nitric acid	AAS	No
2190	in house	Acid digestion	H ₂ SO ₄ + H ₂ O ₂	ICP	No
2192	EPA 3052/6010-C	Microwave	Nitric acid	ICP-OES	No
2194	in house	Acid digestion, microwave	HNO ₃	ICP-OES	No
2196	in house	Acid digestion	HNO ₃	AAS	No
2197	ISO 11885	Acid digestion, microwave	HNO ₃ + H ₂ O ₂	ICP-OES	No
2198	--				
2199	EPA 3052/EPA 6010-B	Microwave	HCl + HNO ₃ + HF	ICP	Yes
2201	EPA 3052	Microwave	Nitric acid	ICP	Yes
2202	EN 1122	Acid digestion	HNO ₃ + H ₂ O ₂ + H ₂ SO ₄	ICP	No
2203	Varian-ICP-OES appl. Note 40	HNO ₃ +H ₂ O ₂ digestion	HNO ₃ +H ₂ O ₂	AAS-Furnace	No
2204	EPA 3052/EPA 6010-B	Microwave	Nitric acid	ICP	No
2205	EPA 3052/EPA 6010-C	Microwave	Nitric acid	ICP-OES	No
2206	EPA 3052	Microwave	Nitric acid	ICP-OES	No

2208	--				
2209	EPA 3052	Acid digestion	$\text{HNO}_3 + \text{H}_2\text{O}_2 + \text{H}_2\text{SO}_4$	AAS	No
3101	EPA 3050 (XRF)	Acid digestion	Mixture of $\text{HNO}_3 + \text{H}_2\text{O}_2 + \text{HCl}$	ICP-OES	--
3102	EN 1122	Acid digestion	$\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$	ICP	No
3104	in house	Acid digestion, ashing at 430 °C	H_2SO_4	AAS	No
3106	XRF Sreening	NA	NA	XRF	NA
3107	EPA 3052	Microwave digestion	HNO_3 approx. 9 M	ICP-OES	No
3110	EN 1122	Acid digestion	$\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$	AAS-ICP	No
3115	XRF-uniqueant	--	--	XRF-uniqueant	Yes
3134	in house	Acid digestion	$\text{HNO}_3 + \text{H}_2\text{SO}_4$	AAS-Flame	No
3142	ICP	Acid digestion	$\text{HNO}_3 + \text{H}_2\text{O}_2$	ICP	No
3146	ISO 11885	Acid digestion, microwave	Conc. $\text{HNO}_3 + \text{H}_2\text{O}_2$	ICP-OES	Yes
3153	EN 1122-B	Acid digestion, microwave	Nitric acid	ICP	No
3154	EN 1122	Acid digestion, microwave	$\text{HNO}_3 + \text{H}_2\text{O}_2$	ICP	No
3159	EPA 3051	Acid digestion, microwave	$\text{HNO}_3 + \text{H}_2\text{O}_2$	ICP	No
3160	in house	microwave	HNO_3	ICP-OES	No
3162	EPA 3052	Microwave	HNO_3	ICP	No
3163	XRF	--	--	XRF	No
3169	in house	Combustion	Nitric acid	ICP-OES	No
3172	--				
3180	EN 1122	Microwave digestion	9mL HNO_3 +1mL HCl +2mL H_2SO_4	ICP-MS	No
3182	EN 1122	Hotplate digestion	$\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$	ICP-OES	No
3184	EPA 3052	Microwave	HNO_3	ICP-MS	No
3185	EPA 3052	Microwave	HNO_3	ICP-OES	No
3191	in house	--	--	--	--
3192	--				
3197	IEC 62321	Acid digestion, microwave	Nitric acid	ICP-OES	Yes
3199	EPA 3051	Acid digestion, microwave	Nitric acid	ICP-AES	No
3203	EPA 3052	Microwave	Nitric acid	ICP	No
3213	EPA 3052/EPA 6020-B	Microwave	$\text{HNO}_3 + \text{H}_2\text{O}_2$	ICP	No
3218	EPA 3052	Microwave	$\text{HNO}_3 + \text{H}_2\text{O}_2 + \text{HF}$	ICP-XRF	Yes
3223	--				
3225	Microwave digestion	Microwave digestion	$\text{HNO}_3 + \text{H}_2\text{O}_2$	AAS	No
