Results of Proficiency Test
AZO dyes in textile
March 2014

CONTENTS

Appendices:

1   INTRODUCTION

The Institute for Interlaboratory Studies (iis) organizes every year a proficiency test (PT) for banned aromatic amines from AZO dyes in textile since 1997. During the annual proficiency testing program 2013/2014, it was decided to continue the PT for the analysis of banned aromatic amines dyes in textile. In this interlaboratory study, 159 laboratories in 33 different countries have participated (see appendix 4).
In this report, the results of the 2014 PT are presented and discussed. This report is also electronically available through the iis internet site http://www.iisnl.com.

2   SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organizer of this proficiency test. It was decided to use in this proficiency test 2 different textile samples (labelled #14020 and #14021), each dyed with different AZO dyes. Both samples were especially prepared by a third party. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory.
The participants were requested to report results with one extra figure. These results with an extra figure are preferably used for statistical evaluation. The participants were asked to report the analytical results using the indicated units on the report form.

2.1   ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires

2.2   PROTOCOL

The protocol followed in the organization was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

2.3   CONFIDENTIALITY STATEMENT

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

2.4   SAMPLES

The two different bulk textile samples, a blue cotton (sample #14020) and a red cotton (sample #14021) were dyed with different AZO-dyes. From the first batch, 174 samples with an equal piece of fabric of approximately 3 gram were prepared and labelled #14020. From the second batch, 157 samples with an equal piece of fabric of approximately 1.5 gram were prepared and labelled #14021. The homogeneity of the subsamples #14020 and #14021 was checked by determination of an aromatic amine on respectively 5 and 4 stratified randomly selected samples. See the following tables for the test results.

	3,3'-Dimethoxybenzidine in mg/kg
sample #14020-1	190
sample #14020-2	210
sample #14020-3	200
sample #14020-4	200
sample #14020-5	210

Table 1: homogeneity test results of subsamples #14020

	4,4'-diaminodiphenylmethane in mg/kg
sample #14021-1	420
sample #14021-2	386
sample #14021-3	412
sample #14021-4	396

Table 2: homogeneity test results of subsamples #14021

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the target method in agreement with the procedure of ISO 13528, Annex B2 in the next tables:

	3,3'-Dimethoxybenzidine in mg/kg	4,4'-diaminodiphenylmethane in mg/kg
r (observed) #14020	23.4	--
r (observed) #14021	--	43.0
reference method	EN14362-1:2012	EN14362-1:2012
0.3 x R (reference method)	26.1	68.4

Table 3: repeatabilities of subsamples #14020 and #14021

The calculated repeatabilities of the test results were in agreement with 0.3 times the reproducibilities mentioned in (or estimated from) the reference method EN14362-1. Therefore, homogeneity of the subsamples was assumed.

To the participating laboratories was sent 1 sample labelled #14020 and 1 sample labelled #14021 on March 5, 2014.

2.5 ANALYSES

The participants were asked to determine the concentrations of 23 forbidden aromatic amines and *o*-anisidine, applying the analysis procedure that is routinely used in the laboratory. To get comparable results reported, a detailed report form, on which the requested amines and the units were pre-printed, was sent together with each set of samples. A letter of instructions was sent along.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated in the appendices of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not yet reported. 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 data are placed under 'Remarks' in the result tables in appendix 1. A list of abbreviations used in the tables can be found in appendix 4.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3)
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<…' or '>…" were in general not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

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 and by R(0.01) for the Rosner General ESD test (see appendix 4, no.15). Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibities 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. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. 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 5; nos.13 and 14). Also a normal Gauss curve was projected over the Kernel Density Graph.

3.3   Z-SCORES

To evaluate the performance of the individual participating laboratories the z-scores were calculated. In order to be able to have an objective evaluation of the performance of the individual participants, it was decided to evaluate this performance against the literature requirements. Therefore the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8.
The $z_{(target)}$-scores were calculated according to:

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

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

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

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

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

4   EVALUATION

During the execution of this proficiency test some reporting problems occurred. Twenty-five participants reported the results after the deadline and three participants did not report any test result. Finally, 156 participants did report 907 numerical results. Observed were 35 outlying results, which is 3.9% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.
Not all data sets proved to have a normal distribution. Not normal (or suspect) distributions were found for 3,3'-Dimethoxybenzidine, 4-Chloro-o-toluidine, *o*-Toluidine and 4,4'-Diaminodiphenylmethane. Therefore, the statistical evaluations should be used with care. There are no reproducibility requirements for 4-Chloro-o-toluidine reported in EN14362-1:2012, therefore the target reproducibilities were estimated from the reproducibilities of all aromatic amine compounds mentioned in EN14362-1:2012.

4.1   EVALUATION PER SAMPLE AND PER COMPONENT

In this section, the results are discussed per sample. All statistical results reported on the textile samples are summarised in appendix 1 and all other reported results of the most relevant aromatic amines present are summarised in appendix 2.

Textile sample #14020:

4-Aminodiphenyl (CASno.92-67-1):
The consensus value of this aromatic amine was found at a concentration level of 5.9 mg/kg, which is near or below the detection limit of the test method. Therefore no significant conclusions were drawn.

Benzidine (CASno.92-87-5):
The determination of this aromatic amine at a concentration level of 77 mg/kg was problematic for a number of laboratories. Fifteen statistical outliers and one false negative test result were detected. The numerical test results reported by the participants vary from 2.04 – 2085.7 mg/kg.
However, the observed reproducibility after rejection of the statistical outliers is in full agreement with the reproducibility requirement estimated from the test method EN14362-1:2012.

3,3'-Dimethoxybenzidine (CASno.119-90-4):
The determination of this aromatic amine at a concentration level of 220 mg/kg was problematic. Seven statistical outliers and two false negative test results were detected. The numerical test results reported by the participants vary from 2.75 – 550.5 mg/kg. The observed reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility requirement estimated from the test method EN14362-1:2012.

Textile sample #14021:

4-Chloro-o-toluidine (CASno.95-69-2):
The determination of this aromatic amine at a concentration level of 508 mg/kg was problematic. Four statistical outliers and one false negative test result were detected. The numerical test results reported by the participants vary from 11.10 – 2661.8 mg/kg. The observed reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility requirement estimated from the test method EN14362-1:2012.

4,4'-Diaminodiphenylmethane (CASno.101-77-9):
The determination of this aromatic amine at a concentration level of 406 mg/kg, was problematic. Six statistical outliers and two false negative test results were detected. The numerical test results reported by the participants vary from 'nd' – 8914.1 mg/kg. The observed reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility requirement estimated from the test method EN14362-1:2012.

o-Toluidine (CASno.95-53-4) in sample #14021
The determination of this aromatic amine at a concentration level of 295 mg/kg was problematic. Only one statistical outlier and one false negative test result were detected. The test results reported by the participants vary from 'nd'- 1071.2 mg/kg. The observed reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility requirement estimated from the test method EN14362-1:2012.

o-Aminoazotoluene (CASno.97-56-3):
The determination of this aromatic amine was problematic. Seventy-one laboratories reported the absence of this amine via a 'less than' test result or 'not detected'. Surprisingly however, twenty-nine other laboratories did report a numerical test result for this amine in the range 2.927 – 573.59 mg/kg. Therefore no consensus value could be determined. See also the discussion in chapter 6.

<u>p-Chloroaniline (CASno.106-74-8):</u>
The determination of this aromatic amine was problematic. Seventy-five laboratories reported a 'less than' test result or 'not detected'. However, thirty other laboratories reported numerical test results in the range 5 - 104.3 mg/kg. Therefore no consensus value could be determined. See also the discussion in chapter 6.

<u>General:</u>
Ten participants reported also the presence of other aromatic amines at different concentration levels in sample #14020. Nine participants reported small amounts of o-anisidine. (see Appendix 2).
Ten participants reported also the presence of other aromatic amines at different concentration levels in sample #14021 (see Appendix 2). A number of participants identified the amines incorrectly or may have reported test results in the wrong row of the report form.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities as declared by the relevant standard test methods and the reproducibilities as found for the group of participating laboratories.

The number of significant results, the average results, the calculated reproducibilities (standard deviation*2.8) and the target reproducibilities, derived (or estimated) from the official test method EN14362-1 (equivalent to LFGB 82.02-2), are compared in the next two tables.

Parameter	unit	n	average	2.8 * sd	R(target)
4-Aminodiphenyl	mg/kg	81	5.87	3.38	(2.66)*
Benzidine	mg/kg	140	76.7	32.0	29.7
3,3'-Dimethoxybenzidine	mg/kg	147	220	128	77

Table 4: reproducibilities of the aromatic amines in textile sample #14020

Parameter	unit	n	average	2.8 * sd	R(target)
4-Chloro-o-toluidine	mg/kg	149	509	346	230
4,4'-Diaminodiphenylmethane	mg/kg	145	406	242	172
o-Toluidine	mg/kg	152	295	266	180

Table 5: reproducibilities of the aromatic amines in textile sample #14021

* value between brackets is for concentration near or below the detection limit

Without further statistical calculations, it can be concluded that the group of participating laboratories has difficulties with the analysis at the investigated concentration levels, for all present aromatic amines. See also the discussion in paragraphs 4.1 and 6.

5   COMPARISON WITH PREVIOUS INTERLABORATORY STUDIES

The spreads in the results of the aromatic amines are all in line with the spreads as observed in previous PTs and almost in agreement with the target reproducibilities estimated from the standardized test method EN14362-1:2012, see below table.

Parameter	April 2014	March 2013	March 2012	March 2011	March 2010	March 2009	March 2008	EN 14362
4-Aminodiphenyl	(21%)*	n.e.	18%	(31%)*	18%	n.e.	n.e.	59%
Benzidine	15%	n.e.	21%	18%	19%	n.e.	20%	14%
3,3'-Dimethylbenzidine	n.e.	n.e.	n.e.	n.e.	17%	n.e.	32%	18%
3,3'-Dimethoxybenzidine	21%	16%	17%	17%	n.e.	n.e.	n.e.	13%
o-Toluidine	31%	27%	n.e.	n.e.	19%	n.e.	n.e.	22%
2-Naphthylamine	n.e.	n.e	n.e.	n.e.	n.e.	n.e.	27%	59%
4,4'-Diaminodiphenylmethane	21%	n.e.	n.e.	n.e.	n.e.	21%	n.e.	15%
4,4'-Diaminodiphenylsulfide	n.e.	n.e.	n.e.	n.e.	18%	n.e.	26%	16%
p-Chloroaniline	n.e.**	n.e.**	n.e.	n.e.	n.e.	27%	n.e.	16%
4,4'-Diaminodiphenylether	n.e.	n.e.	n.e.	15%	n.e.	n.e.	n.e.	16%
4,4'-Diamino-3,3'-dichlorodiphenylmethane	n.e.	n.e.	n.e.	20%	n.e.	23%	n.e.	16%
2,4-Diaminoanisol	n.e.	52%	n.e.	n.e.	n.e.	n.e.	n.e.	16%
4-Chloro-o-toluidine	24%	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	16%
o-Aminoazotoluene	n.e.**	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	16%

Table 6: long term development of uncertainties of aromatic amines in textile samples

* Concentration of this component was near or below detection limit

** See the discussion in chapter 6

From the above table it is clear that hardly any quality improvement is visible for the detected banned dyes.

6   DISCUSSION

From the reported details of analyses, it is clear that most participants treated the fabric samples according identical test methods.
Although several test details vary strongly, it is not clear which part may explain the observed spreads of the aromatic amines.

Sample #14020 is a fabric that was dyed with AZO dyes that are known to release both Benzidine and 3,3'-Dimethoxybenzidine. Sample #14021 is a fabric that was dyed with AZO dyes that are known to release 4-Chloro-o-toluidine, 4,4'-Diaminodiphenylmethane and o-Toluidine.

