

**Results of Proficiency Test
AZO dyes in textile
February 2016**

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 2015/2016, it was decided to continue the PT for the analysis of banned aromatic amines from AZO dyes in textile. In this interlaboratory study, 163 laboratories in 36 different countries registered for participation (see appendix 4). In this report, the results of the 2016 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 #16515 and #16516), each dyed with different AZO dyes. Both samples were especially prepared by a third party. Analysis for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. The participants were requested to report test results using the indicated units and to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

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 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 organisation of this PT was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis internet site www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The two different bulk textile samples, a purple cotton textile (sample #16515) and a beige cotton textile (sample #16516) were dyed with different AZO-dyes. From the first batch, 200 samples with small pieces of fabric, of approximately 3 gram were prepared and labelled #16515. From the second batch, also 200 samples with small pieces of fabric, of approximately 3 gram were prepared and labelled #16516. The homogeneity of the subsamples #16515 and #16516 was checked by determination of aromatic amines on 8, resp. 7 stratified randomly selected samples. See the following tables for the test results.

	<i>benzidine in mg/kg</i>	<i>3,3'-dimethoxybenzidine in mg/kg</i>
sample #16515-1	94.2	91.5
sample #16515-2	85.3	79.9
sample #16515-3	93.5	90.9
sample #16515-4	94.7	85.0
sample #16515-5	99.7	95.5
sample #16515-6	88.5	90.2
sample #16515-7	86.9	83.9
sample #16515-8	93.9	89.5

Table 1: homogeneity test results of subsamples #16515

	<i>benzidine in mg/kg</i>
sample #16516-1	8.89
sample #16516-2	8.82
sample #16516-3	8.73
sample #16516-4	8.18
sample #16516-5	7.94
sample #16516-6	8.12
sample #16516-7	8.63

Table 2: homogeneity test results of subsamples #16516

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>benzidine in mg/kg</i>	<i>3,3'-dimethoxybenzidine in mg/kg</i>
r (observed) #16515	13	14
r (observed) #16516	1.1	--
reference test method	EN14362-1:2012	EN14362-1:2012
r (reference test method)	1.6 / 16	16

Table 3: repeatabilities of subsamples #16515 and #16516

The calculated repeatabilities of the test results were in agreement with the repeatabilities mentioned in (or estimated from) the reference test method EN14362-1. Therefore, homogeneity of the subsamples was assumed. To the participating laboratories was sent 1 sample labelled #16515 and 1 sample labelled #16516 on February 10, 2016.

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 a detailed report form, on which the units were prescribed as well as the reference test method and a letter of instructions were prepared and made available on the data entry portal www.kmpd.co.uk/sgs-iis-cts/. The laboratories were also requested to report some of the test conditions that the laboratory has used. A form to confirm receipt of the samples and a letter of instructions were added to the sample package.

3 RESULTS

During five weeks after sample despatch, the results of the individual laboratories were gathered via the data entry portal www.kmpd.co.uk/sgs-iis-cts/. The reported test results are tabulated per sample in appendix 1 of this report. The laboratories are represented by the code numbers.

Directly after the deadline, a reminder 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. Results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

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 the original results per determination were submitted subsequently to Dixon, Grubbs and or Rosner General ESD 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. 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 General ESD test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have 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 on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected 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. 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.

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

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.

4 EVALUATION

During the execution of this proficiency test some reporting problems occurred. Seven participants reported the test results after the deadline and two participants did not report any test results. Finally, 161 participants did report 486 numerical results. Observed were 15 outlying results, which is 3.1% 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.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section, the results are discussed per sample and per component. 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 #16515:

Benzidine (CASNo. 92-87-5):

The determination of this aromatic amine at a concentration level of 97 mg/kg was problematic. Five statistical outliers and three false negative test results were detected. 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.

3,3'-Dimethoxybenzidine (CASno. 119-90-4):

The determination of this aromatic amine at a concentration level of 98 mg/kg was problematic. Four statistical outliers and one false negative test result were detected. 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 #16516:Benzidine (CASno. 92-87-5):

The determination of this aromatic amine at a concentration level of 20 mg/kg was problematic. Six statistical outliers and five false negative test results were detected. 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.

