

Results of Proficiency Test
AZO dyes in textile
March 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

The Institute for Interlaboratory Studies (iis) organizes a proficiency test (PT) for banned aromatic amines from AZO dyes in textile every year since 1997. During the annual proficiency testing program 2014/2015, it was decided to continue the PT for the analysis of banned aromatic amine dyes in textile. In this interlaboratory study, 184 laboratories in 37 different countries have participated (see appendix 4).

In this report, the results of the 2015 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 Spijkensisse was the organizer of this proficiency test. It was decided to use in this proficiency test 2 different textile samples (labelled #15020 and #15021), 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 Spijkensisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the 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). The protocol can be downloaded from iis website <http://www.iisnl.com>.

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 red polyester based textile (sample #15020) and a pink cotton (sample #15021) were dyed with different AZO-dyes. From the polyester batch, 204 samples with small pieces of fabric of approximately 3 gram were prepared and labelled #15020. From the second batch, also 204 samples with small pieces of fabric of approximately 3 gram were prepared and labelled #15021. The homogeneity of the subsamples #15020 and #15021 was checked by determination of aromatic amines on 8 stratified randomly selected samples. See the following tables for the test results.

	<i>o</i> -toluidine in mg/kg
sample #15020-1	491
sample #15020-2	517
sample #15020-3	530
sample #15020-4	526
sample #15020-5	478
sample #15020-6	510
sample #15020-7	505
sample #15020-8	528

Table 1: homogeneity test results of subsamples #15020

	<i>benzidine</i> in mg/kg
sample #15021-1	48.8
sample #15021-2	55.2
sample #15021-3	47.8
sample #15021-4	50.0
sample #15021-5	52.4
sample #15021-6	53.6
sample #15021-7	51.9
sample #15021-8	53.1

Table 2: homogeneity test results of subsamples #15021

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

	<i>o</i> -toluidine in mg/kg	<i>benzidine</i> in mg/kg
r (observed) #15020	52.23	--
r (observed) #15021	--	7.08
reference method	EN14362-1:2012	EN14362-1:2012
r (reference method)	99.57	9.18

Table 3: repeatabilities of subsamples #15020 and #15021

The calculated repeatabilities of the test results were in agreement with the repeatabilities 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 #15020 and 1 sample labelled #15021 on March 4, 2015.

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 5.

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 5, 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 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. 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 $z_{(\text{target})}$ -scores were calculated according to:

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

The $z_{(\text{target})}$ -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, 180 participants did report 619 numerical results. Observed were 18 outlying results, which is 2.9% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

There are no reproducibility requirements for o-aminoazotoluene stated in EN14362-1:2012. Because of the instability of o-aminoazotoluene, the reproducibility is expected to be higher than the reproducibility of o-toluidine. Therefore the reproducibility of another ‘difficult’ to determine substance was used: 4-Aminodiphenyl (see ref. 6).

Since EN14362-1 states that o-aminoazotoluene (CASno. 97-56-3) is further reduced to o-toluidine (CAS-number 95-53-4), also a summation of test results of both substances found in #15020 was made.

The target reproducibility of the summation of the test results for o-aminoazotoluene and o-toluidine was estimated from the reproducibilities of o-toluidine mentioned in EN14362-1:2012 and the reproducibility of o-aminoazotoluene based on the reproducibility of 4-Aminodiphenyl.

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 #15020:

o-Aminoazotoluene (CASno.97-56-3):

The determination of this aromatic amine was problematic. Thirty-four laboratories reported the absence of this amine via a 'less than' test result, 'not detected' or '0' (zero). One laboratory reported 'positive' without reporting a value. However, one-hundred-six laboratories did report a numerical test result, varying from 'not detected' or '0' (zero) to 400.2 mg/kg. These results were statistically evaluated. Eleven statistical outliers were observed. Since this substance may reduce to o-toluidine during analysis, it was decided not to calculate z-scores for o-aminoazotoluene, but only for the values of the summation of o-aminoazotoluene and o-toluidine. See also the discussion in chapter 6.

o-Toluidine (CASno.95-53-4)

The determination of this aromatic amine was very problematic. No statistical outliers were observed. However, the test results reported by the participants vary from 'nd'-565 mg/kg. Furthermore the Kernel Density graph clearly shows the presence of two groups of test results. Since o-aminoazotoluene may reduce to o-toluidine, it was decided not to calculate z-scores for o-toluidine, but only for the values of the summation of o-aminoazotoluene and o-toluidine. See also the discussion in chapter 6.

Summation of o-aminoazotoluene and o-Toluidine (CASno.95-53-4)

The determination of the sum of these aromatic amines may not be problematic. Three statistical outliers were observed and forty-four test results were excluded for reasons explained in chapter 6. However, the observed reproducibility after rejection of the suspect data is in full agreement with the reproducibility requirement estimated from the test method EN14362-1:2012 and from the reproducibility of o-aminoazotoluene based on the reproducibility of 4-Aminodiphenyl (ref. 6). See also the discussion in chapter 6.

Textile sample #15021:

Benzidine (CASno.92-87-5):

The determination of this aromatic amine at a concentration level of 48 mg/kg was problematic. Two statistical outliers and three false negative test results were detected. The observed reproducibility after rejection of the statistical outliers is in not agreement with the reproducibility requirement estimated from the test method EN14362-1:2012.

3,3'-Dimethylbenzidine (CASno.119-93-7):

The determination of this aromatic amine at a concentration level of 62 mg/kg was not problematic. Five statistical outliers and three false negative test results were detected. 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.

General:

Eight participants reported also the presence of other aromatic amines at different concentration levels in sample #15020 (see Appendix 2).

Eight participants reported also the presence of other aromatic amines at different concentration levels in sample #15021 (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.

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R(target)</i>
<i>o</i> -aminoazotoluene	mg/kg	95	(48)	(64)	(38)
<i>o</i> -Toluidine	mg/kg	162	(186)	(364)	(114)
Sum. of <i>o</i> -aminoazotoluene and <i>o</i> -Toluidine	mg/kg	128	258	247	258

Table 4: reproducibilities of the aromatic amines in textile sample #15020

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R(target)</i>
Benzidine	mg/kg	174	48	27	18
3,3'-Dimethylbenzidine	mg/kg	170	62	27	30

Table 5: reproducibilities of the aromatic amines in textile sample #15021

* value between brackets means that the z-score was not calculated

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 except 3,3'-Dimethylbenzidine. 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 and from ref. 6 for 4-Aminodiphenyl, see below table.

<i>Parameter</i>	<i>March 2015</i>	<i>April 2014</i>	<i>March 2013</i>	<i>March 2012</i>	<i>March 2011</i>	<i>March 2010</i>	<i>March 2009</i>	<i>target</i>
4-Aminodiphenyl	n.e.	(21%)*	n.e.	18%	(31%)*	18%	n.e.	28%
Benzidine	20%	15%	n.e.	21%	18%	19%	n.e.	14%
4-Chloro- <i>o</i> -toluidine	n.e.	24%	n.e.	n.e.	n.e.	n.e.	n.e.	16%
<i>o</i> -Aminoazotoluene	(48%)**	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	28%
<i>p</i> -Chloroaniline	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	27%	16%
2,4-Diaminoanisol	n.e.	n.e.	52%	n.e.	n.e.	n.e.	n.e.	16%
4,4'-Diaminodiphenylmethane	n.e.	21%	n.e.	n.e.	n.e.	n.e.	21%	15%
3,3'-Dimethoxybenzidine	n.e.	21%	16%	17%	17%	n.e.	n.e.	13%
3,3'-Dimethylbenzidine	15%	n.e.	n.e.	n.e.	n.e.	17%	n.e.	18%
4,4'-Diamino-3,3'-dichlorodiphenylmethane	n.e.	n.e.	n.e.	n.e.	20%	n.e.	23%	16%
4,4'-Diaminodiphenylether	n.e.	n.e.	n.e.	n.e.	15%	n.e.	n.e.	16%
4,4'-Diaminodiphenylsulfide	n.e.	n.e.	n.e.	n.e.	n.e.	18%	n.e.	16%
<i>o</i> -Toluidine	(70%)**	31%	27%	n.e.	n.e.	19%	n.e.	22%
Sum of <i>o</i> -aminoazotoluene and <i>o</i> -Toluidine	34%	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	36%

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

Sample #15020 is a polyester fabric that was dyed with an AZO dye that may release o-Aminoazotoluene that may further release o-Toluidine.

Sample #15021 is a cotton fabric that was dyed with AZO dyes that are known to release Benzidine and 3,3'-Dimethylbenzidine.

From the reported details of analyses, it is clear that most participants treated the fabric samples as the test method described. According to the method, the polyester fabric had to be extracted with chlorobenzene, while that was not required for the cotton fabric.

The following should be noted. During the preparation of this PT it was found that the chlorobenzene extraction was a critical step. The method states in 9.1.1: 'Concentrate the chlorobenzene extract in the evaporation apparatus at a temperature of 45 °C to 60 °C to a small residual quantity'. But what is considered a small quantity? Would it be OK to leave just a drop of chlorobenzene or should all chlorobenzene be evaporated? This last step takes a considerable amount of extra time. Tests were done on these samples with a drop of chlorobenzene left after the extraction and on samples in which the chlorobenzene was completely evaporated. The results were that the samples with a drop of chlorobenzene showed significant lower test results on o-toluidine than the samples where the chlorobenzene was completely evaporated. Since the method is not clear on how much chlorobenzene should or should not be left after the extraction, this may have an influence on the cleavage step. This may also (partly) explain why such a large spread is found for o-aminoazotoluene and/or o-toluidine.