Both samples were used before in respective: 2011 PT iis11A01T and 2005 PT iis05A01:

Parameter	unit	#11018 in iis11A01T			#14020 in iis14A01T		
		n	average	uncertainty	n	average	uncertainty
4-Aminodiphenyl	mg/kg	54	5.2	31%	81	5.87	21%
Benzidine	mg/kg	95	72.7	18%	140	76.7	15%
3,3'-Dimethoxybenzidine	mg/kg	91	228.1	17%	147	220	21%

Table 7: comparison of results (+uncertainly) of identical samples in iis11A01T and iis14A01T

Parameter	unit	#0518 in iis05A01			#14021 in iis14A01T		
		n	average	uncertainty	n	average	uncertainty
4-Chloro-o-toluidine	mg/kg	45	459	27%	149	509	24%
4,4'-Diaminodiphenylmeth.	mg/kg	46	391	32%	145	406	21%
o-Toluidine	mg/kg	45	298	39%	152	295	32%

Table 8: comparison of results (+uncertainly) of identical samples in iis05A01 and iis14A01T

Some quality improvement is visible. The uncertainties found in the 2014 PT are all, except for 3,3'-Dimethoxybenzidine, small in comparison with the uncertainties observed in the previous round in which the samples were used.

In sample #14021, beside the three above mentioned banned aromatic amines, two other banned aromatic amines could be detected:
Surprisingly, a number of participants reported a positive test result for o-Aminoazotoluene. The detection of this aromatic amine is unforeseen, due to the poor stability of this aromatic amine that is also an AZO-derivative. EN14362-1:2012 remarks that this amine is further reduced to o-toluidine. Obviously this is not the case with the sample used in this PT. For unknown reasons, o-Aminoazotoluene is formed by reduction of the original AZO dye, but not all is directly reduced to o-toluidine. And it appears to be stable enough to be separated and detected by a significant number of laboratories. The details of analysis given by the laboratories do not give a clue for the reason why o-Aminoazotoluene could be detected by these 29 laboratories.
Also, a number of participants reported the presence of p-Chloroaniline. However, when two different separation and detection systems are used to identify the aromatic amine, it will be clear that the isomer m-Chloroaniline is present and not p-Chloroaniline. A misidentification can be made easily, as m-Chloroaniline elutes almost at the same time as p-Chloroaniline.

It can be concluded that the spread observed in this interlaboratory study is not caused by just one critical point in the analysis. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.
Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

APPENDIX 1

Determination of 4-Aminodiphenyl (CASno.92-67-1) in sample #14020; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110		5.83		-----	2439		5.6		-----
213		8.5		-----	2441		5		-----
348		7.42		-----	2442		5.92		-----
362		<5.0		-----	2452		-----		-----
551		n.d.		-----	2453		4.382		-----
1213		8.9		-----	2456		5.2		-----
2102		-----		-----	2459		-----		-----
2108		-----		-----	2469		-----		-----
2115		-----		-----	2472		5.9		-----
2121		5.08		-----	2476		n.d.		-----
2129		6.16		-----	2481		6.4		-----
2132		6.6		-----	2482		4.64		-----
2135		5.0		-----	2483		n.d.		-----
2137		n.d.		-----	2486		n.d.		-----
2139		<5		-----	2489		n.d.		-----
2146		-----		-----	2492		-----		-----
2165		<5		-----	2493		-----		-----
2166		<5		-----	2495		-----		-----
2170		-----		-----	2497		4.03		-----
2172		n.d.		-----	2508		4.24		-----
2184		<5		-----	2511		5.30		-----
2190		7.57		-----	2514		6.62		-----
2201		5.3		-----	2516		<5		-----
2213		8.0		-----	2522		n.d.		-----
2217		3.67		-----	2523		5.335		-----
2232		n.d.		-----	2528		≤10		-----
2236		5.68		-----	2532		5.1		-----
2238		5.1		-----	2534		n.d.		-----
2245		6		-----	2540		-----		-----
2246		<10		-----	2546		<20		-----
2247		n.d.		-----	2549		5.1		-----
2254		62.131	R(0.01)	-----	2553		-----		-----
2255		8.24		-----	2559		8.00		-----
2256		-----		-----	2560		7.15		-----
2265		5.1		-----	2562		-----		-----
2284		<5		-----	2565		<5		-----
2286		≤10		-----	2566		<5		-----
2287		≤10		-----	2570		-----		-----
2289		-----		-----	2581		n.d.		-----
2290		<5		-----	2582		n.d.		-----
2291		n.d.		-----	2583		6.86		-----
2293		2.541		-----	2590		-----		-----
2295		5.8		-----	2596		-----		-----
2296		-----		-----	2601		6.20		-----
2297		5.0		-----	2605		5.0		-----
2301		5.5434		-----	2613		8.02		-----
2310		5.6		-----	2614		5.37		-----
2311		5.4		-----	2617		-----		-----
2313		5.4		-----	2618		9.071		-----
2359		6.44		-----	3100		n.d.		-----
2364		6		-----	3104		<5		-----
2366		5.7		-----	3107		5.78		-----
2367		6		-----	3109		3.7		-----
2368		7		-----	3116		-----		-----
2370		5.49		-----	3117		-----		-----
2372		n.d.		-----	3118		n.d.		-----
2373		7		-----	3146		<10		-----
2375		5.8		-----	3150		7.0		-----
2379		n.d.		-----	3151		4.9		-----
2380		6.53		-----	3153		6		-----
2381		6.395		-----	3154		6.94		-----
2386		5.0		-----	3167		4.7		-----
2390		-----		-----	3172		6.4		-----
2403		6.79		-----	3176		4.46		-----
2410		-----		-----	3180		-----		-----
2413		-----		-----	3182		6.81		-----
2415		4.5		-----	3185		<5		-----
2425		5.5		-----	3190		6.7		-----
2426		n.d.		-----	3191		<10		-----
2428		6		-----	3195		4.18		-----
2429		5.80		-----	3197		-----		-----
2432		6.4		-----	3199		<10		-----
3200		n.d.		-----					

3204	n.d.	-----
3209	-----	-----
3210	-----	-----
3214	n.d.	-----
3216	4.80	-----
3218	5	-----
3220	n.d.	-----
3222	5.4	-----
3228	<5	-----
3237	6.9239	-----
3242	5.42	-----
3243	-----	-----
3248	-----	-----
8005	-----	-----

|	
normality	OK
n	81
outliers	1
mean (n)	5.868
st.dev. (n)	1.2071
R(calc.)	3.380
R(EN14362-1:12)	(2.657)
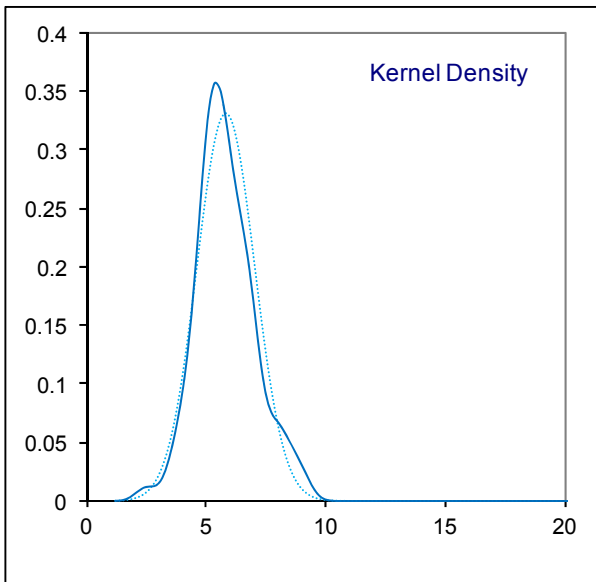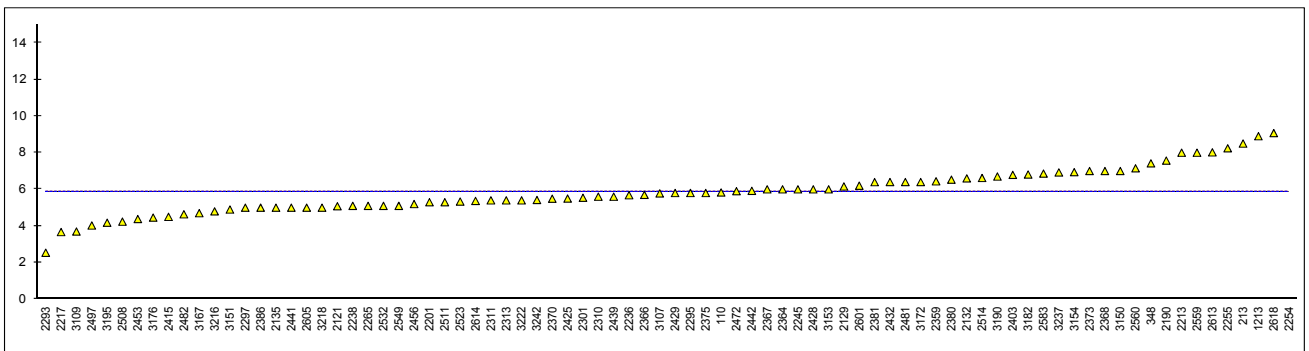
Determination of Benzidine (CASno.92-87-5) in sample #14020; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110		67.33		-0.88	2439		80.8		0.39
213		98.7		2.08	2441		68		-0.82
348		81.02		0.41	2442		73.36		-0.31
362		135	R(0.01)	5.51	2452		52.39		-2.29
551		57.05	C	-1.85	2453		18.854	R(0.01)	-5.46
1213		74.9		-0.17	2456		71.8		-0.46
2102		56.09		-1.94	2459		77.54		0.08
2108		90.0		1.26	2469		71		-0.53
2115		-----		-----	2472		82.6		0.56
2121		66.43		-0.97	2476		76.0		-0.06
2129		89.5		1.21	2481		90.2		1.28
2132		81.9		0.49	2482		79.3		0.25
2135		74.9		-0.17	2483		71.74		-0.46
2137		94		1.64	2486		61.5		-1.43
2139		75		-0.16	2489		80.88		0.40
2146		70.4		-0.59	2492		80		0.32
2165		83		0.60	2493		-----		-----
2166		45.1	R(0.01)	-2.98	2495		59.3		-1.64
2170		79.9		0.31	2497		83.74		0.67
2172		71.9		-0.45	2508		21.60	R(0.01)	-5.20
2184		86		0.88	2511		74.80		-0.18
2190		82.01		0.51	2514		80.38		0.35
2201		70.6		-0.57	2516		54.7		-2.07
2213		89.0		1.16	2522		98.6		2.07
2217		61.99		-1.38	2523		82.680		0.57
2232		64.3		-1.17	2528		72		-0.44
2236		77.28		0.06	2532		73		-0.35
2238		80.2		0.33	2534		n.d.	C, false -?	-----
2245		82		0.50	2540		68.11		-0.81
2246		70.33		-0.60	2546		75.35		-0.12
2247		85.96		0.88	2549		70.0		-0.63
2254		2085.697	R(0.01)	189.62	2553		197.7	C,R(0.01)	11.42
2255		76.5		-0.01	2559		173.4	R(0.01)	9.13
2256		75.09		-0.15	2560		79.90		0.31
2265		75.3		-0.13	2562		-----		-----
2284		71		-0.53	2565		53.3		-2.20
2286		68		-0.82	2566		75.7		-0.09
2287		64		-1.19	2570		100.80		2.28
2289		70		-0.63	2581		48		-2.70
2290		79		0.22	2582		81.28		0.44
2291		71		-0.53	2583		91.12		1.36
2293		84.438		0.73	2590		44.5	R(0.01)	-3.04
2295		72.1		-0.43	2596		22	R(0.01)	-5.16
2296		58.54		-1.71	2601		93.0		1.54
2297		68.8		-0.74	2605		80.9		0.40
2301		59.4616		-1.62	2613		70.05		-0.62
2310		80.2		0.33	2614		75.27		-0.13
2311		79.8		0.30	2617		71.24		-0.51
2313		78.7		0.19	2618		91.00		1.35
2359		92.30		1.48	3100		81.2		0.43
2364		77		0.03	3104		80		0.32
2366		66.2		-0.99	3107		113.70		3.50
2367		66		-1.01	3109		55.0		-2.04
2368		67		-0.91	3116		83.8		0.67
2370		83.8		0.67	3117		78		0.13
2372		76.3		-0.03	3118		32.51	R(0.01)	-4.17
2373		64		-1.19	3146		59		-1.67
2375		93.8		1.62	3150		79.0		0.22
2379		84.30		0.72	3151		76		-0.06
2380		102.35		2.42	3153		85		0.79
2381		103.064		2.49	3154		105.94		2.76
2386		80.4		0.35	3167		81.3		0.44
2390		40.410	R(0.01)	-3.42	3172		81		0.41
2403		79.57		0.27	3176		71.05		-0.53
2410		75		-0.16	3180		29.24	R(0.01)	-4.48
2413		2.04	R(0.01)	-7.04	3182		81.25		0.43
2415		65.1		-1.09	3185		83.1		0.61
2425		70.57		-0.57	3190		81.7		0.48
2426		70.4648		-0.58	3191		60		-1.57
2428		74		-0.25	3195		65.5	C	-1.05
2429		63.75		-1.22	3197		81		0.41
2432		76.3		-0.03	3199		91.5		1.40
3200		79		0.22					