General:

The large majority of participating laboratory did not detect any other banned aromatic amines than benzidine and 3,3'-dimethoxybenzidine. Two participants reported also the presence of other banned aromatic amines at different concentration levels in sample #16515 (see Appendix 2). Ten participants reported also the presence of other banned aromatic amines at different concentration levels in sample #16516 (see Appendix 2). A number of participants reported low concentrations of aniline in both samples. One participant most probably did mix-up (the results of) the two samples. This was corrected before the evaluation.

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)
Benzidine	mg/kg	153	97	45	37
3,3'-Dimethoxybenzidine	mg/kg	152	98	43	34

Table 4: reproducibilities of the aromatic amines in textile sample #16515

Parameter	unit	n	average	2.8 * sd	R(target)
Benzidine	mg/kg	144	20	10	8

Table 5: reproducibilities of the aromatic amines in textile sample #16516

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

5 COMPARISON WITH PREVIOUS INTERLABORATORY STUDIES

The dispersions in the reported test results of the in this PT evaluated 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 table 6.

Parameter	Feb. 2016	March 2015	April 2014	March 2013	March 2012	2004 - 2011 PTs	target
4-Aminodiphenyl	n.e.	n.e.	(21%)*	n.e.	18%	18-36%	28%
Benzidine	17-18%	20%	15%	n.e.	21%	18-35%	14%
4-Chloro-o-toluidine	n.e.	n.e.	24%	n.e.	n.e.	n.e.	16%
<i>o</i> -Aminoazotoluene	n.e.	(48%)*	n.e.	n.e.	n.e.	n.e.	28%
<i>p</i> -Chloroaniline	n.e.	n.e.	n.e.	n.e.	n.e.	27%	16%
2,4-Diaminoanisole	n.e.	n.e.	n.e.	52%	n.e.	24%	16%
4,4'-Diaminodiphenylmethane	n.e.	n.e.	21%	n.e.	n.e.	21%	15%
3,3'-Dimethoxybenzidine	16%	n.e.	21%	16%	17%	17-31%	13%
3,3'-Dimethylbenzidine	n.e.	15%	n.e.	n.e.	n.e.	17-32%	18%
4,4'-Diamino-3,3'-dichlorodiphenylmethane	n.e.	n.e.	n.e.	n.e.	n.e.	20-35%	16%
4,4'-Diaminodiphenylether	n.e.	n.e.	n.e.	n.e.	n.e.	15%	16%
4,4'-Diaminodiphenylsulfide	n.e.	n.e.	n.e.	n.e.	n.e.	18-26%	16%
4,4'-Methyl-bis(2-chloro-aniline)	n.e.	n.e.	n.e.	n.e.	n.e.	43%	22%
2-Naphtylamine	n.e.	n.e.	n.e.	n.e.	n.e.	27-41%	18%
<i>o</i> -Toluidine	n.e.	(70%)*	31%	27%	n.e.	19-38%	22%
Sum of <i>o</i> -aminoazotoluene and <i>o</i> -Toluidine	n.e.	34%	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 or otherwise arbitrary

From the above table it is clear that the quality for the detected banned dyes is quite stable over the last years.

6 DISCUSSION

In the previous PT (iis15A02T) iis tried to find a cause for the observed large dispersion of the reported test results in the details of the extraction step and the subsequent removal of the extraction solvent:

quote

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.