The second part of the analytical details was about the test conditions after cleavage at 70°C and before the analytical measurements. For example, the temperature during the evaporation of MTBE should not exceed 50°C, otherwise the aromatic amines may deteriorate and may not be detected anymore. Regretfully the phrasing of the questions left room for different interpretations and the laboratories reported a mix of evaporation temperatures for the different steps during the analysis. Therefore no conclusions could be drawn from this information.

In sample #15020, the majority of the laboratories (106) reported the presence of o-Aminoazotoluene. And perhaps even more laboratories did detect this amine, but they may have not realized that this would be possible because Method EN14362-1:2012 remarks that this amine is further reduced to o-toluidine. However in the HPLC/DAD-chromatogram in figure A.1 of EN14362-1 the peak of o-Aminoazotoluene (nr. 5) is clearly visible.

Similar was the case in last year's PT iis14A01T, in which a number of laboratories also reported the presence of o-aminoazotoluene, but in 2014 a different AZO dye was applied to the textile. In this 2015 PT an artificial dye known to release o-aminoazotoluene and/or o-toluidine was made and used to colour the polyester fabric.

For better understanding one has to realize that *o*-Aminoazotoluene is formed by reductive cleavage of the original AZO dye, but not all *o*-Aminoazotoluene will be reduced to *o*-toluidine directly. In this case the reduction of *o*-Aminoazotoluene obviously is slower than the formation of *o*-Aminoazotoluene from the original dye which leads to a significant concentration of *o*-Aminoazotoluene and a small concentration of *o*-toluidine in the test solution. However, during subsequent analysis steps (extraction, evaporation, etc.) the amount of *o*-Aminoazotoluene will decrease and the amount of *o*-toluidine will increase. Therefore conditions like (small) residue of chlorobenzene, the temperature during evaporation of MTBE and the length of time taken for completing the analysis will have a significant influence on the concentrations *o*-Aminoazotoluene and *o*-toluidine that will be quantified.

Therefore it is not strange that some of the laboratories reported only *o*-aminoazotoluene, while others reported only *o*-toluidine, and again other laboratories reported both. The above is the reason that it was decided to look at the performance of the participating laboratories on the summation of *o*-aminoazotoluene and *o*-toluidine and not on the results of the separate amines.

The Kernel Density graph of this summation shows two groups of results: one group (25%) around 60 mg/kg (with a standard deviation of 30 mg/kg) and one group (75%) around 260 mg/kg (with a standard deviation of around 100 mg/kg). The bimodality cannot be explained from the reported analytical details or the fact that some laboratories only reported *o*-aminoazotoluene or only *o*-toluidine.

The 25% lowest values were excluded from the statistical evaluation for being suspect because:

- laboratories may not have seen *o*-aminoazotoluene assuming that it cannot be present
- laboratories may not have quantified *o*-aminoazotoluene correctly due to a lack of calibrant and/or due to unknown purity of the (unstable) calibrant
- laboratories may have had a very low recovery for the unstable *o*-aminoazotoluene

All of the above conditions will lead to a decrease of *o*-aminoazotoluene and/or *o*-toluidine in the test result.

APPENDIX 1

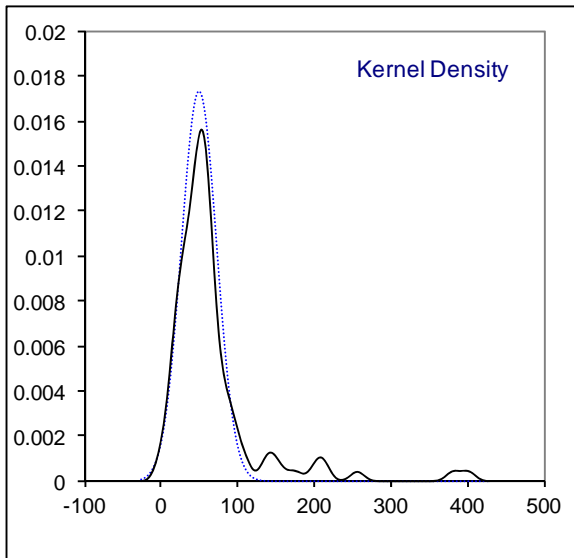
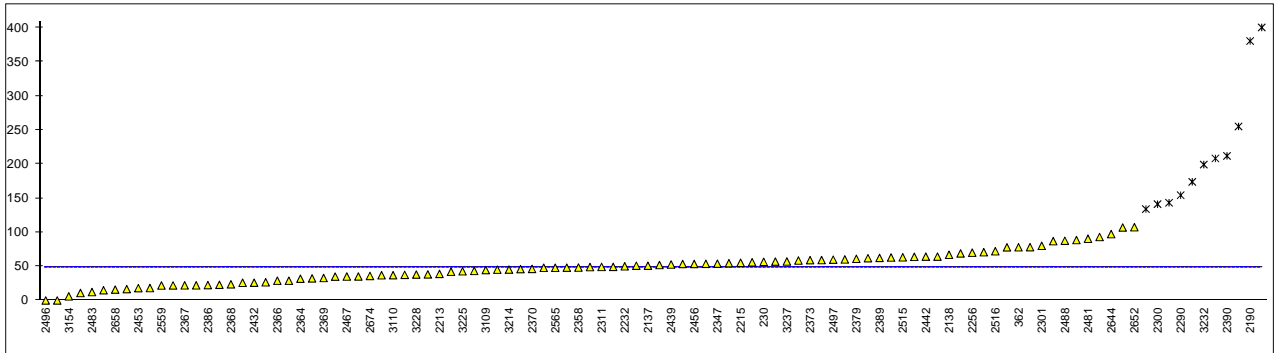
Determination of *o*-Aminoazotoluene (CASno.97-56-3) in sample #15020; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	EN14362-1	n.d.		----	2386	EN14362-1	22.6		----
213		----		----	2389	EN14362-1	62.41		----
230	EN14362-1	56.7		----	2390	EN14362-1	211.913	R(0.01)	----
348	in house	53.83		----	2403	EN14362-1	n.d.		----
362	in house	78		----	2410	EN14362-1	56		----
551	EN14362-1	143.24	R(0.05)	----	2413		----		----
622	EN14362-1	107		----	2415		----		----
2115		----		----	2425		----		----
2120		----		----	2426	EN14362-1	n.d.		----
2121	EN14362-1	22.11		----	2429	EN14362-1	n.d.		----
2129		----		----	2432	EN14362-1	26.15		----
2132	EN14362-1	<5		----	2439	EN14362-1	52.7		----
2137	EN14362-1	51		----	2442	in house	64.26		----
2138	EN14362-1	67.0		----	2449		----		----
2139	EN14362-1	69		----	2452	EN14362-1	n.d.		----
2146		----		----	2453	EN14362-1	18.05		----
2165	EN14362-1	37		----	2456	EN14362-1	53.5		----
2166		----		----	2459		----		----
2169		----		----	2467	in house	35.16		----
2170		----		----	2472	EN14362-1	49		----
2172	EN14362-1	70.8		----	2475		----		----
2173	EN14362-1	22.2		----	2476	EN14362-1	n.d.		----
2184	EN14362-1	35		----	2479	EN14362-1	37.6		----
2190	EN14362-1	380.3	R(0.01)	----	2481	EN14362-1	90.5		----
2201	EN14362-1	<5		----	2482		----		----
2213	EN14362-1	39		----	2483	EN14362-1	12.38	C	----
2215	EN14362-1	55.3		----	2488	EN14362-1	87.53		----
2217	EN14362-1	29.01		----	2489	EN14362-1	n.d.		----
2228	CPSD-AN-00607/00107	77.89	C	----	2492		----		----
2229	EN14362-1	<10.0		----	2493	in house	<3		----
2230	EN14362-1	78.2		----	2495		----		----
2232	EN14362-1	50.11		----	2496	EN14362-1	0.0		----
2238	EN14362-1	<5		----	2497	EN14362-1	59.84		----
2244		----		----	2504	EN14362-1	0		----
2246	EN14362-1	n.d.		----	2508	EN14362-1	26.70		----
2247	EN14362-1	n.d.		----	2511		----		----
2255		----		----	2514		----		----
2256	EN14362-1	70.1		----	2515	EN14362-1	63.3		----
2264		----		----	2516	EN14362-1	72.2		----
2271	EN14362-1	26		----	2523	EN14362-1	42.176		----
2284	EN14362-1	63		----	2524		----		----
2286	EN14362-1	62		----	2528	EN14362-1	11		----
2287	EN14362-1	134	R(0.05)	----	2532	EN14362-1	positive		----
2289	EN14362-1	n.d.		----	2534	EN14362-1	48		----
2290	EN14362-1	154.3	R(0.05)	----	2538	64LFGB82.02-4	<15		----
2291	EN14362-1	n.d.		----	2546	EN14362-1	<20		----
2293		----		----	2549	EN14362-1	n.d.		----
2295		----		----	2553	EN14362-1	n.d.		----
2296		----		----	2559	EN14362-1	22		----
2297	EN14362-1	58.6		----	2562		----		----
2300	EN14362-1	141.2	R(0.05)	----	2563	EN14362-2	16.7		----
2301	EN14362-1	80.14		----	2565	EN14362-1	48.0		----
2310	EN14362-1	51.0		----	2566	EN14362-1	n.d.		----
2311	EN14362-1	49.2		----	2567	EN14362-1	n.d.		----
2313	EN14362-1	43.3		----	2572	EN14362-1	n.d.		----
2314	EN14362-1	53.5		----	2590	EN14362-1	93.04	C	----
2320	EN14362-1	59.2684		----	2604	EN14362-1	n.d.		----
2330	EN14362-1	48.10		----	2605		----		----
2347	EN14362-1	54		----	2613		----		----
2352	EN14362-1	23		----	2629	EN14362-1	400.2	R(0.01)	----
2357	EN14362-1	32.4		----	2638	EN14362-1	49.36		----
2358	EN14362-1	48.3		----	2643	EN14362-1	64		----
2364	EN14362-1	32		----	2644	EN14362-1	97.3		----
2365	EN14362-1	46		----	2649		----		----
2366	EN14362-1	29		----	2650	EN14362-1	87		----
2367	EN14362-1	22.2		----	2652	EN14362-1	107.4		----
2368	EN14362-1	23.7		----	2654		----		----
2369	EN14362-1	33		----	2658	EN14362-1	15.97		----
2370	EN14362-1	46.2		----	2662	EN14362-1	57	C	----
2373	EN14362-1	59.2		----	2663	EN14362-1	64.3		----
2375	EN14362-1	60.2		----	2668		----		----
2379	EN14362-1	61.112		----	2671		----		----