3204	31.0	R(0.01)	-4.31
3209	96.6		1.88
3210	72.1		-0.43
3214	74.43		-0.21
3216	52.57		-2.27
3218	86	C	0.88
3220	130.0	R(0.01)	5.03
3222	76.1		-0.05
3228	83		0.60
3237	78.0789		0.13
3242	65.96		-1.01
3243	73		-0.35
3248	90.8		1.33
8005	84.2		0.71

normality	OK	
n	140	
outliers	15	
mean (n)	76.658	
st.dev. (n)	11.4450	
R(calc.)	32.046	
R(EN14362-1:12)	29.667	

Lab 551 : first reported 53.36
Lab 2534: first reported 45
Lab 2553: first reported 400.3
Lab 3195: first reported 175.2
Lab 3218: first reported 228
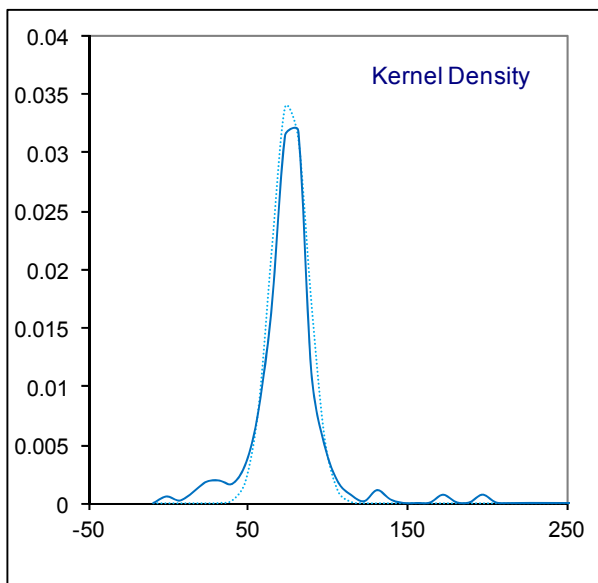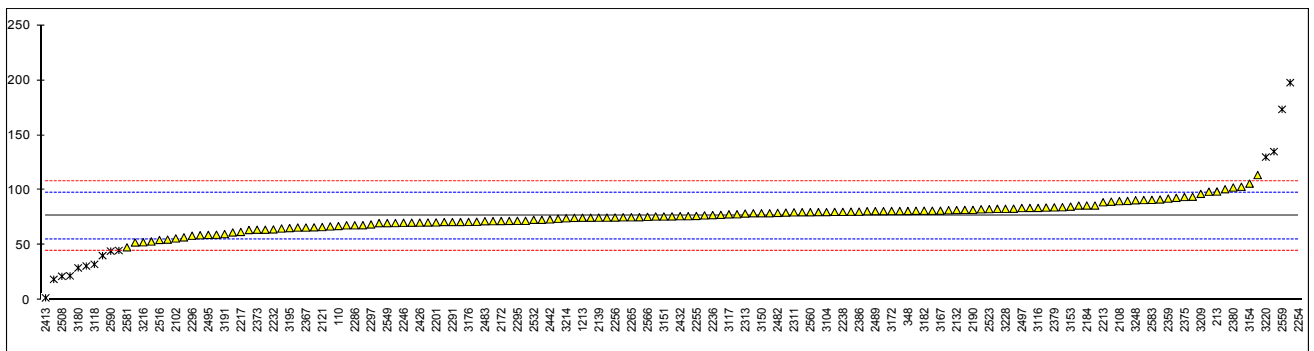
Determination of 3,3'-Dimethoxybenzidine (CASno.119-90-4) in sample #14020; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110		189.81		-1.10	2439		242.6		0.82
213		282.5		2.26	2441		214		-0.22
348		223.9		0.14	2442		208.23		-0.43
362		248		1.01	2452		189.64		-1.10
551		343.41	C	4.47	2453		17.025	C,R(0.05)	-7.36
1213		280.3		2.18	2456		268.40		1.75
2102		89.68		-4.73	2459		197.58		-0.82
2108		272.4		1.90	2469		167		-1.92
2115		-----		-----	2472		169.1	C	-1.85
2121		213.28		-0.25	2476		399.4	R(0.05)	6.50
2129		278		2.10	2481		223.4		0.12
2132		214.8		-0.19	2482		166		-1.96
2135		274.5		1.97	2483		n.d.	false -?	-----
2137		230		0.36	2486		200		-0.73
2139		235		0.54	2489		239.80		0.71
2146		184.4		-1.29	2492		213		-0.26
2165		211		-0.33	2493		-----		-----
2166		125.9		-3.41	2495		181.0		-1.42
2170		238.0		0.65	2497		165.42		-1.98
2172		201		-0.69	2508		95.21		-4.53
2184		218		-0.08	2511		200.20		-0.72
2190		192.18		-1.01	2514		241.37		0.77
2201		200.5		-0.71	2516		161.4		-2.13
2213		299		2.86	2522		262.0		1.52
2217		192.14		-1.01	2523		229.318		0.33
2232		199.5		-0.75	2528		199		-0.77
2236		219.88		-0.01	2532		244		0.87
2238		224.0		0.14	2534		30	C,R(0.05)	-6.89
2245		248		1.01	2540		248.67		1.04
2246		245.34		0.91	2546		<20	false -?	<-7.25
2247		236.75		0.60	2549		208		-0.44
2254		599.698	R(0.01)	13.76	2553		38.3	R(0.05)	-6.59
2255		219.8		-0.01	2559		550.5	R(0.01)	11.97
2256		240.20		0.73	2560		250.00		1.08
2265		220.2		0.00	2562		-----		-----
2284		194		-0.95	2565		148.0		-2.61
2286		208		-0.44	2566		256		1.30
2287		195		-0.91	2570		200.07		-0.73
2289		212		-0.29	2581		299		2.86
2290		228		0.29	2582		228.56		0.31
2291		201		-0.69	2583		279.18		2.14
2293		196.887		-0.84	2590		150		-2.54
2295		217		-0.11	2596		126		-3.41
2296		184.32		-1.30	2601		262.0		1.52
2297		195		-0.91	2605		230.3		0.37
2301		198.2054		-0.79	2613		220.06		0.00
2310		235		0.54	2614		250.3		1.09
2311		226		0.21	2617		225.62		0.20
2313		235		0.54	2618		250.00		1.08
2359		249.65		1.07	3100		233.0		0.47
2364		190		-1.09	3104		223		0.10
2366		183.0		-1.34	3107		335.89		4.20
2367		182		-1.38	3109		195.6		-0.89
2368		199		-0.77	3116		240.7		0.75
2370		239		0.68	3117		191		-1.05
2372		224		0.14	3118		79.03		-5.11
2373		176		-1.60	3146		142		-2.83
2375		265.1		1.63	3150		225		0.18
2379		245.24		0.91	3151		214		-0.22
2380		248.16		1.02	3153		234		0.50
2381		246.416		0.95	3154		272.64		1.90
2386		203		-0.62	3167		214		-0.22
2390		114.406		-3.83	3172		201		-0.69
2403		242.48		0.81	3176		242.13		0.80
2410		220		0.00	3180		106.98		-4.10
2413		2.75	R(0.05)	-7.88	3182		331.98		4.05
2415		251.7		1.14	3185		218.2		-0.07
2425		245.90		0.93	3190		214.2		-0.21
2426		194.1030		-0.94	3191		170		-1.82
2428		202		-0.66	3195		175.2		-1.63
2429		216.48		-0.13	3197		232		0.43
2432		268.0		1.74	3199		265.2		1.63
3200		233		0.47					

3204	148		-2.61
3209	318.0		3.55
3210	198		-0.80
3214	220.90		0.03
3216	297.89		2.82
3218	228	C	0.29
3220	354.0		4.85
3222	238.1		0.65
3228	208		-0.44
3237	269.5934		1.79
3242	250.06		1.09
3243	207		-0.48
3248	257.2		1.34
8005	239.8		0.71

normality	suspect
n	147
outliers	7
mean (n)	220.110
st.dev. (n)	45.7725
R(calc.)	128.163
R(EN14362-1:12)	77.259

Lab 551: first reported 797.56
Lab 2453: first reported n.d.
Lab 2472: first reported 422.7
Lab 2534: first reported 196
Lab 3218: first reported 86
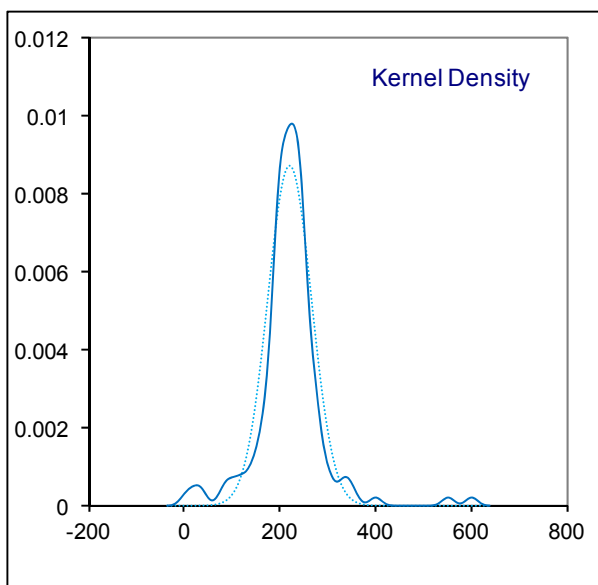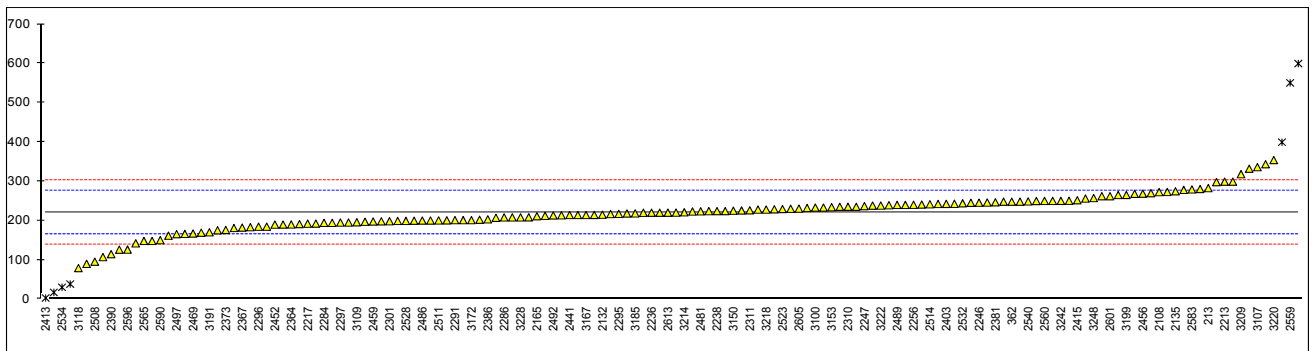
Determination of 4-Chloro-o-toluidine (CASno.95-69-2) in sample #14021; results in mg/kg