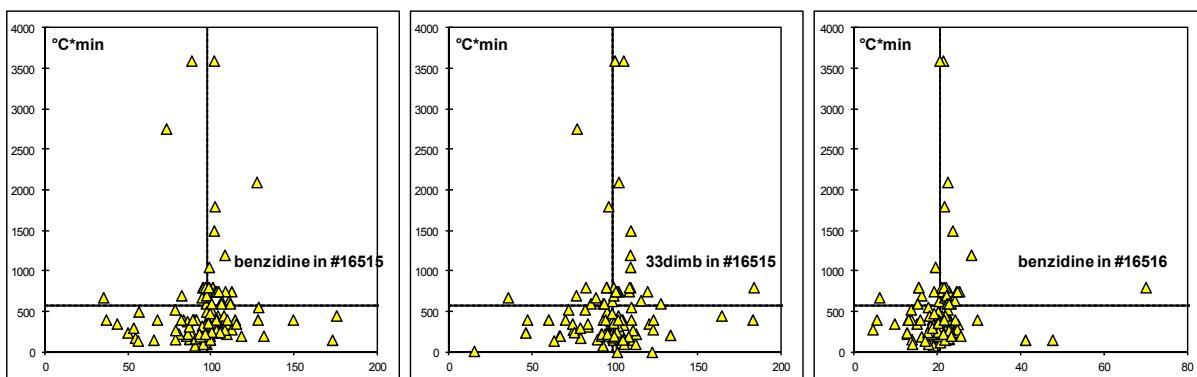
unquote

Therefore in this PT the participants were requested to report some details about this. From the reported details, it is clear that most participants treated the fabric samples as the test method described. According to the method, the two cotton fabrics need not be extracted with chlorobenzene and the majority of the laboratories did follow this. Only 14 (=9%) laboratories did perform the extraction with chlorobenzene. They did not report any significant deviating test results. And 9 laboratories reported to have used another extraction solvent, of which two laboratories (2727 & 2738) reported to have used a citrate solution with pH=6.

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. Also the time needed between reduction and actual analysis may be important.

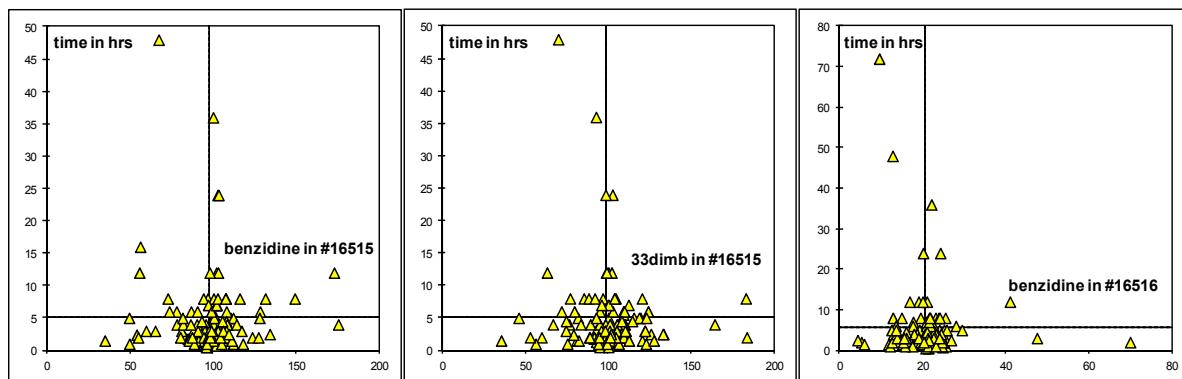
The time and temperatures used to evaporate the extraction solvent varies over a wide range from only 5 minutes at 30 °C upto 60 minutes at 70°C. The majority of the laboratories used temperatures between 40 and 50°C during 5-20 minutes.

Surprisingly no relation could be found between the conditions used and the concentration of aromatic amine detected, see below graphs in which temperature in °C times time in minutes is plotted against the reported concentrations in mg/kg.

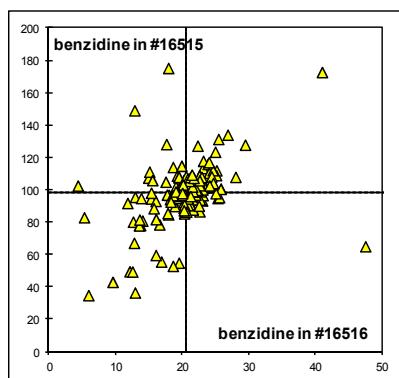


The 16 laboratories that evaporated the extraction solvent to complete dryness needed minimum 1.5 hours and maximum 8 hours (on average 4.5 hours) and did not report any significant deviating test results.

The total time from the start of the reduction step till start of HPLC/GC analysis varies over a wide range from 30 minutes upto 7 days! The laboratory that needed only 30 minutes for this period evaporated the MTBE until 1 ml residue in only 5-6 minutes at a temperature of 50°C. Surprisingly no relation could be found between the time needed and the concentration of aromatic amine detected, see below graphs.