2674	EN14362-1	36		----	3190	EN14362-1	n.d.	----
2677	EN14362-1	18.27	C	----	3191	EN14362-1	<10	----
2678		----		----	3192		----	----
3100	EN14362-1	n.d.		----	3197		----	----
3109	EN14362-1	44.6		----	3199	CPSD-AN-00607	208.2	R(0.01)
3110	EN14362-1	37.1		----	3200	EN14362-1	15	
3116		----		----	3204	EN14362-1	255.1	R(0.01)
3117	EN14362-1	52		----	3210	EN14362-1	45.2	
3118	EN14362-1	n.d.		----	3214	EN14362-1	45.31	
3122	EN14362-1	38.24		----	3216	EN14362-1	n.d.	
3146	EN14362-1	n.d.		----	3218		----	----
3149		----		----	3220	EN14362-1	35.2	
3150		----		----	3222	EN14362-1	88.57	
3153	EN14362-1	n.d.		----	3225	EN14362-1	42.82	
3154	BVLB82.02-2	6.05	C	----	3228	EN14362-1	38	
3167		----		----	3232	EN14362-1	199.17	R(0.01)
3172		----		----	3237	in house	57.1889	
3176	EN14362-1	173.87	R(0.01)	----	3242		----	----
3182	EN14362-1	n.d.		----	3246	EN14362-1	54.7	
3185	EN14362-1	<5		----	3248		----	----

normality OK
n 95
outliers 11
mean (n) (48.432)
st.dev. (n) (23.0303)
R(calc.) (64.485)
R(Stb. 399:2012) (38.242)

Lab 2228 first reported: 0
Lab 2483 first reported: n.d.
Lab 2590 first reported result as 4,4'-diaminodiphenylmethane
Lab 2662 first reported: n.d.
Lab 2677 first reported: 11.94
Lab 3154 first reported: 6.05



Determination of o-Toluidine (CASno.95-53-4) in sample #15020; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	EN14362-1	80.7		----	2386	EN14362-1	50.6		----
213		----		----	2389	EN14362-1	45.49		----
230	EN14362-1	54.5		----	2390	EN14362-1	42.763		----
348	in house	131.51		----	2403	EN14362-1	334.0		----
362	in house	14		----	2410	EN14362-1	280		----
551	EN14362-1	144.68		----	2413	EN14362-1	9.57		----
622	EN14362-1	<5		----	2415	EN14362-1	248.1		----
2115		----		----	2425	EN14362-1	245.1		----
2120	EN14362-1	33		----	2426	EN14362-1	240.6127		----
2121	EN14362-1	57.71		----	2429	EN14362-1	421.76		----
2129	EN14362-1	565		----	2432		----		----
2132	EN14362-1	288.6		----	2439	EN14362-1	204.1		----
2137	EN14362-1	274		----	2442		----		----
2138	EN14362-1	231.0		----	2449		----		----
2139	EN14362-1	240		----	2452	EN14362-1	n.d.		----
2146	EN14362-1	313		----	2453	EN14362-1	164.5		----
2165	EN14362-1	220		----	2456	EN14362-1	145.5		----
2166	EN14362-1	230		----	2459	EN14362-1	213.235		----
2169		----		----	2467	in house	269.84		----
2170	EN14362-1	68.9		----	2472	EN14362-1	6	C	----
2172	EN14362-1	19.8		----	2475		----		----
2173	EN14362-1	425.1		----	2476	EN14362-1	205.2		----
2184	EN14362-1	210		----	2479	EN14362-1	412.8		----
2190	EN14362-1	<5		----	2481	EN14362-1	72.6		----
2201	EN14362-1	343.2		----	2482	EN14362-1	147.671		----
2213	EN14362-1	369.2		----	2483	EN14362-1	n.d.		----
2215	EN14362-1	220.5		----	2488	EN14362-1	90.44		----
2217	EN14362-1	122.33		----	2489	EN14362-1	390.10		----
2228	CPSD-AN-00607/00107	47.50	C	----	2492	EN14362-1	317.0		----
2229	EN14362-1	353.1		----	2493	in house	26.92		----
2230	EN14362-1	13.1		----	2495	EN14362-1	108.0		----
2232		----		----	2496	EN14362-1	348.0		----
2238	EN14362-1	310.6		----	2497	EN14362-1	3.97		----
2244	EN14362-1	320.0		----	2504	EN14362-1	376.75		----
2246	EN14362-1	88.44		----	2508	EN14362-1	20.40		----
2247	EN14362-1	409.35		----	2511	EN14632-1	41.21		----
2255	EN14362-1	209.3		----	2514	EN14362-1	215.5		----
2256	EN14362-1	114		----	2515	EN14362-1	183		----
2264	EN14362-1	209.34		----	2516	EN14362-1	<5		----
2271	EN14362-1	288		----	2523	EN14362-1	24.766		----
2284	EN14362-1	182		----	2524	EN14362-1	3.14		----
2286	EN14362-1	36		----	2528	EN14362-1	147		----
2287	EN14362-1	15		----	2532	EN14362-1	322		----
2289	EN14362-1	422		----	2534	EN14362-1	146		----
2290	EN14362-1	66.8		----	2538	64LFGB82.02-4	281.9		----
2291	EN14362-1	317.0		----	2546	EN14362-1	<20		----
2293	EN14362-1	548.539		----	2549	EN14362-1	244.6		----
2295	EN14362-1	34	C	----	2553	EN14362-1	176.36		----
2296	EN14362-1	224.19		----	2559		----		----
2297	EN14362-1	233.6		----	2562	GB/T17592	310.6		----
2300	EN14362-1	347.5		----	2563	EN14362-2	1.31		----
2301		----		----	2565	EN14362-1	<5		----
2310	EN14362-1	228		----	2566	EN14362-1	176		----
2311	EN14362-1	238		----	2567	EN14362-1	253.76		----
2313	EN14362-1	255		----	2572	EN14362-1	61		----
2314	EN14362-1	245		----	2590		----		----
2320	EN14362-1	9.5403		----	2604	EN14362-1	124		----
2330	EN14362-1	49.36	C	----	2605	EN14362-1	323.3		----
2347	EN14362-1	151		----	2613	EN14362-1	204.0		----
2352	EN14362-1	304		----	2629	EN14362-1	n.d.		----
2357	EN14362-1	171.3		----	2638	EN14362-1	126.5		----
2358	EN14362-1	21.1		----	2643	EN14362-1	287		----
2364	EN14362-1	217		----	2644	EN14362-1	30.7		----
2365	EN14362-1	176		----	2649	EN14362-1	293.06		----
2366	EN14362-1	409		----	2650	EN14362-1	16		----
2367	EN14362-1	204.0		----	2652	EN14362-1	17.7		----
2368	EN14362-1	205.4		----	2654	EN14362-1	2.56		----
2369	EN14362-1	211		----	2658	EN14362-1	<5		----
2370	EN14362-1	184		----	2662	EN14362-1	20	C	----
2373	EN14362-1	217.0		----	2663	EN14362-1	235.6		----
2375	EN14362-1	40.2		----	2668	EN14362-1	242.4		----
2379	EN14362-1	150.529		----	2671	EN14362-1	173.34		----
2674	EN14362-1	225		----	3190	EN14362-1	347.11		----

2677	EN14362-1	13.63	C	----	3191	EN14362-1	183	----
2678	EN14362-1	42.5		----	3192	EN14362-1	189.7	----
3100	EN14362-1	314		----	3197	EN14362-1	198.1	----
3109	EN14362-1	186.4		----	3199	CPSD-AN-00607	42.8	----
3110	EN14362-1	273.8		----	3200	EN14362-1	557	----
3116	EN14362-1	56.15		----	3204	EN14362-1	32.4	C
3117	EN14362-1	16		----	3210	EN14362-1	29.3	----
3118	EN14362-1	190.42		----	3214	EN14362-1	375.74	----
3122	EN14362-1	8.45		----	3216	EN14362-1	97.71	----
3146	EN14362-1	290		----	3218	EN14362-1	235	----
3149		----		----	3220	EN14362-1	214.9	----
3150	EN14362-1	86.0		----	3222	EN14362-1	143.83	----
3153	EN14362-1	183		----	3225	EN14362-1	8.80	----
3154	BVLB82.02-2	2.36	C	----	3228	EN14362-1	230	----
3167	EN14362-1	345.5		----	3232	EN14362-1	131.63	----
3172		----		----	3237	in house	381.1921	----
3176	EN14362-1	27.43		----	3242	EN14362-1	280	----
3182	EN14362-1	57.39		----	3246	EN14362-1	206	----
3185	EN14362-1	327		----	3248	EN14362-1	350	----
normality		OK						
n		162						
outliers		0						
mean (n)		(186.031)						
st.dev. (n)		(130.1670)						
R(calc.)		(364.467)						
R(EN14362-1:12)		(113.665)						

Lab 2120 performed 4 extractions: >100, 39, 8 and 27. Result was calculated as (39+27)/2=33

Lab 2228 first reported: 14.78

Lab 2295 first reported: 4

Lab 2230 first reported: n.d.