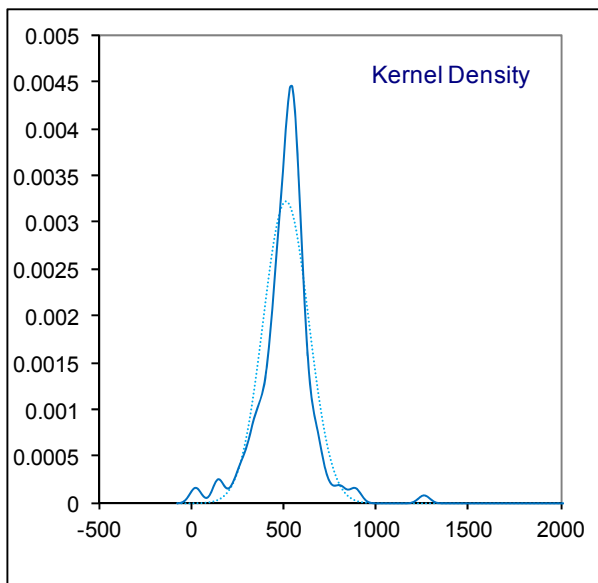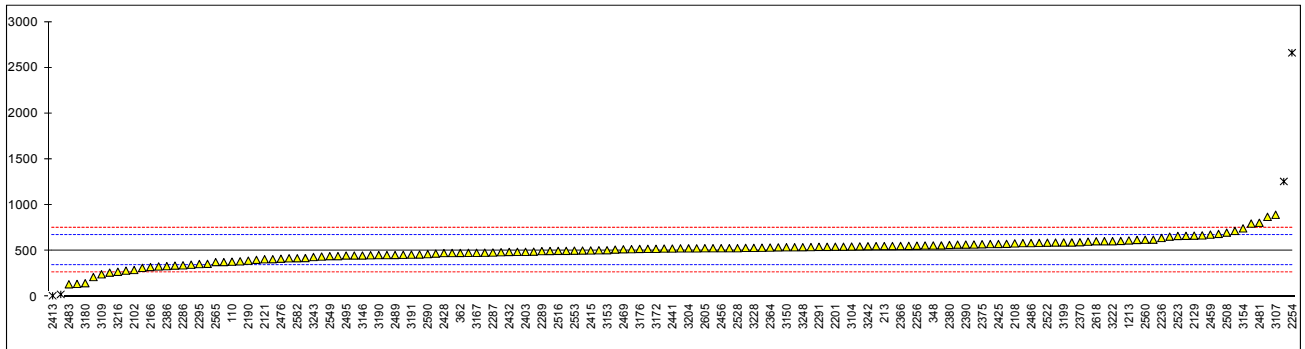| lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110		382.73		-1.53	2439		718.1		2.55
213		553.2		0.54	2441		526		0.21
348		559.54		0.62	2442		577.68		0.84
362		478		-0.37	2452		575.44		0.81
551		546.27	C	0.46	2453		25.810	C,R(0.05)	-5.87
1213		614.6		1.29	2456		530.0		0.26
2102		291.96		-2.63	2459		678.35		2.07
2108		582.1		0.89	2469		518		0.12
2115		-----		-----	2472		457.9		-0.62
2121		409.60		-1.20	2476		415.1		-1.14
2129		667		1.93	2481		807.1		3.63
2132		585.4		0.93	2482		285		-2.72
2135		798.6		3.53	2483		136.11	C	-4.53
2137		656		1.79	2486		586		0.94
2139		872		4.42	2489		455.36		-0.65
2146		669.5		1.96	2492		559		0.61
2165		545		0.44	2493		-----		-----
2166		324.4		-2.24	2495		450		-0.71
2170		491.9		-0.20	2497		315.23		-2.35
2172		523		0.18	2508		699.71		2.33
2184		539		0.37	2511		499.5		-0.11
2190		393.79		-1.40	2514		587.45		0.96
2201		546.2		0.46	2516		500.7		-0.10
2213		331		-2.16	2522		589.9		0.99
2217		685.75		2.16	2523		663.576		1.89
2232		386.9		-1.48	2528		530		0.26
2236		641.60		1.62	2532		479		-0.36
2238		542.0		0.41	2534		140	C	-4.48
2245		422		-1.05	2540		359.07		-1.82
2246		477.61		-0.38	2546		<20	false -?	<-5.94
2247		438.57		-0.85	2549		443		-0.80
2254		2661.759	R(0.01)	26.18	2553		503.0		-0.07
2255		591.3		1.01	2559		1258.0	R(0.01)	9.11
2256		557.10		0.59	2560		622.00		1.38
2265		420.1		-1.08	2562		-----		-----
2284		530		0.26	2565		378.4		-1.58
2286		343		-2.01	2566		450		-0.71
2287		483		-0.31	2570		454.84		-0.65
2289		498		-0.13	2581		340		-2.05
2290		623		1.39	2582		420.27		-1.07
2291		545		0.44	2583		378.43		-1.58
2293		215.279		-3.57	2590		465		-0.53
2295		357		-1.84	2596		461		-0.58
2296		525.94		0.21	2601		-----		-----
2297		533		0.30	2605		529.8		0.26
2301		410.0834		-1.20	2613		592.12		1.02
2310		606		1.19	2614		453.2		-0.67
2311		601		1.12	2617		514.58		0.07
2313		620		1.36	2618		605.454		1.18
2359		570.23		0.75	3100		549.7		0.50
2364		537		0.35	3104		548		0.48
2366		554.6		0.56	3107		894.73		4.70
2367		528		0.24	3109		245.3		-3.20
2368		519		0.13	3116		552.2		0.53
2370		595		1.05	3117		609		1.22
2372		350		-1.93	3118		443.56		-0.79
2373		501		-0.09	3146		450		-0.71
2375		572.9		0.78	3150		540		0.38
2379		529.98		0.26	3151		504		-0.05
2380		565.34		0.69	3153		508		-0.01
2381		568.902		0.73	3154		746.12		2.89
2386		334		-2.12	3167		479		-0.36
2390		569.375		0.74	3172		523		0.18
2403		489.99		-0.23	3176		522.23		0.17
2410		403		-1.28	3180		148.20		-4.38
2413		11.10	R(0.05)	-6.05	3182		553.51		0.55
2415		505.1		-0.04	3185		534.9		0.32
2425		575.88		0.82	3190		454.2		-0.66
2426		485.7143		-0.28	3191		460		-0.59
2428		477		-0.38	3195		-----		-----
2429		480.64		-0.34	3197		471		-0.46
2432		489.0		-0.24	3199		592.1		1.02
3200		540		0.38					

3204	528	0.24
3209	559.9	0.62
3210	507	-0.02
3214	489.80	-0.23
3216	272.5	-2.87
3218	528	0.24
3220	263.0	-2.99
3222	606.0	1.19
3228	533	0.30
3237	664.9245	1.90
3242	551.5	0.52
3243	434	-0.91
3248	541.0	0.40
8005	555.2	0.57

normality	suspect
n	149
outliers	4
mean (n)	508.514
st.dev. (n)	123.3988
R(calc.)	345.517
R(EN14362-1:12)	230.255

Lab 551: first reported 518.18
Lab 2453: first reported 25.810
Lab 2483: first reported 1265.87
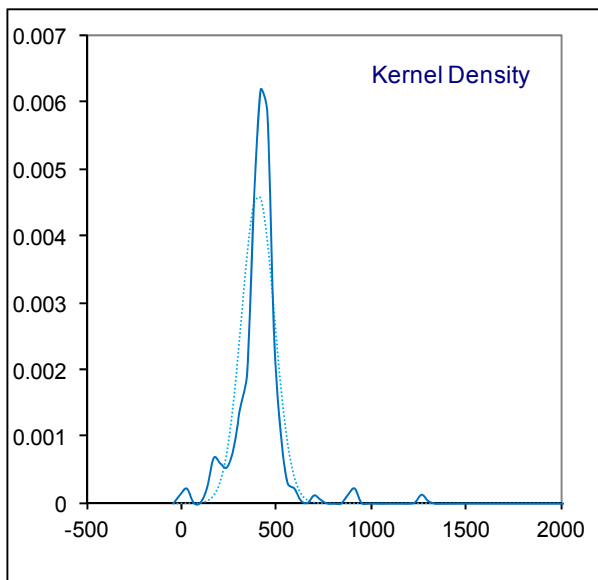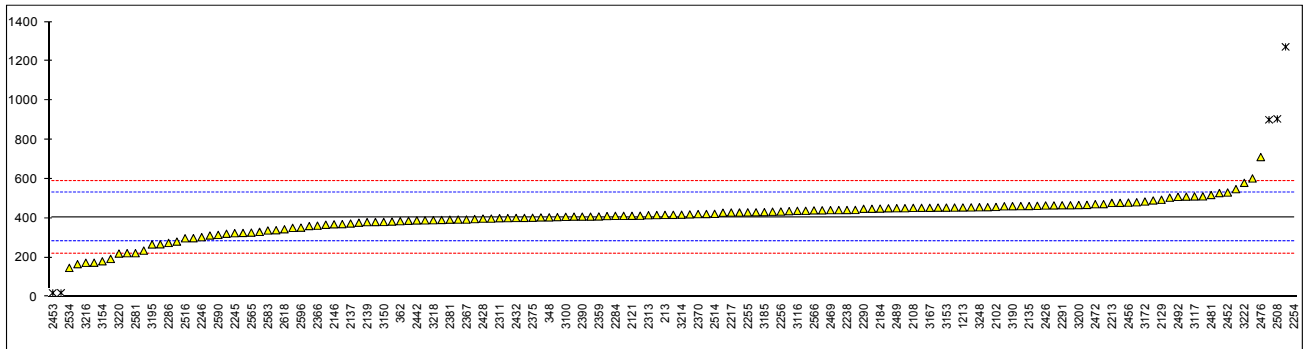Lab 2534: first reported 1227