However, some correlation is observed when the test results for benzidine in both samples is compared, see below graph.



The tentative conclusion from the above investigation may be that the (in)stability of the investigated aromatic amines benzidine and 3,3'-dimethoxybenzidine is not the cause for the observed dispersion of the test results as suggested in previous PTs. Other factors like for example the calibration standards may be the cause of the observed dispersion.

7 CONCLUSION

The dispersion in the test results observed may be caused by the preparation of the sample (reduction to release the aromatic amine and subsequent concentration) and/or by the analytical identification and quantification (incl. calibration). Consequently, the reproducibility may not be improved by only one change in the analysis. Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

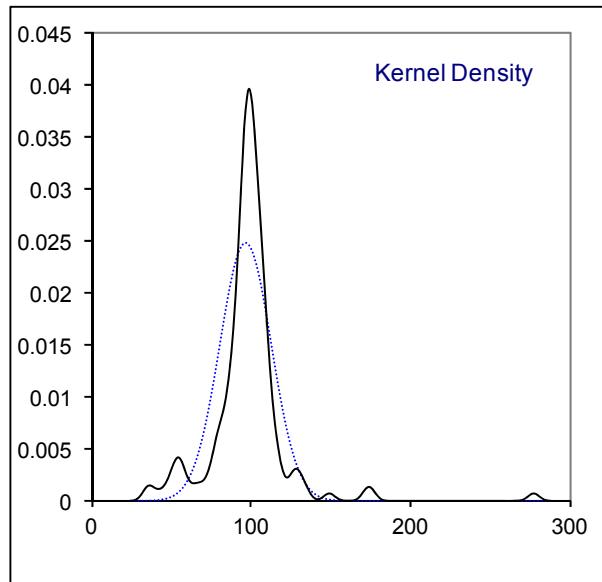
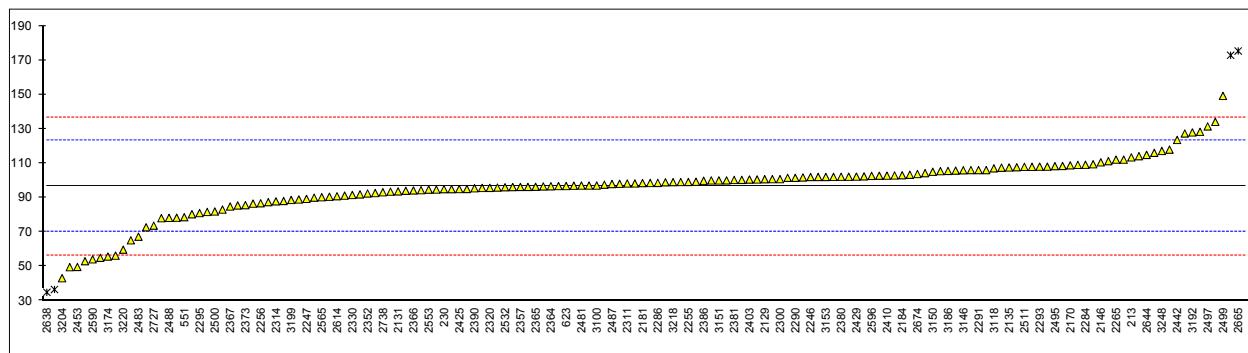
Determination of Benzidine (CASno.92-87-5) in sample #16515; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EN14362-1	83.03		-1.01	
213	EN14362-1	113.4		1.26	
230	EN14362-1	94.9		-0.13	
348	In house	93.44		-0.24	
362	EN14362-1	56.2		-3.02	
551	EN14362-1	78.60850469		-1.35	
623	EN14362-1	96.74		0.01	
840	EN14362-1	102.36		0.43	
2102	In house	91.85		-0.35	
2108	EN14362-1	101.5		0.37	
2115	EN14362-1	172.81	R(0.01)	5.71	
2120	EN14362-1	94.65		-0.14	
2129	EN14362-1	100.7		0.31	
2131	EN14362-1	93.59		-0.22	
2132	EN14362-1	100.02		0.26	
2135	EN14362-1	107.5		0.82	
2138	EN14362-1	116.03		1.46	
2139	EN14362-1	106		0.71	
2146	EN14362-1	110.5		1.04	
2165	EN14362-1	102		0.41	