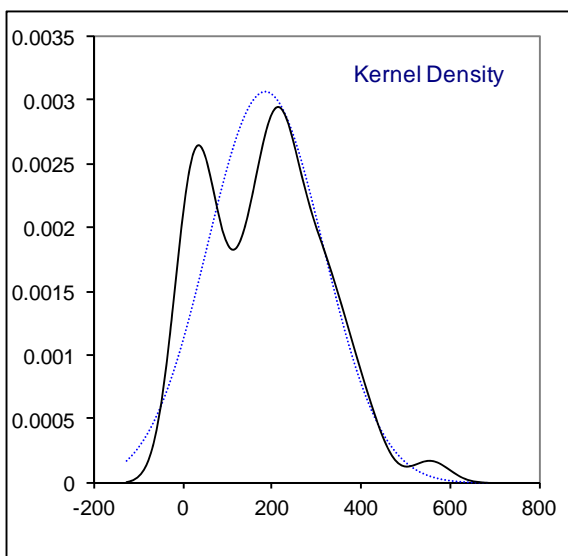
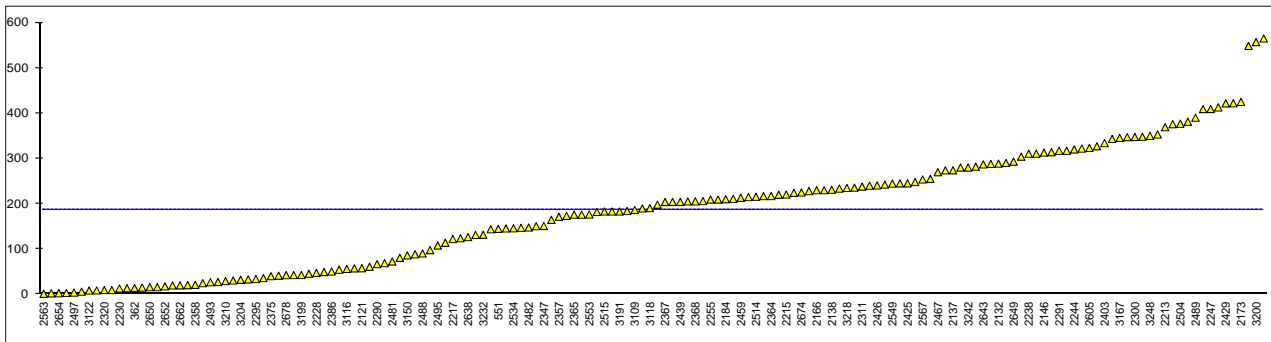
Lab 2472 first reported: <5

Lab 2662 first reported: 8

Lab 2677 first reported: 4.89

Lab 3154 first reported: 5.25

Lab 3204 first reported: 8.8

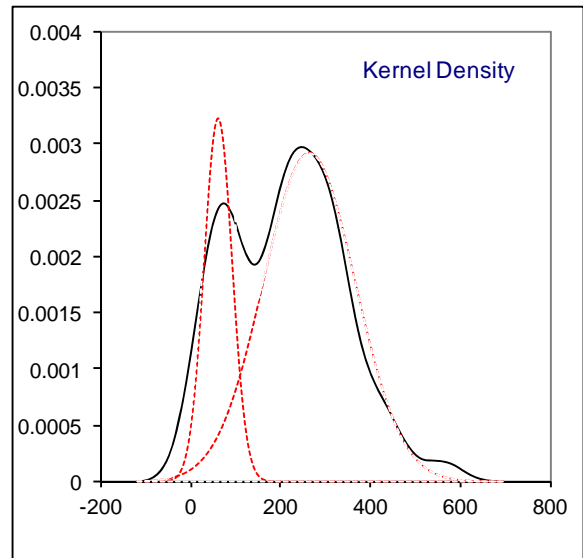
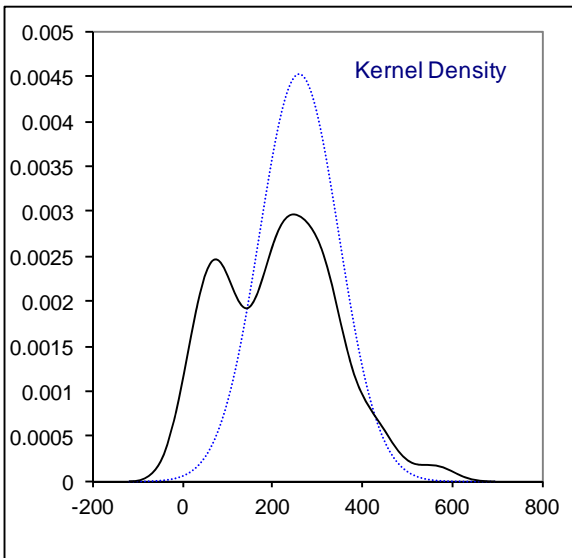
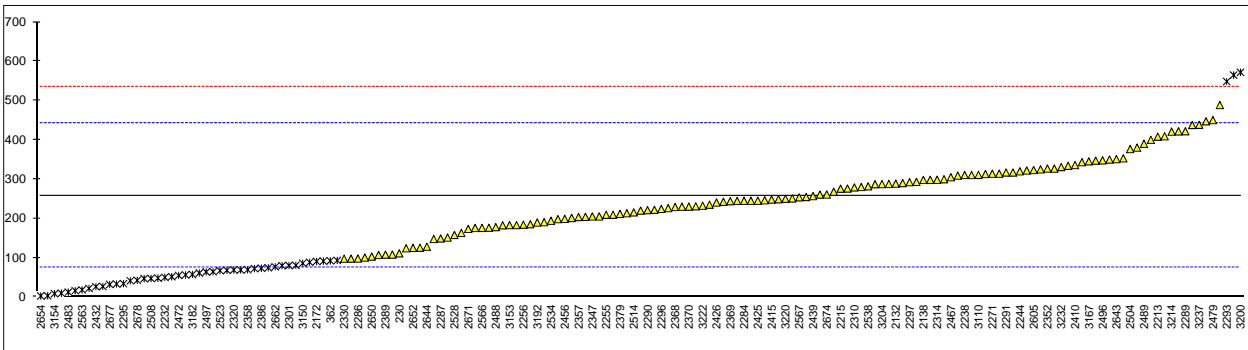