Determination of 4,4'-Diaminodiphenylmethane (CASno.101-77-9) in sample #14021; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110		n.d.	false -?	-----	2439		478.3		1.17
213		416.9		0.18	2441		404		-0.03
348		404.24		-0.03	2442		388.79		-0.28
362		385		-0.34	2452		530.93		2.03
551		528.07	C	1.98	2453		19.373	C,R(0.01)	-6.29
1213		454.7		0.79	2456		479.4		1.19
2102		457.29		0.83	2459		547.98		2.31
2108		452.5		0.75	2469		441		0.57
2115		-----		-----	2472		471.3		1.06
2121		412.25		0.10	2476		711.4		4.96
2129		494		1.43	2481		518.2		1.82
2132		456.2		0.81	2482		235		-2.78
2135		462.0		0.91	2483		173.57		-3.78
2137		373	C	-0.54	2486		339		-1.09
2139		380		-0.42	2489		450.86		0.73
2146		369.0		-0.60	2492		509		1.67
2165		454		0.78	2493		-----		-----
2166		298.3		-1.75	2495		325		-1.32
2170		481.1		1.22	2497		366.75		-0.64
2172		393		-0.21	2508		905.69	R(0.01)	8.12
2184		449		0.70	2511		460.92		0.89
2190		511.21		1.71	2514		422.23		0.26
2201		465.2		0.96	2516		297.7		-1.76
2213		478		1.17	2522		490.5		1.37
2217		428.86		0.37	2523		504.572		1.60
2232		350.9		-0.90	2528		510		1.69
2236		471.83		1.07	2532		451		0.73
2238		441.7		0.58	2534		147	C	-4.21
2245		324		-1.34	2540		377.12		-0.47
2246		303.56		-1.67	2546		<20	false -?	<-6.28
2247		453.0		0.76	2549		455		0.80
2254		8914.056	R(0.01)	138.35	2553		380.6		-0.41
2255		429.8		0.39	2559		1272.4	R(0.01)	14.09
2256		433.82		0.45	2560		441.30		0.57
2265		>300.0		-----	2562		-----		-----
2284		412		0.10	2565		326.8		-1.29
2286		274		-2.15	2566		440		0.55
2287		466		0.97	2570		419.50		0.22
2289		463		0.93	2581		222		-2.99
2290		448		0.68	2582		461.45		0.90
2291		466		0.97	2583		337.50		-1.12
2293		192.677		-3.47	2590		315		-1.48
2295		222		-2.99	2596		352		-0.88
2296		602.45		3.19	2601		417.3		0.18
2297		391		-0.25	2605		450.5		0.72
2301		369.9881		-0.59	2613		400.34		-0.09
2310		408		0.03	2614		454.4		0.79
2311		400		-0.10	2617		416.10		0.16
2313		415		0.14	2618		343.636		-1.02
2359		408.79		0.04	3100		407.1		0.02
2364		436		0.49	3104		428		0.36
2366		362.0		-0.72	3107		901.40	R(0.01)	8.05
2367		393		-0.21	3109		280.9		-2.04
2368		433		0.44	3116		437.6		0.51
2370		421		0.24	3117		511		1.71
2372		267		-2.26	3118		n.d.	false -?	-----
2373		398		-0.13	3146		412		0.10
2375		402.4		-0.06	3150		381		-0.41
2379		401.68		-0.07	3151		430		0.39
2380		395.67		-0.17	3153		454		0.78
2381		392.627		-0.22	3154		180.70		-3.67
2386		387		-0.31	3167		453		0.76
2390		408.041		0.03	3172		485		1.28
2403		382.91		-0.38	3176		330.40		-1.23
2410		389		-0.28	3180		19.76	R(0.01)	-6.28
2413		-----		-----	3182		421.38		0.25
2415		405.6		-0.01	3185		431.1		0.41
2425		408.06		0.03	3190		461.3		0.90
2426		464.8810		0.96	3191		360		-0.75
2428		398		-0.13	3195		266.5		-2.27
2429		429.00		0.37	3197		448		0.68
2432		401.2		-0.08	3199		467.8		1.00
3200		467		0.99					

3204	166	-3.90
3209	437.8	0.52
3210	321	-1.38
3214	417.83	0.19
3216	173.04	-3.79
3218	390	-0.26
3220	220.0	-3.03
3222	580.0	2.83
3228	442	0.58
3237	410.8594	0.08
3242	412.8	0.11
3243	312	-1.53
3248	456.0	0.81
8005	440.1	0.55

normality	suspect
n	145
outliers	6
mean (n)	406.107
st.dev. (n)	86.2675
R(calc.)	241.549
R(EN14362-1:12)	172.189

Lab 551: first reported 1602.29
Lab 2137: first reported n.d.
Lab 2453: first reported 11.912
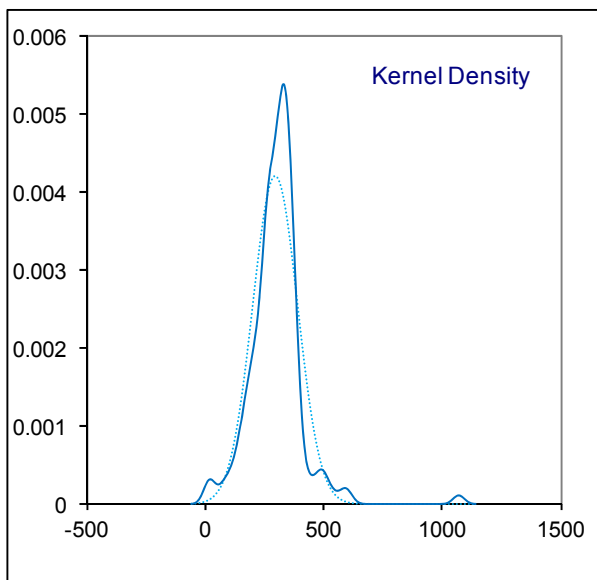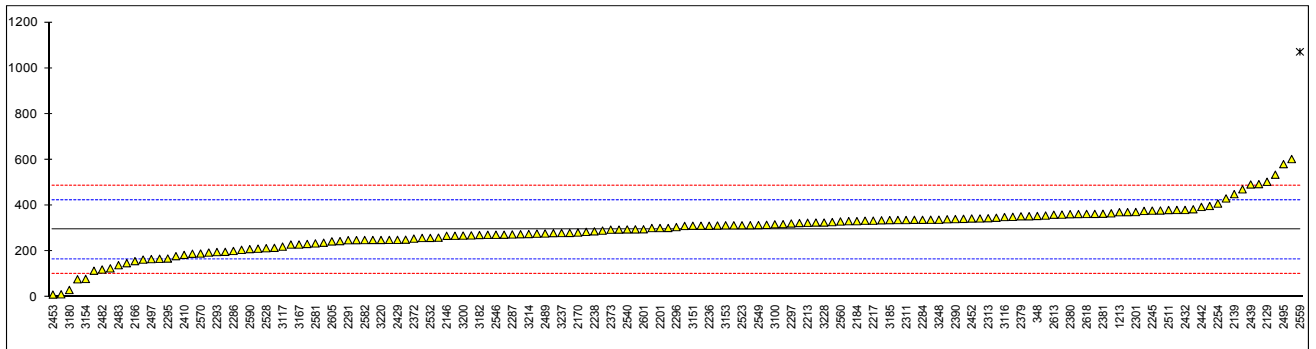Lab 2534: first reported 1430

Determination of *o*-Toluidine (CASno.95-53-4) in sample #14021; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110		n.d.	false -?	-----	2439		492.2		3.07
213		398.3		1.61	2441		332		0.58
348		354.61		0.93	2442		394.30		1.55
362		384		1.39	2452		343.73		0.76
551		296.23	C	0.02	2453		11.615		-4.40
1213		371.0		1.19	2456		230.0		-1.01
2102		-----		-----	2459		335.93		0.64
2108		249.4		-0.70	2469		334		0.61
2115		-----		-----	2472		164.3		-2.03
2121		215.68		-1.23	2476		342.3		0.74
2129		504		3.26	2481		603.2		4.80
2132		280.8		-0.22	2482		121		-2.70
2135		471.2		2.75	2483		140.10	C	-2.40
2137		535		3.74	2486		273		-0.34
2139		450		2.42	2489		278.40		-0.25
2146		268.2		-0.41	2492		326		0.49
2165		337		0.66	2493		-----		-----
2166		157.8		-2.13	2495		581		4.45
2170		282.4		-0.19	2497		166.47		-1.99
2172		324		0.46	2508		493.65		3.10
2184		332		0.58	2511		380.72		1.34
2190		179.44		-1.79	2514		363.82		1.08
2201		302.1		0.12	2516		277.7		-0.26
2213		325		0.47	2522		431.4		2.13
2217		334.17		0.61	2523		314.196		0.30
2232		378.6		1.31	2528		214		-1.25
2236		312.06		0.27	2532		259		-0.55
2238		287.7		-0.11	2534		195	C	-1.55
2245		378		1.30	2540		296.00		0.02
2246		258.67		-0.56	2546		272.8		-0.34
2247		268.31		-0.41	2549		315		0.32
2254		408.983		1.78	2553		198.1		-1.50
2255		371.5		1.20	2559		1071.2	R(0.01)	12.08
2256		353.51		0.92	2560		330.70		0.56
2265		250.2		-0.69	2562		-----		-----
2284		338		0.67	2565		207.0		-1.36
2286		202		-1.44	2566		270		-0.38
2287		274		-0.32	2570		190.80		-1.62
2289		238		-0.88	2581		235		-0.93
2290		381		1.34	2582		249.58		-0.70
2291		249		-0.71	2583		78.31		-3.36
2293		197.229		-1.52	2590		210		-1.32
2295		169		-1.95	2596		-----		-----
2296		306.12		0.18	2601		296.8		0.03
2297		322		0.43	2605		243.8		-0.79
2301		371.9730		1.20	2613		360.35		1.02
2310		338		0.67	2614		272.5		-0.34
2311		337		0.66	2617		232.56		-0.97
2313		345		0.78	2618		363.636		1.07
2359		366.43		1.12	3100		318.4		0.37
2364		319		0.38	3104		351		0.88
2366		311.2		0.26	3107		338.24		0.68
2367		328		0.52	3109		115.7		-2.78
2368		302		0.11	3116		350.1		0.86
2370		316		0.33	3117		221		-1.15
2372		256		-0.60	3118		285.20		-0.15
2373		295		0.01	3146		275		-0.31
2375		376.9		1.28	3150		291		-0.06
2379		353.31		0.91	3151		312		0.27
2380		362.50		1.06	3153		314		0.30
2381		364.486		1.09	3154		79.78		-3.34
2386		126		-2.62	3167		230		-1.01
2390		341.259		0.73	3172		260		-0.54
2403		314.31		0.31	3176		295.32		0.01
2410		185		-1.71	3180		31.70		-4.09
2413		12.86		-4.38	3182		271.48		-0.36
2415		343.8		0.76	3185		336.7		0.65
2425		360.67		1.03	3190		251.3		-0.67
2426		302.3810		0.12	3191		280		-0.23
2428		212		-1.29	3195		167.9		-1.97
2429		250.24		-0.69	3197		313		0.29
2432		381.0		1.34	3199		314.0		0.30
3200		269		-0.40					

3204	245	-0.77
3209	363.1	1.06
3210	250	-0.69
3214	275.89	-0.29
3216	148.81	-2.27
3218	312	0.27
3220	250.0	-0.69
3222	356.6	0.96
3228	326	0.49
3237	280.1974	-0.22
3242	340.3	0.71
3243	190	-1.63
3248	338.4	0.68
8005	346.9	0.81

normality	suspect
n	152
outliers	1
mean (n)	294.642
st.dev. (n)	94.8910
R(calc.)	265.695
R(EN14362-1:12)	180.026

Lab 551: first reported 350.17
Lab 2483: first reported 462.08
Lab 2534: first reported 1760