2166	EN14362-1	95.60		-0.07	
2170	EN14362-1	108.76		0.91	
2181	EN14362-1	98.44		0.14	
2184	EN14362-1	103		0.48	
2190	EN14362-1	ND		-----	false negative test result?
2201	EN14362-1	100.32		0.28	
2232	EN14362-1	89.97		-0.50	
2246	EN14362-1	101.98		0.40	
2247	EN14362-1	89.25		-0.55	
2255	EN14362-1	99.1		0.19	
2256	EN14362-1	86.7		-0.74	
2265	EN14362-1	112.074		1.16	
2284	EN14362-1	109.1		0.94	
2286	EN14362-1	98.55		0.15	
2287	EN14362-1	105.7		0.68	
2290	EN14362-1	101.52		0.37	
2291	EN14362-1	106.0		0.71	
2293	EN14362-1	108	C	0.86	first reported 216.0
2295	EN14362-1	81		-1.17	
2296	EN14362-1	72.735		-1.79	
2300	EN14362-1	100.74		0.31	
2310	EN14362-1	93.96		-0.20	
2311	EN14362-1	98.2		0.12	
2313	EN14362-1	88.91		-0.57	
2314	EN14362-1	87.83		-0.66	
2320	EN14362-1	95.6635		-0.07	
2330	EN14362-1	91.5	C	-0.38	possibly sample mix-up; reported this test result for sample #16516
2347	EN14362-1	95		-0.12	
2350	EN14362-1	107.65		0.83	
2352	EN14362-1	92.4		-0.31	
2357	EN14362-1	96.2		-0.03	
2358	EN14362-1	101.71		0.38	
2364	EN14362-1	96.6		0.00	
2365	EN14362-1	96.3		-0.02	
2366	EN14362-1	94.16		-0.18	
2367	EN14362-1	84.8		-0.88	
2368	EN14362-1	96.5		-0.01	
2369	EN14362-1	97		0.03	
2370	EN14362-1	99.03		0.18	
2373	EN14362-1	85.6		-0.82	
2375	EN14362-1	91.15		-0.41	
2379	EN14362-1	Not detected		-----	false negative test result?
2380	EN14362-1	102.10		0.41	
2381	EN14362-1	100.25		0.28	
2386	EN14362-1	99.8		0.24	
2389	EN14362-1	102.02		0.41	
2390	EN14362-1	95.414		-0.09	
2403	EN14362-1	100.51		0.29	
2410	EN14362-1	102.75		0.46	
2425	EN14362-1	94.93		-0.12	
2426		-----		-----	
2429	EN14362-1	102.2		0.42	
2432	EN14362-1	96.91		0.02	