Summation of o-Aminoazotoluene and o-Toluidine in sample #15020; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	EN14362-1	80.7	ex	-1.93	2386	EN14362-1	73.2	ex	-2.01
213		-----		-----	2389	EN14362-1	107.9		-1.63
230	EN14362-1	111.2		-1.60	2390	EN14362-1	254.676		-0.04
348	in house	185.34		-0.79	2403	EN14362-1	334.0		0.82
362	in house	92	ex	-1.81	2410	EN14362-1	336		0.84
551	EN14362-1	287.92		0.32	2413	EN14362-1	9.57	ex	-2.70
622	EN14362-1	107		-1.64	2415	EN14362-1	248.1		-0.11
2115		-----		-----	2425	EN14362-1	245.1		-0.14
2120	EN14362-1	33	ex	-2.45	2426	EN14362-1	240.6127		-0.19
2121	EN14362-1	79.82	ex	-1.94	2429	EN14362-1	421.76		1.77
2129	EN14362-1	565	DG(0.05)	3.33	2432		26.15	ex	-2.52
2132	EN14362-1	288.6		0.33	2439	EN14362-1	256.8		-0.02
2137	EN14362-1	325		0.72	2442		64.26	ex	-2.11
2138	EN14362-1	298		0.43	2449		-----		-----
2139	EN14362-1	309		0.55	2452	EN14362-1	n.d.	false-?	-----
2146	EN14362-1	313		0.59	2453	EN14362-1	182.55		-0.82
2165	EN14362-1	220		-0.42	2456	EN14362-1	199		-0.64
2166	EN14362-1	230		-0.31	2459	EN14362-1	213.235		-0.49
2169		-----		-----	2467	in house	305		0.51
2170	EN14362-1	68.9	ex	-2.06	2472	EN14362-1	55	ex	-2.21
2172	EN14362-1	90.6	ex	-1.82	2475		-----		-----
2173	EN14362-1	447.3		2.05	2476	EN14362-1	205.2		-0.58
2184	EN14362-1	245		-0.14	2479	EN14362-1	450.4		2.09
2190	EN14362-1	380.3		1.32	2481	EN14362-1	163.1		-1.03
2201	EN14362-1	343.2		0.92	2482	EN14362-1	147.671		-1.20
2213	EN14362-1	408.2		1.63	2483	EN14362-1	12.38	ex	-2.67
2215	EN14362-1	275.8		0.19	2488	EN14362-1	177.97		-0.87
2217	EN14362-1	151.34		-1.16	2489	EN14362-1	390.10		1.43
2228	CPSD-AN-00607/00107	125.39		-1.44	2492	EN14362-1	317.0		0.64
2229	EN14362-1	353.1		1.03	2493	in house	26.92	ex	-2.51
2230	EN14362-1	91.3	ex	-1.81	2495	EN14362-1	108.0		-1.63
2232		50.11	ex	-2.26	2496	EN14362-1	348		0.97
2238	EN14362-1	310.6		0.57	2497	EN14362-1	63.81	ex	-2.11
2244	EN14362-1	320.0		0.67	2504	EN14362-1	376.75		1.29
2246	EN14362-1	88.44	ex	-1.84	2508	EN14362-1	47.1	ex	-2.29
2247	EN14362-1	409.35		1.64	2511	EN14632-1	41.21	ex	-2.36
2255	EN14362-1	209.3		-0.53	2514	EN14362-1	215.5		-0.47
2256	EN14362-1	184.1		-0.81	2515	EN14362-1	246.3		-0.13
2264	EN14362-1	209.34		-0.53	2516	EN14362-1	72.2	ex	-2.02
2271	EN14362-1	314		0.60	2523	EN14362-1	66.942	ex	-2.08
2284	EN14362-1	245		-0.14	2524	EN14362-1	3.14	ex	-2.77
2286	EN14362-1	98		-1.74	2528	EN14362-1	158		-1.09
2287	EN14362-1	149		-1.19	2532	EN14362-1	322		0.69
2289	EN14362-1	422		1.78	2534	EN14362-1	194		-0.70
2290	EN14362-1	221.1		-0.40	2538	64LFG82.02-4	281.9		0.26
2291	EN14362-1	317.0		0.64	2546	EN14362-1	<40	false-?	-----
2293	EN14362-1	548.539	G(0.05)	3.15	2549	EN14362-1	244.6		-0.15
2295	EN14362-1	34	ex	-2.44	2553	EN14362-1	176.36		-0.89
2296	EN14362-1	224.19		-0.37	2559		22	ex	-2.57
2297	EN14362-1	292.2		0.37	2562	GB/T17592	310.6		0.57
2300	EN14362-1	488.7		2.50	2563	EN14362-2	18.01	ex	-2.61
2301		80.14	ex	-1.93	2565	EN14362-1	48.0	ex	-2.28
2310	EN14362-1	279		0.22	2566	EN14362-1	176		-0.89
2311	EN14362-1	287.2		0.31	2567	EN14362-1	253.76		-0.05
2313	EN14362-1	298.3		0.43	2572	EN14362-1	61	ex	-2.14
2314	EN14362-1	298.5		0.44	2590		93.04	ex	-1.79
2320	EN14362-1	68.8087	ex	-2.06	2604	EN14362-1	124		-1.46
2330	EN14362-1	97.46		-1.75	2605	EN14362-1	323.3		0.71
2347	EN14362-1	205		-0.58	2613	EN14362-1	204.0		-0.59
2352	EN14362-1	327		0.75	2629	EN14362-1	400.2		1.54
2357	EN14362-1	203.7		-0.59	2638	EN14362-1	175.86		-0.90
2358	EN14362-1	69.4	ex	-2.05	2643	EN14362-1	351		1.01
2364	EN14362-1	249		-0.10	2644	EN14362-1	128		-1.41
2365	EN14362-1	222		-0.39	2649	EN14362-1	293.06		0.38
2366	EN14362-1	438		1.95	2650	EN14362-1	103		-1.69
2367	EN14362-1	226.2		-0.35	2652	EN14362-1	125.1		-1.45
2368	EN14362-1	229.1		-0.32	2654	EN14362-1	2.56	ex	-2.78
2369	EN14362-1	244		-0.16	2658	EN14362-1	15.97	ex	-2.63
2370	EN14362-1	230.2		-0.31	2662	EN14362-1	77	ex	-1.97
2373	EN14362-1	276.2		0.19	2663	EN14362-1	299.9		0.45
2375	EN14362-1	100.4		-1.71	2668	EN14362-1	242.4		-0.17
2379	EN14362-1	211.641		-0.51	2671	EN14362-1	173.34		-0.92
2674	EN14362-1	261		0.03	3190	EN14362-1	347.11		0.96

2677	EN14362-1	31.9	ex	-2.46	3191	EN14362-1	183	-0.82
2678	EN14362-1	42.5	ex	-2.34	3192	EN14362-1	189.7	-0.75
3100	EN14362-1	314		0.60	3197	EN14362-1	198.1	-0.65
3109	EN14362-1	231		-0.30	3199	CPSD-AN-00607	251	-0.08
3110	EN14362-1	310.9		0.57	3200	EN14362-1	572	DG(0.05) 3.41
3116	EN14362-1	56.15	ex	-2.19	3204	EN14362-1	287.5	0.32
3117	EN14362-1	68	ex	-2.07	3210	EN14362-1	74.5	ex -2.00
3118	EN14362-1	190.42		-0.74	3214	EN14362-1	421.05	1.77
3122	EN14362-1	46.69	ex	-2.30	3216	EN14362-1	97.71	-1.74
3146	EN14362-1	290		0.34	3218	EN14362-1	235	-0.25
3149		----		----	3220	EN14362-1	250.1	-0.09
3150	EN14362-1	86.0	ex	-1.87	3222	EN14362-1	232.4	-0.28
3153	EN14362-1	183		-0.82	3225	EN14362-1	51.62	ex -2.24
3154	BVLB82.02-2	8.41	ex	-2.71	3228	EN14362-1	268	0.10
3167	EN14362-1	345.5		0.95	3232	EN14362-1	330.8	0.79
3172		----		----	3237	in house	438.3810	1.95
3176	EN14362-1	201.3		-0.62	3242	EN14362-1	280	0.24
3182	EN14362-1	57.39	ex	-2.18	3246	EN14362-1	260.7	0.03
3185	EN14362-1	327		0.75	3248	EN14362-1	350	1.00
normality		OK						
n		128						
outliers		3 (+44ex)						
mean (n)		258.338						
st.dev. (n)		88.2328						
R(calc.)		247.052						
R(EN14362-1:12)		257.923						