3231	EPA 3051	Microwave	$\text{HNO}_3 + \text{H}_2\text{O}_2$	ICP-AES	No
4055	Compendium of methods for food analysis, first ed. 2003	Microwave digestion	Conc. HNO_3	AAS-Flame	No
8001	in house	Ashing	Nitric acid	ICP	Yes

Method information regarding Hexavalent Chromium determination

Lab	Analysis method	Size of reduced sample	Technique to Release / extract	Extraction conditions	Corr for recov.	Other details
110	--	--	--	--	--	--
310	--	--	--	--	--	--
317	--	--	--	--	--	--
324	EPA 3060-A	As delivered	NaOH + Na ₂ CO ₃	60 minutes at 90-100 °C	No	--
339	--	--	--	--	--	--
358	EPA 3060-A/7196-A	No reducing	Alkaline digestion solution	1 hour, 95 °C, water bath	No	Not able to reduce the sample 0751
390	EPA 3060-A/7196-A	<1 mm	0.28 M Na ₂ CO ₃ / 0.5 M NaOH	60 minutes, 95 °C	No	--
452	--	--	--	--	--	--
499	Photometry	<1 mm	NaOH (20g/l)+Na ₂ CO ₃ (30g/l)+phosphate buffer pH7	60 minutes at 90-95 °C, shaking	No	Recovery for the spiked samples was >80%
551	EPA 3060-A	2*2*2 mm	Alkaline solution/ buffer solution	60 minutes at 90-95 °C	No	--
607	EPA 3060-A/7196-A	1 mm ³	Alkaline solution (NaOH and Na ₂ CO ₃)	60 minutes at 90-95 °C, continuous stirring	No	--
622	In house	0.300 mm (50 Mesh)	Alkaline solution (NaOH and Na ₂ CO ₃), MgCl ₂ and buffer	60 minutes at 90-95 °C, shaking waterbath	No	--
632	EPA 3060/7196	<2*2*2 mm	NaOH/ Na ₂ CO ₃	60 minutes at 90-95 °C	No	Sample wt 2.5 g; order of analysis the conc. blanc solution .
662	EPA 3060-A	<2*2*2 mm	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C, continuous stirring	No	--
663	--	--	--	--	--	--
823	EPA 3060-A	<2*2*2 mm	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C, continuous stirring	No	Sample weigh 2.5 g
840	EPA 3060-A	2*2 mm	Mixture of NaOH and Na ₂ CO ₃ , phosphate buffer pH7	60 minutes at 95 °C, magnetic stirring	No	--
852	EPA 3060-A	2*2*2 mm	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C, continuous stirring	No	NIL
853	EPA 3060-A	2*2*2 mm	NaOH/ Na ₂ CO ₃	60 minutes at 95 °C	No	--
854	EPA 3060-A	<2*2*2 mm	NaOH/ Na ₂ CO ₃	60 minutes at 90-95 °C, shaking at 111 rpm	No	--
862	EPA 3060	<2*2*2 mm	NaOH and Na ₂ CO ₃	60 minutes at 92.5 °C, continuous stirring	No	--
864	EPA 3060A	<0.5*0.5*0.5 mm	NaOH, Na ₂ CO ₃ , MgCl ₂ and Phosphate troton x-100	60 minutes at 90-95 °C, continuous stirring	No	--
868	EPA 3060	<1*1*1 mm	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C, continuous stirring	No	None
869	EPA 3060A	<2*2*2 mm	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C	No	UV-vis
880	EPA 3060A	<2*2*2 mm	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C, continuous stirring	No	Sample weight 2.5 g. order of analysis: analysis the cal. Blanc. Sol. .