2442	In house	123.54	2.02
2453	EN14362-1	49.63	-3.52
2456	EN14362-1	80.3	-1.22
2459	EN14362-1	100.55	0.30
2467	EN14362-1	85.42	-0.84
2472	EN14362-1	96.21	-0.03
2475	EN14362-1	109.01	0.93
2476	EN14362-1	105.4	0.66
2481	In house	97.0	0.03
2482	EN14362-1	128.27	2.37
2483		67.25	-2.20
2487	EN14362-1	97.90	0.10
2488	EN14362-1	78.18	-1.38
2489	EN14362-1	98.49	0.14
2492	EN14362-1	112.1	1.16
2495	EN14362-1	108.3	0.88
2497	EN14362-1	131.42	2.61
2499	EN14362-1	149.170	C 3.94 first reported 141.911
2500	EN14362-1	81.90	-1.10
2511	EN14362-1	107.9	0.85
2514	EN14362-1	95.82	-0.06
2516	EN14362-1	114.1	1.31
2522	EN14362-1	134.08	2.81
2528	EN14362-1	90.50	-0.46
2532	EN14362-1	96	-0.04
2534	EN14362-1	94.9	-0.13
2553	EN14362-1	94.64	-0.15
2563	EN14362-1	78	-1.39
2565		90.2	-0.48
2567	EN14362-1	108.0	0.86
2569	EN14362-1	88	-0.64
2572	EN14362-1	102.13	0.42
2590	EN14362-1	54.04	-3.19
2596	In house	102.64	0.45
2605	EN14362-1	97.5	0.07
2609	EN14362-1	127.3	2.30
2612	EN14362-1	< 10	<-6.49 false negative test result?
2614	EN14362-1	90.8	-0.43
2629	EN14362-1	94.28	-0.17
2638	EN14362-1	34.877	C,R(0.05) -4.62 first reported 51.781
2643	EN14362-1	102.87	0.47
2644	EN14362-1	114.85	1.37
2662	EN14362-1	96.7	0.01
2665	EN14362-1	175.3	R(0.01) 5.90
2674	EN14362-1	103.7	0.53
2678	EN14362-1	98.91	0.17
2706	In house	49.5	-3.53
2713	EN14362-1	81.62	-1.12
2717	EN14362-1	100.72	0.31
2718	EN14362-1	54.9449	-3.12
2723	EN14362-1	36.52	R(0.05) -4.50
2724	EN14362-1	65.13	C -2.36 first reported 206.18
2727	EN14362-1	73.66	-1.72
2734	EN14362-1	78.3	-1.37
2738	In house	93.1	-0.26
2749		----	----
3100	EN14362-1	97.0	0.03
3109	In house	100.0	0.26
3110	EN14362-1	99.21	0.20
3116	EN14362-1	108.39	0.88
3117	EN14362-1	92.64	-0.29
3118	EN14362-1	107.02	0.78
3122	EN14362-1	86.48	-0.76
3146	EN14362-1	105.9	0.70
3150	EN14362-1	105	0.63
3151	EN14362-1	100.00	0.26
3153	EN14362-1	102.0	0.41
3154	EN14362-1	277.5	R(0.01) 13.55
3172	EN14362-1	104.23	0.57
3174	EN14362-1	55.67	-3.06
3176	EN14362-1	111.21	1.10
3185	EN14362-1	96.13	-0.03
3186	EN14362-1	105.5	0.67
3191	EN14362-1	103.3	0.50
3192	EN14362-1	127.9	2.35
3197	EN14362-1	107.9	0.85
3199	EN14362-1	88.6	-0.60