For the excluded test results, see §4 and §6



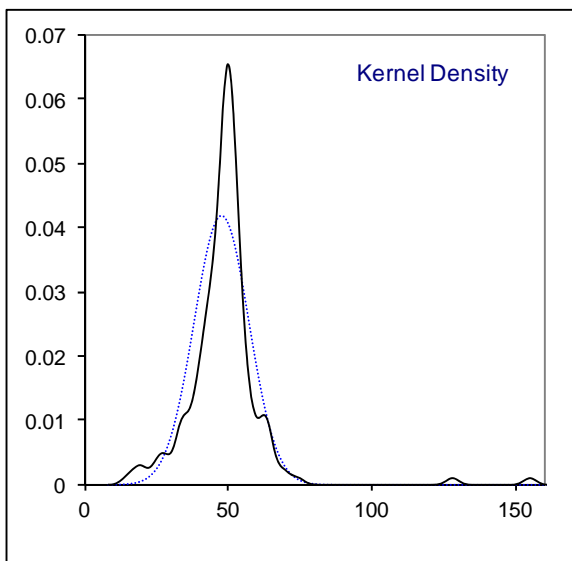
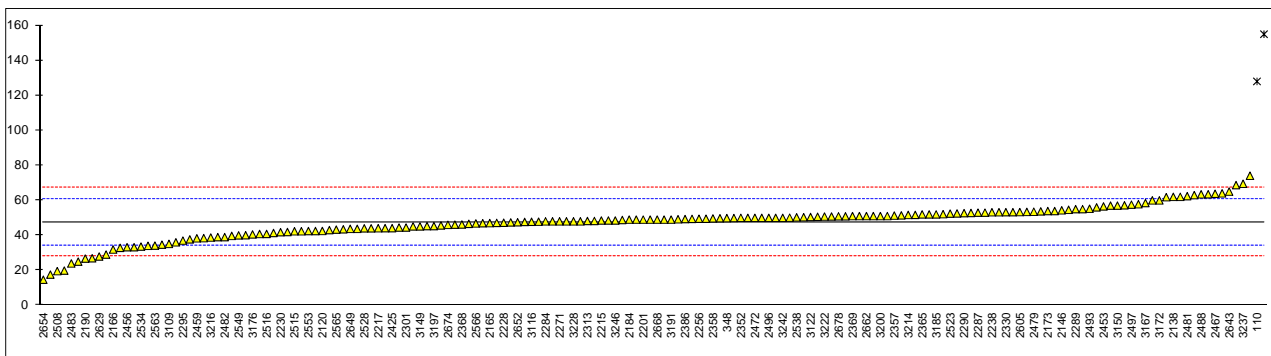
Determination of Benzidine (CASno.92-87-5) in sample #15021; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	EN14362-1	128	R(0.01)	12.26	2386	EN14362-1	49.3		0.27
213		-----		-----	2389	EN14362-1	46.94		-0.09
230	EN14362-1	34.7		-1.95	2390	EN14362-1	45.650		-0.28
348	in house	49.83		0.35	2403	EN14362-1	51.7		0.64
362	in house	155	R(0.01)	16.37	2410	EN14362-1	56		1.29
551	EN14362-1	74.09		4.05	2413		-----		-----
622	EN14362-1	<5	false -?	<-6.47	2415	EN14362-1	43.48		-0.61
2115		-----		-----	2425	EN14362-1	44.1		-0.52
2120	EN14362-1	42.5		-0.76	2426	EN14362-1	49.7725		0.34
2121	EN14362-1	68.74		3.23	2429	EN14362-1	47.13		-0.06
2129	EN14362-1	49.2		0.26	2432	EN14362-1	63.5		2.43
2132	EN14362-1	57.8		1.57	2439	EN14362-1	48.5		0.15
2137	EN14362-1	34		-2.06	2442	in house	40.78		-1.03
2138	EN14362-1	62.0		2.21	2449		-----		-----
2139	EN14362-1	60		1.90	2452	EN14362-1	n.d	false -?	-----
2146	EN14362-1	54.3		1.03	2453	EN14362-1	56.53		1.37
2165	EN14362-1	47		-0.08	2456	EN14362-1	33.2		-2.18
2166	EN14362-1	31.9		-2.38	2459	EN14362-1	38.295		-1.40
2169		-----		-----	2467	in house	63.78	C	2.48
2170	EN14362-1	53.7		0.94	2472	EN14362-1	50		0.38
2172	EN14362-1	47.7		0.03	2475	EN14362-1	51		0.53
2173	EN14362-1	53.9		0.97	2476	EN14362-1	45.1		-0.37
2184	EN14362-1	49		0.23	2479	EN14362-1	53.5		0.91
2190	EN14362-1	26.7		-3.17	2481	EN14362-1	62.4		2.27
2201	EN14362-1	49.0		0.23	2482	EN14362-1	39.011		-1.29
2213	EN14362-1	49		0.23	2483	EN14362-1	23.92		-3.59
2215	EN14362-1	48.5		0.15	2488	EN14362-1	63.45		2.43
2217	EN14362-1	44.07		-0.52	2489	EN14362-1	50.10		0.39
2228	CPSD-AN-00607/00107	47.24		-0.04	2492	EN14362-1	43.0		-0.69
2229	EN14362-1	49.9		0.36	2493	in house	55.22		1.17
2230	EN14362-1	41.8		-0.87	2495	EN14362-1	33.2		-2.18
2232	EN14362-1	44.10		-0.52	2496	EN14362-1	50		0.38
2238	EN14362-1	53.2		0.87	2497	EN14362-1	57.54		1.53
2244	EN14362-1	55.0		1.14	2504	EN14362-1	42.34		-0.79
2246	EN14362-1	50.63		0.47	2508	EN14362-1	19.54		-4.26
2247	EN14362-1	52.55		0.77	2511	EN14362-1	47.8		0.04
2255	EN14362-1	41.9		-0.85	2514	EN14362-1	40.1		-1.13
2256	EN14362-1	49.5		0.30	2515	EN14362-1	42.31		-0.79
2264	EN14362-1	19.84		-4.21	2516	EN14362-1	40.8		-1.02
2271	EN14362-1	48		0.07	2523	EN14362-1	52.423		0.75
2284	EN14362-1	48		0.07	2524	EN14362-1	17.55		-4.56
2286	EN14362-1	48		0.07	2528	EN14362-1	44		-0.53
2287	EN14362-1	53		0.84	2532	EN14362-1	61.8		2.18
2289	EN14362-1	55		1.14	2534	EN14362-1	33.6		-2.12
2290	EN14362-1	52.7		0.79	2538	64LFG82.02-2	50.3		0.42
2291	EN14362-1	50.8		0.50	2546	EN14362-1	25		-3.43
2293	EN14362-1	38.966		-1.30	2549	EN14362-1	40.0		-1.14
2295	EN14362-1	37		-1.60	2553	EN14362-1	42.45		-0.77
2296	EN14362-1	37.72		-1.49	2559	EN14362-1	36		-1.75
2297	EN14362-1	48.8		0.20	2562	GB/T17592	54.6		1.08
2300	EN14362-1	53.9		0.97	2563	EN14362-1	34.13		-2.04
2301	EN14362-1	44.48		-0.46	2565	EN14362-1	43.3		-0.64
2310	EN14362-1	52.8		0.81	2566	EN14362-1	46.8		-0.11
2311	EN14362-1	48		0.07	2567	EN14362-1	46.12		-0.21
2313	EN14362-1	48.2		0.10	2572	EN14362-1	63		2.36
2314	EN14362-1	49.5		0.30	2590	EN14362-1	29.01		-2.82
2320	EN14362-1	42.4842		-0.77	2604	EN14362-1	49.4		0.29
2330	EN14362-1	53.25		0.87	2605	EN14362-1	53.3		0.88
2347	EN14362-1	50		0.38	2613	EN14362-1	45.3		-0.34
2352	EN14362-1	50		0.38	2629	EN14362-1	27.9		-2.99
2357	EN14362-1	51.3		0.58	2638	EN14362-1	26.91		-3.14
2358	EN14362-1	49.7		0.33	2643	EN14362-1	65		2.66
2364	EN14362-1	50		0.38	2644	EN14362-1	53.4		0.90
2365	EN14362-1	52		0.68	2649	EN14362-1	43.70		-0.58
2366	EN14362-1	52		0.68	2650	EN14362-1	50		0.38
2367	EN14362-1	46.6		-0.14	2652	EN14362-1	47.5		0.00
2368	EN14362-1	46.2		-0.20	2654	EN14362-1	14.66		-5.00
2369	EN14362-1	51		0.53	2658	EN14362-1	<5	false -?	<-6.47
2370	EN14362-1	51.4		0.59	2662	EN14362-1	51		0.53
2373	EN14362-1	47.4		-0.02	2663	EN14362-1	57.0		1.44
2375	EN14362-1	49.0		0.23	2668	EN14362-1	49		0.23
2379	EN14362-1	50.366		0.43	2671	EN14362-1	44.45		-0.47

2674	EN14362-1	46	-0.23	3190	EN14362-1	53.26	0.88
2677	EN14362-1	32.85	-2.23	3191	EN14362-1	49	0.23
2678	EN14362-1	50.9	0.52	3192	EN14362-1	53.2	0.87
3100	EN14362-1	51	0.53	3197	EN14362-1	45.3	-0.34
3109	EN14362-1	35.1	-1.89	3199	CPSD-AN-00607	48.2	0.10
3110	EN14362-1	49.0	0.23	3200	EN14362-1	51	0.53
3116	EN14362-1	47.76	0.04	3204	EN14362-1	43.7	-0.58
3117	EN14362-1	64	2.51	3210	EN14362-1	38.4	-1.39
3118	EN14362-1	57.18	1.47	3214	EN14362-1	51.67	0.63
3122	EN14362-1	50.50	0.45	3216	EN14362-1	38.75	-1.33
3146	EN14362-1	52.1	0.70	3218	EN14362-1	51	0.53
3149	EN14362-1	45.1	-0.37	3220	EN14362-1	62.0	2.21
3150	EN14362-1	57.0	1.44	3222	EN14362-1	50.74	0.49
3153	EN14362-1	53	0.84	3225	EN14362-1	44.0	-0.53
3154	BVLB82.02-2	41.36	-0.94	3228	EN14362-1	48	0.07
3167	EN14362-1	58.5	1.67	3232	EN14362-1	50.90	0.52
3172	EN14362-1	60	1.90	3237	in house	69.4903	3.35
3176	EN14362-1	40.55	-1.06	3242	EN14362-1	50	0.38
3182	EN14362-1	39.68	-1.19	3246	EN14362-1	48.5	0.15
3185	EN14362-1	52	0.68	3248	EN14362-1	48	0.07

normality suspect
n 174
outliers 2
mean (n) 47.512
st.dev. (n) 9.5328
R(calc.) 26.692
R(EN14362-1:12) 18.387