Kernel Density

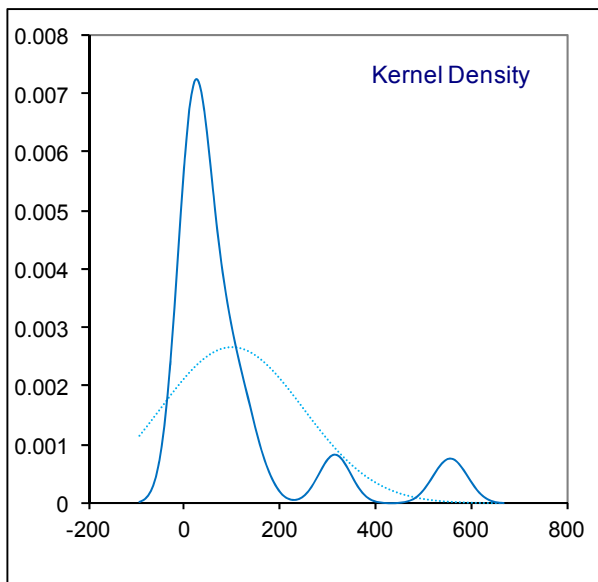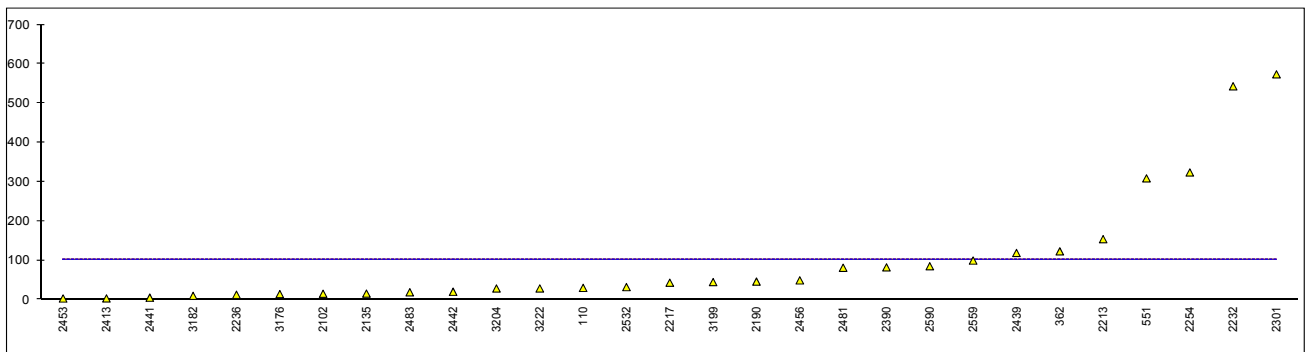## Determination of *o*-Aminoazotoluene (CASno.97-56-3) in sample #14021; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110		29.93		-----	2439		118.9`		-----
213		-----		-----	2441		5		-----
348		<5		-----	2442		20.05		-----
362		123		-----	2452		-----		-----
551		308.79	C	-----	2453		2.927		-----
1213		-----		-----	2456		49.1		-----
2102		14.97		-----	2459		-----		-----
2108		-----		-----	2469		-----		-----
2115		-----		-----	2472		<5		-----
2121		-----		-----	2476		n.d.		-----
2129		-----		-----	2481		81.1		-----
2132		<5		-----	2482		-----		-----
2135		15.4		-----	2483		18.76	C	-----
2137		n.d.		-----	2486		<5		-----
2139		<5		-----	2489		n.d.		-----
2146		-----		-----	2492		-----		-----
2165		<5		-----	2493		-----		-----
2166		<5		-----	2495		-----		-----
2170		-----		-----	2497		-----		-----
2172		n.d.		-----	2508		-----		-----
2184		<5		-----	2511		-----		-----
2190		46.00		-----	2514		-----		-----
2201		<5		-----	2516		<5		-----
2213		154		-----	2522		n.d.		-----
2217		43.11		-----	2523		n.d.		-----
2232		543.2		-----	2528		≤10		-----
2236		12.38		-----	2532		32		-----
2238		<5		-----	2534		n.d.		-----
2245		-----		-----	2540		-----		-----
2246		<10		-----	2546		<20		-----
2247		n.d.		-----	2549		n.d.		-----
2254		323.799		-----	2553		-----		-----
2255		-----		-----	2559		99.5		-----
2256		-----		-----	2560		<5		-----
2265		-----		-----	2562		-----		-----
2284		<5		-----	2565		<5		-----
2286		≤10		-----	2566		<5		-----
2287		≤10		-----	2570		-----		-----
2289		-----		-----	2581		n.d.		-----
2290		<5		-----	2582		n.d.		-----
2291		n.d.		-----	2583		-----		-----
2293		n.d.		-----	2590		85		-----
2295		-----		-----	2596		-----		-----
2296		-----		-----	2601		-----		-----
2297		<5		-----	2605		-----		-----
2301		573.5909		-----	2613		-----		-----
2310		n.d.		-----	2614		<5		-----
2311		n.d.		-----	2617		-----		-----
2313		n.d.		-----	2618		n.d.		-----
2359		n.d.		-----	3100		n.d.		-----
2364		n.d.		-----	3104		<5		-----
2366		n.d.		-----	3107		-----		-----
2367		n.d.		-----	3109		-----		-----
2368		n.d.		-----	3116		-----		-----
2370		n.d.		-----	3117		-----		-----
2372		n.d.		-----	3118		n.d.		-----
2373		n.d.		-----	3146		<10		-----
2375		-----		-----	3150		-----		-----
2379		n.d.		-----	3151		-----		-----
2380		-----		-----	3153		<5		-----
2381		-----		-----	3154		-----		-----
2386		<5		-----	3167		-----		-----
2390		82.20		-----	3172		-----		-----
2403		n.d.		-----	3176		14.20		-----
2410		-----		-----	3180		-----		-----
2413		3.17		-----	3182		9.73		-----
2415		-----		-----	3185		<5		-----
2425		n.d.		-----	3190		n.d.		-----
2426		n.d.		-----	3191		<10		-----
2428		n.d.		-----	3195		<1		-----
2429		n.d.		-----	3197		-----		-----
2432		-----		-----	3199		44.7		-----
3200		n.d.		-----					

3204	28.4	-----
3209	-----	-----
3210	-----	-----
3214	n.d.	-----
3216	n.d.	-----
3218	<5	-----
3220	n.d.	-----
3222	28.5	-----
3228	<5	-----
3237	-----	-----
3242	n.d.	-----
3243	-----	-----
3248	-----	-----
8005	-----	-----

normality	not OK
n	29
outliers	0
mean (n)	n.a.
st.dev. (n)	n.a.
R(calc.)	n.a.
R(EN14362-1:12)	n.a.

Lab 551: first reported 528.55
Lab 2483: first reported 34.31

Determination of *p*-Chloraniline (CASno.106-74-8) in sample #14021; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110		n.d.		-----	2439		1562.8		4.85
213		-----		-----	2441		5		-7.48
348		<5		-----	2442		-----		-----
362		<5.0		-----	2452		1243.31		2.32
551		1157.06	C	1.64	2453		13.527		-7.41
1213		<5.0		-----	2456		996.7		0.37
2102		359.21		-4.67	2459		-----		-----
2108		-----		-----	2469		-----		-----
2115		-----		-----	2472		<5		-----
2121		-----		-----	2476		n.d.		-----
2129		-----		-----	2481		2516.1		12.39
2132		<5		-----	2482		-----		-----
2135		-----		-----	2483		n.d.		-----
2137		n.d.		-----	2486		<5		-----
2139		<5		-----	2489		n.d.		-----
2146		-----		-----	2492		-----		-----
2165		<5		-----	2493		-----		-----
2166		<5		-----	2495		-----		-----
2170		-----		-----	2497		-----		-----
2172		n.d.		-----	2508		1653.47		5.56
2184		<5		-----	2511		-----		-----
2190		678.41		-2.15	2514		-----		-----
2201		<5		-----	2516		<5		-----
2213		n.d.		-----	2522		n.d.		-----
2217		n.d.		-----	2523		n.d.		-----
2232		n.d.		-----	2528		≤10		-----
2236		1325.64		2.97	2532		n.d.		-----
2238		<5		-----	2534		751	C	-1.58
2245		-----		-----	2540		-----		-----
2246		<10		-----	2546		<20		-----
2247		n.d.		-----	2549		n.d.		-----
2254		7899.797	R(0.01)	54.98	2553		1143.8		1.53
2255		-----		-----	2559		1976.6		8.12
2256		-----		-----	2560		<5		-----
2265		-----		-----	2562		-----		-----
2284		<5		-----	2565		<5		-----
2286		≤10		-----	2566		<5		-----
2287		≤10		-----	2570		-----		-----
2289		-----		-----	2581		917		-0.26
2290		<5		-----	2582		n.d.		-----
2291		n.d.		-----	2583		423.01		-4.17
2293		406.712		-4.30	2590		1115		1.30
2295		795		-1.23	2596		1227		2.19
2296		1129.78		1.42	2601		-----		-----
2297		<5		-----	2605		-----		-----
2301		-----		-----	2613		-----		-----
2310		n.d.		-----	2614		<5		-----
2311		n.d.		-----	2617		-----		-----
2313		n.d.		-----	2618		n.d.		-----
2359		n.d.		-----	3100		n.d.		-----
2364		n.d.		-----	3104		<5		-----
2366		n.d.		-----	3107		-----		-----
2367		n.d.		-----	3109		-----		-----
2368		n.d.		-----	3116		-----		-----
2370		n.d.		-----	3117		-----		-----
2372		n.d.		-----	3118		917.55		-0.26
2373		n.d.		-----	3146		<10		-----
2375		-----		-----	3150		-----		-----
2379		n.d.		-----	3151		isomer detected		-----
2380		-----		-----	3153		<5		-----
2381		-----		-----	3154		-----		-----
2386		<5		-----	3167		-----		-----
2390		1401.173		3.57	3172		-----		-----
2403		n.d.		-----	3176		-----		-----
2410		-----		-----	3180		33.37		-7.25
2413		24.16		-7.33	3182		1161.03		1.67
2415		-----		-----	3185		<5		-----
2425		n.d.		-----	3190		n.d.		-----
2426		n.d.		-----	3191		<10		-----
2428		n.d.		-----	3195		<1		-----
2429		n.d.		-----	3197		-----		-----
2432		-----		-----	3199		<10		-----
3200		n.d.		-----					

3204	1077	1.00
3209	-----	-----
3210	-----	-----
3214	n.d.	-----
3216	534.46	-3.29
3218	<5	-----
3220	n.d.	-----
3222	1009.2	0.47
3228	<5	-----
3237	-----	-----
3242	n.d.	-----
3243	-----	-----
3248	-----	-----
8005	-----	-----

normality	OK
n	29
outliers	1
mean (n)	n.a.
st.dev. (n)	n.a.
R(calc.)	n.a.
R(EN14362-1:12)	n.a.