881	EPA 3060	<0.5mm	NaOH/ Na ₂ CO ₃	1 hour, 90-95 °C	No	No
886	EPA 3060A	<2*2*2 mm	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C, continuous stirring	No	Sample weight 2.5 g
902	EPA 3060-A/7196-A	2*2*2 mm	Alkaline digestion	60 minutes heating at 90-95 °C, continuous stirring	No	NA
915	EPA 3060-A/7196-A	<2*2*2 mm	Alkali extraction using NaOH and Na ₂ CO ₃ sol.	60 minutes at 90-95 °C, continuous stirring	No	--
917	EPA 3060-A/7196-A	<2*2*2 mm	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C, continuous stirring	No	--
1051	--	--	--	--	--	--
1126	--	--	--	--	--	--
2117	ISO11885	Nr.0750 not red. Nr.0751: <0.5mm	K ₂ HPO ₄ 0.1 mol/l	3 hour orbital shake at 20°C	No	--
2129	--	--	--	--	--	--
2131	--	--	--	--	--	--
2132	EPA 3060-A/7196-A	0.5-1.0 mm	0.28M Na ₂ CO ₃ +0.5M NaOH, MgCl ₂ and phosphate buffer	1 hour at 90-95 °C, continuous stirring	No	NIL
2146	--	--	--	--	--	--
2149	In house	Min. 500µm	0.5M NaOH + 0.28M Na ₂ CO ₃ , Triton X-100	1 hour at 95±5 °C	No	NA
2152	UV Spectrometry	--	--	--	--	--
2153	EPA 3060-A	0.2*0.2*0.3 f	NaOH/ Na ₂ CO ₃	At 90°C for 1 hour	Yes	--
2154	--	--	--	--	--	--
2156	EPA 3060-A/7196-A	2 mm	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C	No	NIL
2160	--	--	--	--	--	--
2161	In house	Powder by frost shattering	Alkali extraction	1 hour at 95°C	No	--
2169	--	--	--	--	--	--
2172	EPA 3060	5*5 mm	Methanol, k ₂ HPO ₄ + KH ₂ PO ₄	60 minutes at 90-95 °C,	Yes	--
2175	EPA 3060-A/7196-A	2*2*2 mm	NaOH and Na ₂ CO ₃	60 minutes at 95±5 °C	No	NIL
2176	In house	around 2 mm	NaOH and Na ₂ CO ₃ , Buffer:	~1 hour	Yes	Develop the color by diphenyl carbazide.

			K2HPO4 and KH2PO4			Measure by UV-vis at 540nm
2179	EPA 3060-A/7196-A	< 500µm	NaOH and Na ₂ CO ₃ , Buffer: K2HPO4 and KH2PO4,	100 °C, 1 hour	No	None
2182	--	--	--	--	--	--
2184	EPA 3060-A	Around 2*2 mm	NaOH/ Na ₂ CO ₃	60 minutes at 95 °C	No	Cr6+ content measured by UV-vis
2185	IEC 62321	2*2*2 mm	Alkaline solution	Sonication 30 min. MgCl ₂ , at 95 °C for 1 h.	No	--
2190	--	--	--	--	--	--
2191	--	--	--	--	--	--
2194	In house	--	HNO ₃	--	No	Digested (same solv. used for tot. Cr det.
2196	EPA 3060-A/7196-A	< 500 µm	Alkaline digestion sol.(0.28M Na ₂ CO ₃ +0.5M NaOH)	60 minutes at 90-95 °C	No	--
2197	EN17075	--	Phosphate buffer pH 7.5-8.0	1 hour, room temp, 18-28 °C, ultrasonic	No	Sample was not milled, tested as received
2198	--	--	--	--	--	--
2199	EPA 3060-A	< 50µm	NaOH	2 hours, 95 °C	No	--
2201	EPA 3060-A/7196-A	< 1mm	20 g/ml NaOH and 30 g/ml Na ₂ CO ₃	1 hour, 95 °C	Yes	--
2202	in house	Sample was not powdered	Alkali digestion, 0751 used solv. THF	1 hour, 95 °C	No	--
2203	--	--	--	--	--	--
2204	--	--	--	--	--	--
2205	--	--	--	--	--	--
2206	EPA 3060-A/7196-A	3*3 mm	NaOH, Na ₂ CO ₃ , K2HPO4, KH2PO4, MgCl ₂ .6H ₂ O.	70 min., 90-95 °C, water vibration	No	ND
2208	--	--	--	--	--	--
2209	EPA 3060-A	35 mesh	Diphenylcarbazide, Na ₂ CO ₃ , NaOH	60 minutes at 90-95 °C	No	--
3101	IEC 62321	1*1 mm	Digestion sol. (0.28M Na ₂ CO ₃ +0.5M NaOH)	60 minutes at 93 °C,	Yes	--
3102	--	--	--	--	--	--
3104	IEC62321	<2*2 mm	Phosphate buffer	60 minutes at 90-95 °C	No	--