Lab 2452 first reported: 94.83



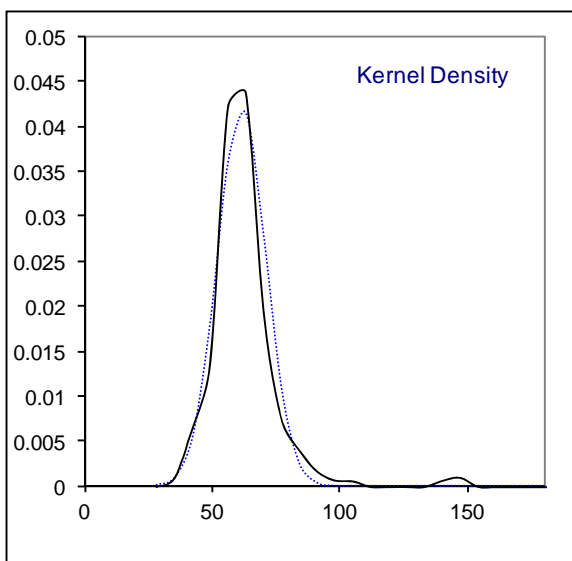
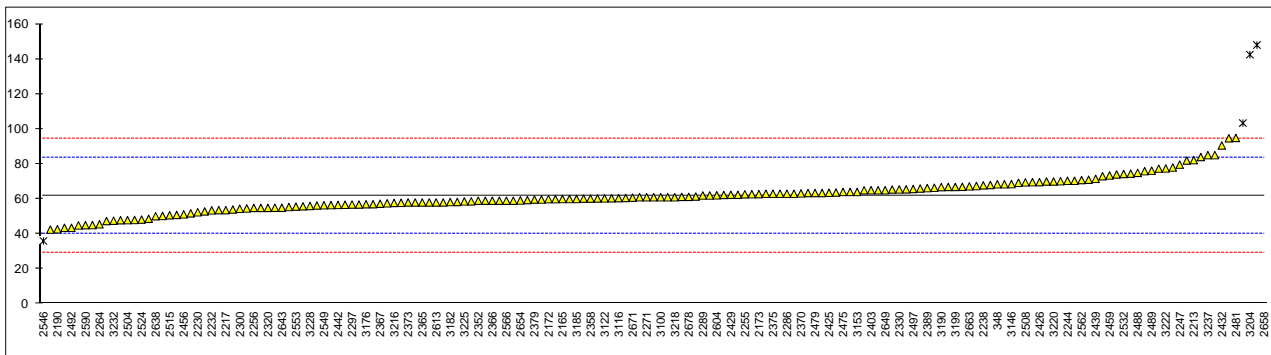
Determination of 3,3'-Dimethylbenzidine (CASno.119-93-7) in sample #15021; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	EN14362-1	148	R(0.01)	7.92	2386	EN14362-1	42.5		-1.79
213		-----		-----	2389	EN14362-1	66.21		0.39
230	EN14362-1	50.3		-1.07	2390	EN14362-1	58.646		-0.30
348	in house	68.40		0.60	2403	EN14362-1	65.0		0.28
362	in house	<5.0	false -?	<-5.24	2410	EN14362-1	63		0.10
551	EN14362-1	103.39	C,R(0.01)	3.82	2413		-----		-----
622	EN14362-1	<5	false -?	<-5.24	2415	EN14362-1	63.41		0.14
2115		-----		-----	2425	EN14362-1	63.54		0.15
2120	EN14362-1	56.5		-0.50	2426	EN14362-1	69.6083		0.71
2121	EN14362-1	64.99		0.28	2429	EN14362-1	62.38		0.04
2129	EN14362-1	71.0		0.83	2432	EN14362-1	90.6		2.64
2132	EN14362-1	64.0		0.19	2439	EN14362-1	71.5		0.88
2137	EN14362-1	48		-1.28	2442	in house	56.59		-0.49
2138	EN14362-1	53.6		-0.77	2449		-----		-----
2139	EN14362-1	70		0.74	2452	EN14362-1	n.d.	false -?	-----
2146	EN14362-1	74.5		1.16	2453	EN14362-1	85.06		2.13
2165	EN14362-1	60		-0.18	2456	EN14362-1	51.2		-0.99
2166	EN14362-1	43.5		-1.70	2459	EN14362-1	73.44		1.06
2169		-----		-----	2467	in house	69.22	C	0.67
2170	EN14362-1	59.4		-0.23	2472	EN14362-1	60		-0.18
2172	EN14362-1	59.9		-0.19	2475	EN14362-1	64		0.19
2173	EN14362-1	62.8		0.08	2476	EN14362-1	65.5		0.33
2184	EN14362-1	61		-0.09	2479	EN14362-1	63.4		0.13
2190	EN14362-1	42.7		-1.77	2481	EN14362-1	94.9		3.03
2201	EN14362-1	62.4		0.04	2482	EN14362-1	54.911		-0.65
2213	EN14362-1	82.2		1.87	2483	EN14362-1	45.07		-1.55
2215	EN14362-1	54.6		-0.68	2488	EN14362-1	74.93		1.20
2217	EN14362-1	53.62		-0.77	2489	EN14362-1	76.18		1.31
2228	CPSD-AN-00607/00107	50.90		-1.02	2492	EN14362-1	43.5		-1.70
2229	EN14362-1	56.8		-0.47	2493	in house	77.35		1.42
2230	EN14362-1	52.4		-0.88	2495	EN14362-1	60.4		-0.14
2232	EN14362-1	53.50		-0.78	2496	EN14362-1	68.0		0.56
2238	EN14362-1	67.8		0.54	2497	EN14362-1	65.68		0.34
2244	EN14362-1	70.4		0.78	2504	EN14362-1	47.89		-1.29
2246	EN14362-1	56.70		-0.48	2508	EN14362-1	69.48		0.69
2247	EN14362-1	79.64		1.63	2511	EN14362-1	58.0		-0.36
2255	EN14362-1	62.7		0.07	2514	EN14362-1	65.0		0.28
2256	EN14362-1	54.9		-0.65	2515	EN14362-1	50.67		-1.04
2264	EN14362-1	45.51		-1.51	2516	EN14362-1	52.9		-0.83
2271	EN14362-1	61		-0.09	2523	EN14362-1	70.124		0.75
2284	EN14362-1	63		0.10	2524	EN14362-1	48.23		-1.26
2286	EN14362-1	63		0.10	2528	EN14362-1	82		1.85
2287	EN14362-1	67		0.47	2532	EN14362-1	74.3		1.14
2289	EN14362-1	62		0.01	2534	EN14362-1	59		-0.27
2290	EN14362-1	65.4		0.32	2538	64LFGB82.02-2	66.9		0.46
2291	EN14362-1	68.4		0.60	2546	EN14362-1	36	R(0.01)	-2.39
2293	EN14362-1	55.405		-0.60	2549	EN14362-1	56.4		-0.51
2295	EN14362-1	57		-0.45	2553	EN14362-1	55.64		-0.58
2296	EN14362-1	55.00		-0.64	2559	EN14362-1	54		-0.73
2297	EN14362-1	56.8		-0.47	2562	GB/T17592	70.8		0.82
2300	EN14362-1	54.4		-0.69	2563		-----		-----
2301	EN14362-1	47.88		-1.29	2565	EN14362-1	60.3		-0.15
2310	EN14362-1	63.4		0.13	2566	EN14362-1	59		-0.27
2311	EN14362-1	58.3		-0.33	2567	EN14362-1	61.19		-0.07
2313	EN14362-1	55.8		-0.57	2572	EN14362-1	74		1.11
2314	EN14362-1	60.6		-0.12	2590	EN14362-1	45.02		-1.56
2320	EN14362-1	54.9235		-0.65	2604	EN14362-1	62.1		0.01
2330	EN14362-1	65.46		0.32	2605	EN14362-1	57.5		-0.41
2347	EN14362-1	62		0.01	2613	EN14362-1	58.0		-0.36
2352	EN14362-1	59		-0.27	2629	EN14362-1	51.8		-0.93
2357	EN14362-1	60.2		-0.16	2638	EN14362-1	50.14		-1.09
2358	EN14362-1	60.2		-0.16	2643	EN14362-1	55		-0.64
2364	EN14362-1	58		-0.36	2644	EN14362-1	94.7		3.02
2365	EN14362-1	58		-0.36	2649	EN14362-1	65.00		0.28
2366	EN14362-1	59		-0.27	2650	EN14362-1	73		1.02
2367	EN14362-1	57.3		-0.43	2652	EN14362-1	59.9		-0.19
2368	EN14362-1	57.8		-0.38	2654	EN14362-1	59.17		-0.25
2369	EN14362-1	59		-0.27	2658	EN14362-1	1804.96	R(0.01)	160.48
2370	EN14362-1	63.2		0.12	2662	EN14362-1	61		-0.09
2373	EN14362-1	58.0		-0.36	2663	EN14362-1	67.2		0.48
2375	EN14362-1	63.0		0.10	2668	EN14362-1	69.6		0.71
2379	EN14362-1	59.649		-0.21	2671	EN14362-1	60.76		-0.11

2674	EN14362-1	58	-0.36	3190	EN14362-1	66.81	0.45
2677	EN14362-1	47.42	-1.34	3191	EN14362-1	61	-0.09
2678	EN14362-1	61.2	-0.07	3192	EN14362-1	66.4	0.41
3100	EN14362-1	61	-0.09	3197	EN14362-1	67.4	0.50
3109	EN14362-1	44.9	-1.57	3199	CPSD-AN-00607	66.9	0.46
3110	EN14362-1	61.4	-0.05	3200	EN14362-1	66	0.37
3116	EN14362-1	60.50	-0.13	3204	EN14362-1	142.5	R(0.01) 7.42
3117	EN14362-1	78	1.48	3210	EN14362-1	48.7	-1.22
3118	EN14362-1	56.25	-0.52	3214	EN14362-1	62.74	0.07
3122	EN14362-1	60.35	-0.15	3216	EN14362-1	57.75	-0.39
3146	EN14362-1	68.5	0.60	3218	EN14362-1	61	-0.09
3149	EN14362-1	59.7	-0.21	3220	EN14362-1	70.0	0.74
3150	EN14362-1	75.9	1.29	3222	EN14362-1	77.44	1.43
3153	EN14362-1	64	0.19	3225	EN14362-1	58.6	-0.31
3154	BVLB82.02-2	63.66	0.16	3228	EN14362-1	56	-0.55
3167	EN14362-1	84.0	2.03	3232	EN14362-1	47.61	-1.32
3172	EN14362-1	63	0.10	3237	in house	85.05	C 2.13
3176	EN14362-1	56.99	-0.46	3242	EN14362-1	70.4	0.78
3182	EN14362-1	58.23	-0.34	3246	EN14362-1	62.3	0.03
3185	EN14362-1	60	-0.18	3248	EN14362-1	59	-0.27

normality suspect
n 170
outliers 5
mean (n) 61.937
st.dev. (n) 9.4967
R(calc.) 26.591
R(EN14362-1:12) 30.411

Lab 551 first reported: 120.75
Lab 2467 first reported: 104.74
Lab 3237 first reported: 113.5476



APPENDIX 2

Summary of other reported aromatic amines in sample #15020

lab	method
2228	4-amino-azobenzene 2.50 mg/kg
2293	benzidine <5 mg/kg, 4-Chloro-o-toluidine <5 mg/kg, 3,3'-dimethylbenzidine <5 mg/kg, 4,4'-diaminodiphenylsulfide <5 mg/kg
2413	o-anisidine 5.02 mg/kg
2452	benzidine: 32.44 mg/kg, 3,3'-dimethylbenzidine 47.96 mg/kg
2456	4-chloro-o-toluidine 2.4 mg/kg
2572	4-amino-azobenzene 134 mg/kg
3154	3,3'-dimethylbenzidine 5.19 mg/kg
3204	4-chloro-o-toluidine 3.6 mg/kg

Summary of other reported aromatic amines in sample #15021

lab	method
2228	4-aminodiphenyl 2.82 mg/kg
2293	4-aminodiphenyl <5 mg/kg
2413	4-aminodiphenyl 307.29 mg/kg, o-anisidine 4.93 mg/kg
2442	o-aminoazotoluene 16.17 mg/kg
2456	o-aminoazotoluene 3.1 mg/kg
2497	o-toluidine 1.04 mg/kg
2563	o-aminoazotoluene 7.58 mg/kg, 4,4'-diaminodiphenylsulfide 42.41 mg/kg
2658	4,4'-dimethyl-diaminodiphenylmethane 344.31 mg/kg