Lab 551 : first reported 718.75
Lab 2534: first reported 5658
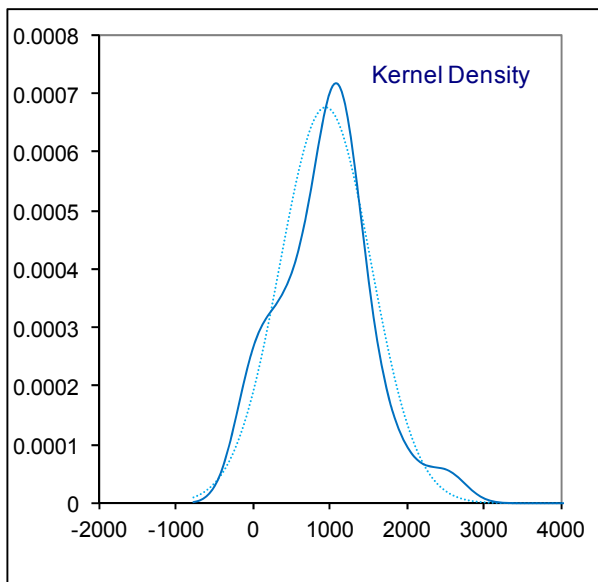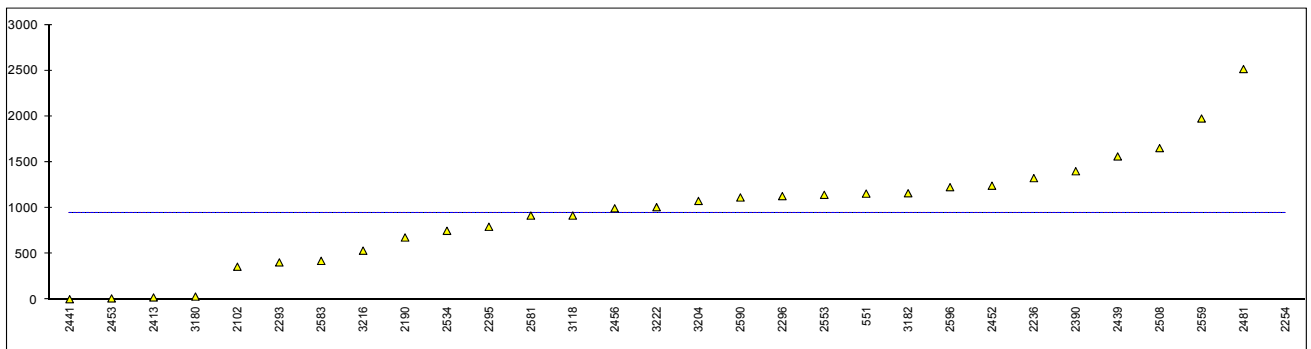
APPENDIX 2

Summary of other reported aromatic amines in sample #14020

lab	method
551	o-anisidine 7.30 mg/kg;
2135	o-anisidine 1.2 mg/kg;
2190	4-chloro-o-toluidine 1.17 mg/kg; p-cresidine 1.24 mg.kg; o-anisidine 1.77 mg/kg;
2217	o-anisidine 1.02 mg/kg;
2293	o-anisidine 0.610 mg/kg;
2413	2-amino-4-nitrotoluene 2.14 mg/kg; 4,4'-diaminodiphenylether 2.54 mg/kg; 4,4'-diaminodiphenylsulfide 3.31 mg/kg;
2453	2,4-diaminoanisol 1.142 mg/kg; o-anisidine 0.538 mg/kg; 4-aminoazobenzene 0.093 mg/kg;
2456	4,4'-diaminodiphenylmethane 1.5 mg/kg; o-anisidine 1.8 mg/kg;
3216	o-anisidine 0.50 mg/kg;
3222	o-anisidine 2.7 mg/kg;

Summary of other reported aromatic amines in sample #14021

lab	method
110	3,3'-dimethylbenzidine : 156.83 mg/kg; 4,4'-diamino-3,3'-dichlorodiphenylmethane: 256.12 mg/kg;
2190	4-aminodiphenyl 1.20 mg/kg; Benzidine 3.30 mg/kg; 2-naphthylamine 1.52 mg/kg; 3,3'-dimethoxybenzidine 3.01 mg/kg; 3,3'-dimethylbenzidine 1.84 mg/kg;
2293	2,4-xylidene 0.357 mg/kg;
2453	Benzidine 6.146 mg/kg; 3,3'-dimethoxybenzidine 11.020 mg/kg; 4-aminoazobenzene 0.133 mg/kg;
2456	Benzidine 1.7 mg/kg; 3,3'-dimethoxybenzidine 3.7 mg/kg; 3,3'-dimethylbenzidine 1.9 mg/kg;
2481	4-aminoazobenzene 10.1 mg/kg;
2483	2,4'-diaminotoluene 77.72 mg/kg;
2495	3,3'-dimethoxybenzidine 8.2 mg/kg;
2590	3'3,-dimethoxybenzidine 16 mg/kg;
3222	Benzidine 3.5 mg/kg; 3,3'-dimethoxybenzidine 5.9 mg/kg; 3,3'-dimethylbenzidine 1.8 mg/kg;

APPENDIX 3

Analytical details

Lab	sample intake	Chlorobenz. Extraction	Reductive cleavage	column used	solvent used	Evap. temp.	solvent for dissolve residue	Chromatographic analysis
110	-	-	30 min	Agilent ChemElt	MTBE	45	Methanol	HPLC/DAD - GC/MSD
213	1g	Yes	30 min	quick meth	Ethylacetate	58	Methanol	HPLC/DAD - GC/MSD
348	0.5 g	No	30 min	--	ethylacetate	--	--	GC/MSD - HPLC/MS/MS
362	1 - 0.75 g	No	30 min	--	-	--	Ethylacetate	GC/MSD
551	1.0 g	Yes	30 min	--	Ethylacetate	45	Methanol	GC/MSD
1213	0.25 - 0.5 g	no	30 min	--	ETBE	--	--	GC/MSD
2102	0.5 g	no	1.5 hrs	--	MTBE	--	--	GC/MSD
2108	0.114 g	no	30 min	Chromabond XTR	MTBE	50	Methanol	HPLC/DAD
2115								
2121	0.7 - 0.8 g	no	30 min	Extrelut NT20	MTBE	45	Methanol	LC/MS/DAD
2129	0.50 g	Xylene	30 min	Chromabond XTR	MTBE	40	--	HPLC/DAD - GC/MSD
2132	1 g	no	30 min	Diatomaceous earth	MTBE	48	Methanol	GC/MSD
2135	1 g - 0.2 g	yes	30 min	Chromabond XTR	MTBE	50	MTBE/Methanol	HPLC/DAD - GC/MSD
2137	0.5 g	no	30 min	--	--	50	MTBE/Methanol	HPLC/MSD - GC/MSD
2139	0.25 g	no	30 min	--	MTBE	60	Methanol	HPLC/DAD - GC/MSD
2146	0.7 - 1.0 g	no	30 min	Extrelut NT20	MTBE	35	Methanol	HPLC/DAD - GC/MSD
2165	1 g	yes	30 min	Diatomaceous earth	MTBE	50	MTBE	GC/MSD - HPLC/MS/MS
2166	0.5 g	no	30 min	Diatomaceous earth	MTBE	45	Methanol	HPLC/DAD
2170	1.0 g	no	30 min	Chromabond XTR	MTBE	40	--	--
2172	1 g	no	30 min	Kieselgur column	MTBE	40	Methanol	HPLC/DAD - GC/MSD
2184	1 g	yes	30 min	Diatomaceous earth	MTBE	50	MTBE	HPLC/DAD - GC/MSD
2190	1.0 g	no	30 min	Varien Chem elut	MTBE	50	Acetonitril	GC/MSD
2201	0.5 g	no	30 min	Kieselgur column	MTBE	40	MTBE	HPLC/DAD - GC/MSD
2213	1 g	yes	30 min	Diatomaceous earth	MTBE	40	Methanol	HPTLC - GC/MSD
2217	1 g	no	30 min	--	MTBE	40	MTBE	HPLC/MSD
2232	--	--	--	--	--	--	--	--
2236	1 g	no	30 min	Chromabond XTR	MTBE	70	MTBE	GC/MSD
2238	0.5 g	no	30 min	Diatomaceous earth	MTBE	40	MTBE	HPLC/DAD - GC/MSD
2245	0.75 - 1.0	yes	60 min	Diatomaceous earth	MTBE	45	Methanol	HPLC/DAD
2246	1.0 g	yes	30 min	Chromabond XTR	MTBE	45	Methanol	HPLC/DAD - GC/MSD
2247	0.2 - 0.5 g	no	30 min	Chromabond XTR	MTBE	40	Methanol	HPLC/DAD - GC/MSD
2254	0.75 - 1 g	no	30 min	Diatomaceous earth	MTBE	50	Acetonitril	GC/MSD
2255	0.3 g	no	30 min	Kieselgur column	MTBE	50	MTBE	GC/MSD
2256	1 g	no	30 min	Kieselgur column	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
2265	1 g	yes	30 min	Agilent ChemElt	MTBE	60	Methanol	HPLC/DAD
2284	0.5 g	--	30 min	Diatomaceous earth	MTBE	45	MTBE	HPLC/DAD - GC/MSD
2286	0.5 g	yes / no	30 min	Diatomaceous earth	MTBE	40	Methanol	HPLC/DAD - GC/MSD
2287	0.5 g	yes / no	30 min	Agilent ChemElt	MTBE	40	Methanol	HPLC/DAD - GC/MSD
2289	1 g	no	30 min	Diatomaceous earth	MTBE	40	MTBE	HPLC/DAD - GC/MSD
2290	0.5 g	no	30 min	Chromabond XTR	MTBE	45	MTB	HPLC/DAD - GC/MSD
2291	0.5 g	yes	30 min	Chromabond XTR	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
2293	1.0 g	no	30 min	--	MTBE	60	Methanol	GC/MSD
2295	1 g	no	30 min	Diatomaceous earth	MTBE	--	Nitrogen gas	HPLC/DAD - GC/MSD
2296	1 g	no	30 min	Agilent ChemElt	MTBE	46	Methanol	HPLC/DAD - GC/MSD
2297	1.0 g	no	30 min	Kieselgur column	MTBE	40	Methanol	GC/MSD
2301	0.5 g	no / yes	30 min	Kieselgur column	--	40	Acetonitril	HPLC/DAD - GC/MSD
2310	1 g	no	30 min	Chem Elut HDPE	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
2311	1g	no	30 min	Chem Elut HDPE	MTBE	30	Methanol	HPLC/DAD - GC/MSD
2313	1 g	no	30 min	Chem Elut HDPE	MTBE	40	Methanol	HPLC/DAD - GC/MSD
2359	0.5 g	no	30 min	Diatomaceous earth	MTBE	45	Methanol	GC/MSD
2364	1.0 g	no	30 min	--	--	50	MTBE	HPLC/DAD - GC/MSD
2366	0.5 - 1.0 g	yes	30 min	Diatomaceous earth	Methanol	57	MTBE	HPLC/MSD
2367	1.0 g	no	30 min	--	--	50	MTBE	HPLC/DAD - GC/MSD
2368	0.5 g	yes / no	30 min	--	--	50	MTBE	HPLC/DAD - GC/MSD
2370	0.5 g	no	30 min	Diatomaceous earth	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
2372	1.0 g	yes	30 min	--	MTBE	room	--	HPLC/DAD - GC/MSD