3106	--	--	--	--	--	--
3107	IEC draft	Not altered	NaOH and Na ₂ CO ₃	3 hours, 90-95 °C	No	ND
3110	EPA 3060-A/7196-A	<2*2*2 mm	0.28M Na ₂ CO ₃ , 0.5M NaOH, MgCl ₂ and Phosphate buffer	60 minutes at 90-95 °C, waterbath	No	--
3115	--	--	--	--	--	--
3134	--	--	--	--	--	--
3142	UV	2*2 mm	Acid digestion	Acid extraction digestion at 90-95 °C for 60 min.	No	The extract is determined by its reaction in acid condition with 1.5-diphenylcarbazide
3146	--	--	--	--	--	--
3153	EPA 3060-A/7196-A	About 1*1mm	Digestion solution	60 minutes at 90-95 °C	No	No
3154	--	--	--	--	--	--
3159	EPA 3060	--	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C,	No	Sample was cut into 5 pieces
3160	--	--	--	--	--	--
3162	EPA 3060-A	0.5*0.5 mm	NaOH+ Na ₂ CO ₃	60 minutes at 90-95 °C	No	--
3163	--	--	--	--	--	--
3169	IEC 62321/ED.1	<0.5 mm	NaOH+ Na ₂ CO ₃ +MgCl ₂	At 90-95 °C for 3 hours	No	Extraction time: 3 hours
3172	--	--	--	--	--	--
3180	DIN17075	As in DIN	K2HPO4 buffer	3 hours, at 20 °C, shake	No	NG:3mg/kg
3182	EPA 3052	2*2 mm	Alkaline digestion	Heat around 90°C, 1 hour	No	--
3184	EPA 3060-A/7196-A	< 500µm	Alkaline Digestion sol. (0.28M Na ₂ CO ₃ +0.5M NaOH)	60 minutes at 90-95 °C	No	--
3185	EPA 3060-A/7196-A	About 0.030 mm	2% NaOH+3% Na ₂ CO ₃	60 minutes at 90-95 °C	No	No
3191	--	--	--	--	--	--
3192	--	--	--	--	--	--
3197	IEC 62321	No treatment	NaOH/ Na ₂ CO ₃ digestion sol, MgCl ₂ , K2HPO4, KH2PO4	3 hours, 90 °C, shaking waterbath	Yes	--
3199	--	--	--	--	--	--
3203	EPA 3060-A	Not cut	NaOH and Na ₂ CO ₃	60 minutes at 90 °C	No	No
3213	EPA 3060-A/7196-A	Freezer mill	NaOH and Na ₂ CO ₃	2 hours at 95 °C, heating	No	--
3218	EPA 3060-A/7196-A	Through a size 500µm sieve	Alkaline sol.:MgCl ₂ , Phosphate sol., Triton X-100	60 minutes at 90-95 °C	No	UV-vis spectrophotometer with 2 cm cell and at 540nm
3223	--	--	--	--	--	--
3225	EPA 3060-A	Powder	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C	No	--
3231	EPA 3060	as received	NaOH+ Na ₂ CO ₃ ; pH>11.5	Heated 60 minutes at 95 °C	No	0750: Triton X-100 was added
4055	EPA 3060-A/7196-A	<0.25 mm	0.28M Na ₂ CO ₃ +0.5M NaOH	60 minutes at 90-95 °C	No	--
8001	EPA 3060-A	2*2*2 mm	NaOH and Na ₂ CO ₃	60 minutes at 90-95 °C	No	NIL

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

Number of laboratories	Country
1 laboratory in	BANGLADESH
1 laboratory in	BELGIUM
1 laboratory in	BRASIL
2 laboratories in	FINLAND
2 laboratories in	FRANCE
7 laboratories in	GERMANY
2 laboratories in	GREECE
17 laboratories in	HONG KONG
2 laboratories in	INDIA
1 laboratory in	INDONESIA
3 laboratories in	ITALY
2 laboratories in	JAPAN
4 laboratories in	KOREA
3 laboratories in	MALAYSIA
19 laboratories in	P.R. of CHINA
2 laboratories in	PHILIPPINES
1 laboratory in	PORTUGAL
2 laboratories in	SINGAPORE
2 laboratories in	SOUTH KOREA
2 laboratories in	SPAIN
2 laboratories in	SWITZERLAND
3 laboratories in	TAIWAN R.O.C.
5 laboratories in	THAILAND
6 laboratories in	THE NETHERLANDS
3 laboratories in	TURKEY
2 laboratories in	U.S.A.
2 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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