APPENDIX 3

Analytical details

Lab	#15020 extracted	#15021 extracted	Vacuum Rotary Evap	temperature used	inert gas flow	Other
110	Yes	No	only #15020	50	N2	
213						
230	Yes	Yes		60		
348	Yes	No	Yes	40	Yes	
362						
551	Yes	Yes		55		
622	Yes	Yes	Yes	50		
2115						
2120	Yes	No	Turbovap Speed Extr.	40	N2 only at the end	
2121	Yes	Yes	Buchi			
2129	No	No	Yes	50		
2132	Yes	No			Yes	
2137	Yes	Yes	Yes	40		
2138	Yes	Yes	Yes	55		
2139	Yes	Yes			Yes only #15020	
2146	Yes	No	only #15020	60	N2	
2165	Yes	Yes			N2	
2166	*	No	Yes	40	Ar	* with butylacetate, microwave 165°C 1 hr
2169						
2170	Yes	No		55		
2172				40		
2173	Yes	Yes		46.0	Yes	
2184	Yes	Yes		45		
2190	Yes	No	Yes	50		
2201	Yes	No		40	N2	
2213	Yes	No		50		
2215	Yes	No	Yes	60		
2217					Yes	
2228	Yes	No	Yes	40		
2229	Yes	Yes		45		
2230	Yes	No	Yes	70	Yes	
2232	Yes	Yes	Yes	70		
2238	Yes	No	Yes	40		
2244	Yes	No	Yes	43	Yes	
2246	Yes	Yes	Yes	40		
2247	Yes	Yes		40	No	
2255		Yes	Yes	50		
2256	Yes	No	Yes	40		
2264	Yes	Yes	Yes	70	He	
2271	Yes	Yes		45	N2	
2284	Yes	No	Yes	45		
2286	Yes	No	Yes	40	Yes	
2287	Yes	No	Yes	40	Yes	
2289	Yes	No	Yes	60		
2290	Yes	No		45		
2291	Yes	Yes		40		
2293	Yes	No		60		
2295	Yes	Yes		50		
2296	Yes	No	Yes	36 (methanol), 56 (Chlorobenzene), 46 (MTBE), 70 (extract.)	N2	
2297	Yes	No	Yes	58	Yes	
2300	Yes	Yes			Yes	
2301	Yes	No		40		
2310	Yes	No		50		

Lab	#15020 extracted	#15021 extracted	Vacuum Rotary Evap	temperature used	inert gas flow	Other
2311	Yes	No		45		
2313	Yes	No	Yes	40		
2314	Yes	No		50		
2320	Yes	Yes	Yes	60	No	
2330	Yes	Yes	Yes	40	Yes	
2347	Yes	No	Yes	60		
2352	Yes	Yes		50		
2357	Yes	Yes		70		
2358	Yes	Yes		30		
2364	Yes	No		45		
2365	Yes	Yes		40		
2366	Yes	Yes	Yes	45		
2367	Yes	No		60		
2368	Yes	No	Yes	45	N2	
2369	Yes	No		40		
2370	Yes	No	Yes	40		
2373	Yes	No	Yes	45	N2	
2375	Yes	No		55		
2379	Yes	Yes	Yes	50		
2386	Yes	No	Turbovap			
2389	Yes	No	Yes	53	n.a.	
2390	Yes	No		52		
2403	Yes	Yes	Yes	40		
2410	Yes	Yes		30		
2413	Yes	No	Yes	40		
2415	Yes	Yes		45		
2425	Yes	No	Yes	49	N2	
2426	Yes	No	Yes	60		
2429	Yes	No	Yes	40		
2432						
2439	Yes	No		52		
2442	No	No		50		
2449						
2452	Yes	No			N2	
2453	Yes	No	Yes	45		
2456	Yes	No	Yes	45		
2459	Yes	No		40		
2467	Yes	No	Yes	55 (chlorobenzene) 40 (MTBE)		
2472	Yes	No		38	N2	
2475	Yes	No	Yes			
2476	Yes	Yes	Yes	50		
2479	Yes	Yes	Yes	46.0	Yes	
2481	Yes	No	Yes	60 (chlorobenzene) 28 (ether)		
2482	Yes	No			Yes	
2483						
2488	Yes	Yes	Yes	40	Yes	
2489	Yes	Yes		40	No	
2492	No	No				
2493	No	No	Yes	35	Yes	
2495	Yes	Yes	Yes	60		
2496	Yes	No	Yes	40		
2497	Yes	No	Yes	40		
2504	Yes	Yes	Yes	60		
2508	Yes	No	Syncore			
2511	Yes	No	Yes	35		
2514	Yes	No	Yes	45		
2515						
2516	Yes	No	Yes	35		
2523						

Lab	#15020 extracted	#15021 extracted	Vacuum Rotary Evap	temperature used	inert gas flow	Other
2524	No	No	Yes	40	N2	
2528	Yes	No		40	Yes	
2532	Yes	Yes	Yes	50 (chlorobenzene) 60 (extraction)		
2534	Yes	Yes	Yes	52		
2538	Yes	Yes			Yes	
2546	Yes	Yes	Yes	45	Yes	
2549	Yes	Yes		40		
2553	Yes	Yes		55		
2559	Yes	Yes	Yes	60		
2562	No	No		45		
2563						
2565	Yes	No	Yes	35		
2566	Yes	No		40		
2567	Yes	No		70		
2572	Yes	No		58		
2590	Yes	No	Yes Polyvap	50		
2604	Yes	No	Analyst	42		extracted with Xylole
2605	Yes	No	Yes	35	Yes	
2613	Yes	Yes		70		
2629				45	Ar	
2638	Yes	Yes	Yes	50	Yes	
2643	Yes	Yes				
2644	Yes	No	Yes	45		
2649	Yes	No	Yes	60 (chlorobenzene) 40 (MTBE)	Yes	
2650	Yes	Yes	Yes	56		
2652	Yes	No	Yes	55 (chlorobenzene) 45 (MTBE)		
2654	Yes	No	Yes	50		
2658	No	No		42		
2662	Yes	No		45		both samples were constanted to 5 ml
2663						
2668	Yes	No			Yes	
2671	Yes	No		50		
2674	Yes	Yes			Yes	
2677	Yes	No	Yes	55		
2678	Yes	Yes		45		
3100	Yes	No		35		
3109	Yes	No	Yes	40	Yes	
3110	Yes	No	Yes	60	Yes	
3116	Yes	No	Yes	40	Yes	
3117	Yes	Yes		70 / 40		extracted with dimethylbenzene
3118	Yes	Yes	Yes	40		
3122	Yes	No	Yes	60		
3146	Yes	Yes	Yes	45		
3149	Yes	No	Yes	50	N2	
3150	Yes	Yes	Yes	40		
3153	Yes	No	Yes	35 (MTBE)		
3154	No	No	Yes	50	Yes	
3167	Yes	Yes	Yes	35		
3172						
3176	No	No			Yes	
3182	Yes	No	Yes	40		
3185	Yes	No	Yes	35	Yes	
3190	Yes	Yes	Yes	40		
3191	No	No	Yes	50	Yes	
3192	Yes	No	Yes	50	Yes	
3197	Yes	No		40		
3199	Yes	No	Yes	40		
3200	Yes	Yes	Yes	60		

Lab	#15020 extracted	#15021 extracted	Vacuum Rotary Evap	temperature used	inert gas flow	Other
3204	Yes	Yes	Yes	60		
3210	Yes	Yes			Yes	
3214	Yes	No	Yes	70 / 35 (watertemperature)		
3216	Yes	No		40	Yes	
3218	Yes	No	Yes	40		
3220	Yes	Yes		40		
3222	Yes	No	Yes	46	Yes	
3225	Yes	No	Yes	58		
3228	Yes	Yes			Yes	
3232	Yes	No		50		
3237	No	No		60		Xylene
3242	Yes	Yes	Turbo e-vaporator			
3246	Yes	No			Yes	
3248	Yes	Yes	Yes	60 (chlorobenzene) 45 (MTBE)	Yes	

APPENDIX 4

Number of participants per country

6 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in CAMBODIA
1 lab in CAMBODIA, Kingdom of
1 lab in EGYPT
1 lab in FINLAND
5 labs in FRANCE
14 labs in GERMANY
1 lab in GREECE
1 lab in GUATEMALA
10 labs in HONG KONG
2 labs in HUNGARY
16 labs in INDIA
3 labs in INDONESIA
11 labs in ITALY
5 labs in JAPAN
6 labs in KOREA
1 lab in MAURITIUS
1 lab in MEXICO
2 labs in MOROCCO
45 labs in P.R. of CHINA
6 labs in PAKISTAN
1 lab in PERU
2 labs in PORTUGAL
1 lab in ROMANIA
2 labs in SINGAPORE
5 labs in SPAIN
2 labs in SRI LANKA
1 lab in SWITZERLAND
5 labs in TAIWAN R.O.C.
3 labs in THAILAND
3 labs in TUNISIA
7 labs in TURKEY
3 labs in U.S.A.
1 lab in UNITED KINGDOM
7 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*, **351**, 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*, **76**, 926, (1993)
- 14 G. Rohm, J. Bohnen & H. Kruesmann, *GIT Labor-Fachzeitschrift*, p 1080, **11**, (1997)
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, **25**(2), pp. 165-172, (1983)