2373	1.0 g	no	30 min	--	--	50	Ethylacetate	HPLC/DAD - GC/MSD
2375	0.3 - 0.4 g	no	--	Chem Elut HDPE	MTBE	--	--	HPLC/DAD - GC/MSD
2379	1 g	yes	30 min	Extrelut NT20	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
2380	1.0 g	no	30 min	--	--	--	--	HPLC/DAD - GC/MSD
2381	1.0 g	no	30 min	--	--	--	--	HPLC/DAD - GC/MSD
2386	0.1 - 0.2 g	no	30 min	TOX - Elut	MTBE	30	Methanol	HPLC/DAD
2390	0.50 g	no	30 min	--	--	--	--	GC/MSD
2403	0.5 g	no	30 min	Kieselgur column	MTBE	35	MTBE	GC/MSD
2410	0.5 g	no	30 min	--	MTBE	50	MTBE	HPLC/DAD - GC/MSD
2413	1.0 g	no	30 min	Kieselgur column	MTBE	40	Methanol	GC/MSD
2415	0.5 g	no	30 min	--	MTBE	40	Methanol	HPTLC - GC/MSD
2425	0.5 g	no	30 min	Diatomaceous earth	MTBE	49	Methanol	HPLC/DAD - GC/MSD
2426	0.5 -1 g	no	30 min	Diatomaceous earth	MTBE	40	MTBE	GC/MSD
2428	1.0 g	no	30 min	Kieselgur column	Ether	35	Ethylacetate	GC/MSD
2429	1.0 g	no	30 min	Chromabond XTR	MTBE	40	MTBE	HPLC/DAD - GC/MSD
2432	0.5 g	no	30 min	--	MTBE	--	--	GC/MSD
2439	1 g	no	30 min	ProElut DIXMA	MTBE	50	Acetonitril	HPLC/DAD - GC/MSD
2441	0.5 g	no	30 min	--	MTBE	40	Methanol	GC/MSD
2442	0.5 g	no	30 min	--	--	--	--	HPLC/DAD - GC/MSD
2452	1 g	no	30 min	Agilent ChemElt	MTBE	40	MTBE	GC/MSD
2453	0.5 - 1 g	no	30 min	Chromabond XTR	MTBE	45	--	GC/MSD
2456	0.7 - 0.9 g	yes	30 min	Diatomaceous earth	MTBE	45	MTBE	GC/MSD
2459	1.0 g	no	30 min	Diatomaceous earth	MTBE	45	Acetonitril	HPLC/MSD - GC/MSD
2469	0.5 g	yes	30 min	Chem Elut HDPE	MTBE	--	--	GC/MSD
2472	1 g	no	30 min	Diatomaceous earth	MTBE	38	Methanol	HPLC/DAD - GC/MSD
2476	1.0 g	no	30 min	Diatomaceous earth	MTBE	50	MTBE	HPLC/DAD - GC/MSD
2481	0.5 - 1 g	heptane	30 min	Chromabond XTR	Ethylacetate	30	DCM	GC/MSD
2482	0.5 g	no	30 min	--	MTBE	50	Methanol	HPLC/MSD - GC/MSD
2483	0.5 g	no	30 min	Diatomaceous earth	MTBE	70	MTBE	GC/MSD
2486	0.3 - 0.5 g	no	30 min	Kieselgur column	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
2489	0.2 - 0.5 g	no	30 min	Chromabond XTR	MTBE	40	Methanol	HPLC/DAD - GC/MSD
2492	0.25 g	no	30 min	Diatomaceous earth	MTBE	35	Methanol	HPLC/MSD
2493	--	--	--	--	--	--	--	--
2495	1 g	no	30 min	Varien Chem elut	MTBE	40	MTBE	HPLC/DAD - GC/MSD
2497	0.5 g	no	30 min	Extrelut NT20	MTBE	50	MTBE	HPLC/MSD
2508	0.5 g	--	30 min	Diatomaceous earth	MTBE	50	--	GC/MSD
2511	1 g	no	30 min	Agilent ChemElt	MTBE	45	Methanol	GC/MSD - LC/MSD
2514	0.2 - 0.3 g	no	30 min	Kieselgur column	MTBE	50	MTBE	HPLC/DAD - GC/MSD
2516	1.0 g	no	30 min	Diatomaceous earth	MTBE	35	Methanol	HPLC/DAD - GC/MSD
2522	1 g	no	30 min	--	MTBE	--	--	GC/MSD
2523	0.7 - 0.9 g	no	30 min	Extrelut NT20	MTBE	35	Methanol	HPLC/DAD - GC/MSD
2528	0.5 g	no	30 min	Diatomaceous earth	MTBE	40	--	HPLC/DAD - GC/MSD
2532	0.4 g	no	30 min	Chromabond XTR	MTBE	40	Methanol	HPLC/DAD - GC/MSD
2534	1 g	no	30 min	Hypersep SLE	MTBE	45	--	GC/MSD
2540	1 g	no	30 min	Diatomaceous earth	MTBE	35	MTBE	GC/MSD
2546	1 g	no	30 min	Diatomaceous earth	MTBE	45	Acetonitril	HPLC/DAD - GC/MSD
2549	0.5 g	no	30 min	Extrelut NT20	MTBE	50	Methanol	UPLC-PDA
2553	1 g	no	30 min	--	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
2559	1.0 g	no	30 min	Agilent ChemElt	MTBE	50	Methanol	GC/MSD
2560	0.3 - 0.4 g	no	30 min	Agilent ChemElt	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
2562	--	--	--	--	--	--	--	--
2565	1.0 g	no	30 min	Diatomaceous earth	MTBE	35	Methanol	HPLC/DAD - GC/MSD
2566	1.0 g	no	30 min	Kieselgur column	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
2570	1.0 g	no	30 min	Diatomaceous earth	MTBE	45	Methanol	HPLC/DAD - GC/MSD
2581	0.5 g	yes	30 min	--	MTBE	50	MTBE	GC/MSD
2582	0.25 g	no	30 min	ChemElut	MTBE	52	MTBE	HPLC/DAD - GC/MSD
2583	1.3 g	no	30 min	Chromabond XTR	MTBE	49	Methanol	HPLC/DAD
2590	1 g	yes	--	ChemElut	MTBE	30	Methanol	HPLC/DAD - GC/MSD
2596	0.7 - 1.0 g	no	30 min	Chromabond XTR	Ethylether	35	Methanol	GC/MSD
2601	0.3 g	no	30 min	Chromabond XTR	MTBE	45	Methanol	HPLC/DAD
2605	0.5 g	yes	30 min	Chromabond XTR	MTBE	35	MTBE	HPLC/DAD - GC/MSD
2613	0.43 g	no	30 min	--	MTBE	50	MTBE	GC/MSD - HPLC/MS/MS

2614	1.0 g	no	30 min	--	MTBE	40	Methanol	GC/MSD
2617	0.5 -1.0 g	no	30 min	Diatomaceous earth	MTBE	40	Methanol	GC/MSD
2618	1.0 g	no	30 min	--	--	--	--	HPLC/DAD - GC/MSD
3100	0.5 g	yes	30 min	Diatomaceous earth	MTBE	35	MTBE	HPLC/DAD - GC/MSD
3104	0.2 - 0.5 g	no	30 min	--	MTBE	--	--	HPTLC - GC/MSD
3107	0.75 - 0.80 g	no	30 min	Agilent ChemElt	MTBE	50	Methanol	HPLC/DAD - GC/MSD
3109	0.6 - 0.8 g	no	30 min	Extrelut NT20	MTBE	40	Acetonitril	HPLC/MSD
3116	1 g	no	30 min	Kieselgur column	MTBE	40	Acetonitril	GC/MSD
3117	1.0 g	no	30 min	Diatomaceous earth	MTBE	34	Ethylacetate	HPLC/DAD - GC/MSD
3118	1	no	30 min	Kieselgur column	MTBE	35	MTBE	GC/MSD
3146	0.25 - 0.5 g	no	30 min	--	MTBE	45	Methanol	HPLC/DAD - GC/MSD
3150	0.5 g	no	30 min	Kieselgur column	MTBE	40	MTBE	GC/MSD
3151	0.1 g	no	30 min	Kieselgur column	MTBE	40	MTBE	GC/MSD
3153	0.5 g	no	30 min	Chromabond XTR	MTBE	35	MTBE	HPLC/DAD - GC/MSD
3154	0.3 g	no	30 min	alkline silica	MTBE	60	MTBE	HPLC/DAD
3167	1 g	no	30 min	Diatomaceous earth	MTBE	40	Methanol	HPLC/DAD - GC/MSD
3172	1.0 g	yes	30 min	--	MTBE	35	Ethylacetate	HPLC/MSD - GC/MSD
3176	0.5 g	yes / no	30 min	--	MTBE	50	Methanol	--
3180	0.35 g	yes	30 min	--	--	50	Methanol	HPLC/DAD - GC/MSD
3182	0.5 g	no	30 min	Agilent ChemElt	MTBE	50	MTBE	GC/MSD
3185	0.5 g	no	30 min	Chromabond XTR	MTBE	35	MTBE	HPLC/DAD - GC/MSD
3190	0.50 g	no	30 min	Diatomaceous earth	MTBE	40	MTBE	HPLC/DAD - GC/MSD
3191	1	yes / no	30 min	Diatomaceous earth	MTBE	50	Methanol	HPLC/DAD - GC/MSD
3195	0.5 g	no	30 min	Extrelut NT20	MTBE	30	Methanol	HPLC/DAD
3197	1 g	no	30 min	ChemElut	MTBE	40	Methanol	GC/MSD
3199	0.5 g	no	30 min	Agilent ChemElt	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
3200	0.5 g	no	30 min	Diatomaceous earth	MTBE	50	Methanol	HPLC/DAD - GC/MSD
3204	0.5 g	no	30 min	--	Methanol	--	--	GC/MSD
3209	0.1 - 0.2 g	no	30 min	Diatomaceous earth	MTBE	42	Methanol	HPLC/DAD - GC/MSD
3210	1 g	no	30 min	--	MTBE	room	Methanol	HPLC/DAD - GC/MS/MS
3214	0.5 g	no	30 min	Chromabond XTR	MTBE	35	MTBE	HPLC/DAD - GC/MSD
3216	0.7 - 1.0 g	no	30 min	Siliceous earth	MTBE	40	Methanol	HPLC/DAD - GC/MSD
3218	1.00 g	no	30 min	Kieselgur column	MTBE	40	MTBE	HPLC/DAD - GC/MSD
3220	0.5 g	yes	30 min	ChemElut	MTBE	35	Methanol	HPLC/DAD - GC/MSD
3222	0.7 - 1.0 g	no	30 min	Diatomaceous earth	MTBE	46	Acetonitril	GC/MSD
3228	1 g	yes	30 min	Diatomaceous earth	MTBE	<50	MTBE	HPLC/DAD - GC/MSD
3237	0.35 g	no	30 min	--	MTBE	--	--	HPLC/DAD - GC/MSD
3242	0.5 g	no	30 min	Extrelut NT20	MTBE	45	Methanol	HPLC/DAD
3243	0.75 - 1.0 g	no	30 min	Nucleodur C18	MTBE	50	MTBE	HPLC/DAD - GC/MSD
3248	1.0 g	n-Hexane	25 min	Kieselgur column	MTBE	40	Acetonitril	HPLC/DAD - GC/MSD
8005	1 g	no	30 min	Kieselgur column	MTBE	40	Acetonitril	HPLC/DAD

APPENDIX 4

Number of participants per country

10 labs in BANGLADESH

1 lab in BRAZIL

1 lab in BULGARIA

1 lab in EGYPT

1 lab in FINLAND

4 labs in FRANCE

16 labs in GERMANY

1 lab in GREECE

1 lab in GUATAMALA

11 labs in HONG KONG

2 labs in HUNGARY

13 labs in INDIA

2 labs in INDONESIA

10 labs in ITALY

5 labs in JAPAN

4 labs in KOREA

1 lab in MOROCCO

36 labs in P.R. of CHINA

3 labs in PAKISTAN

1 lab in PORTUGAL

2 labs in SINGAPORE

1 lab in SLOVENIA

2 labs in SPAIN

2 labs in SRI LANKA

2 labs in SWITZERLAND

5 labs in TAIWAN

2 labs in THAILAND

1 lab in THE NETHERLANDS

2 labs in TUNISIA

5 labs in TURKEY

5 labs in U.S.A.

2 labs in UNITED KINGDOM

4 labs in VIETNAM

APPENDIX 5

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
R(0.01)   = outlier in Rosner's outlier test
R(0.05)   = straggler in Rosner's outlier test
n.e.   = not evaluated
n.d.   = not detected

Literature:

1   DIN 53316
2   LMBG 82.02-2:98
3   LMBG 82.02-3:97
4   LMBG 82.04-2:98
5   EN14362-1, March 2012
6   Staatsblad van het Koninkrijk der Nederlanden 339, bijlage II, 23 april 1998
7   iis-Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation, April 2014
8   XP G 08-014:97
9   P.L. Davies, Fr Z. Anal. Chem, <u>351</u>, 513, (1988)
10   W.J. Conover, Practical; Nonparametric Statistics, J. Wiley&Sons, NY, p.302, (1971)
11   ISO 5725, (1986)
12   ISO 5725, parts 1-6, (1994)
13   M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
14   G. Rohm, J. Bohnen & H. Kruessmann, GIT Labor-Fachzeitschrift, p 1080, <u>11</u>, (1997)
15   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, T*echnometrics*, 25(2), pp. 165-172, (1983)