3204		43.08	-4.01
3210	In house	98.20	0.12
3214	EN14362-1	102.71	0.46
3215	EN14362-1	117.92	1.60
3216	EN14362-1	52.97	-3.27
3218	EN14362-1	99	0.18
3220	EN14362-1	59.69	-2.76
3222	EN14362-1	106.0	0.71
3225	EN14362-1	107.4	0.81
3228	EN14362-1	98	0.11
3233	EN14362-1	109.40	0.96
3237	In house	87.35	-0.69
3248	EN14362-1	117.12	1.54

normality not OK
 n 153
 outliers 5
 mean (n) 96.578
 st.dev. (n) 16.1559
 R(calc.) 45.237
 R(EN14362-1:12) 37.376



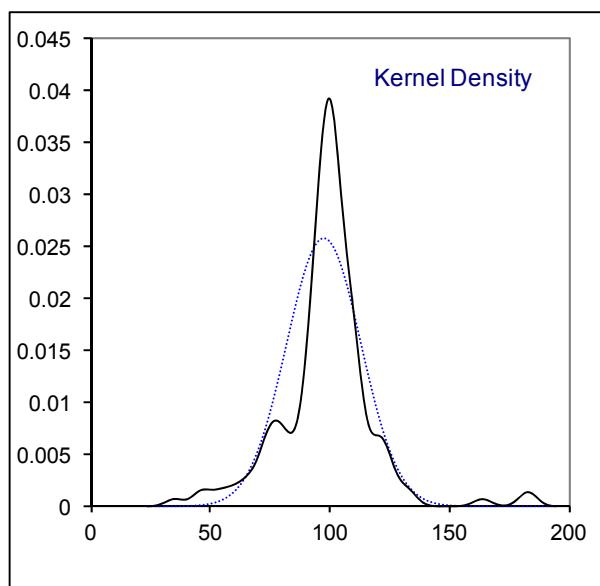
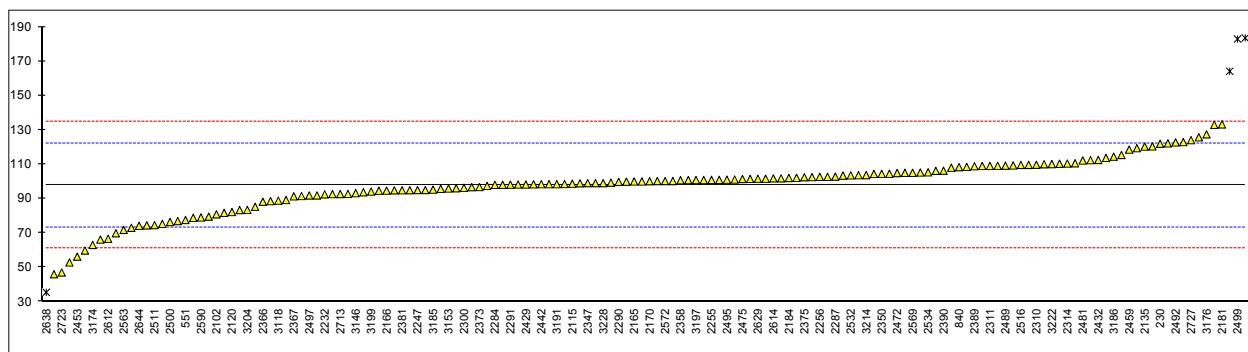
Determination of 3,3'-Dimethoxybenzidine (CASno. 119-90-4) in sample #16515; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EN14362-1	59.8		-3.10	
213	EN14362-1	106.15		0.68	
230	EN14362-1	121.9		1.96	
348	In house	120.37		1.84	
362	----	----		----	
551	EN14362-1	77.5603913		-1.65	
623	EN14362-1	85.26		-1.02	
840	EN14362-1	108.31		0.85	
2102	In house	80.85		-1.38	
2108	EN14362-1	109.3		0.94	
2115	EN14362-1	98.69		0.07	
2120	EN14362-1	82.2		-1.27	
2129	EN14362-1	109.1		0.92	
2131	EN14362-1	113.65		1.29	
2132	EN14362-1	99.86		0.17	
2135	EN14362-1	120.3		1.83	
2138	EN14362-1	103.61		0.47	
2139	EN14362-1	110		0.99	
2146	EN14362-1	115.4		1.43	
2165	EN14362-1	100		0.18	
2166	EN14362-1	94.66		-0.26	
2170	EN14362-1	100.12		0.19	
2181	EN14362-1	133.20		2.88	
2184	EN14362-1	102		0.34	
2190	EN14362-1	73		-2.02	
2201	EN14362-1	96.65		-0.10	
2232	EN14362-1	92.49		-0.44	
2246	EN14362-1	101.46		0.30	
2247	EN14362-1	95.0		-0.23	
2255	EN14362-1	100.9		0.25	
2256	EN14362-1	102.7		0.40	
2265	EN14362-1	119.342		1.75	
2284	EN14362-1	98.0		0.01	
2286	EN14362-1	108.45		0.87	
2287	EN14362-1	102.8		0.41	
2290	EN14362-1	99.73		0.16	
2291	EN14362-1	98.1		0.02	
2293	EN14362-1	109	C	0.91	first reported 218.0
2295	EN14362-1	105		0.59	
2296	EN14362-1	76.942		-1.70	
2300	EN14362-1	96.27		-0.13	
2310	EN14362-1	109.7		0.97	
2311	EN14362-1	109		0.91	
2313	EN14362-1	104.38		0.53	
2314	EN14362-1	110.4		1.03	
2320	EN14362-1	92.7280		-0.42	
2330	EN14362-1	100.8	C	0.24	possibly sample mix-up; reported this test result for sample #16516
2347	EN14362-1	99		0.10	
2350	EN14362-1	104.43		0.54	
2352	EN14362-1	98.2		0.03	
2357	EN14362-1	98.9		0.09	
2358	EN14362-1	100.71		0.24	
2364	EN14362-1	98.4		0.05	
2365	EN14362-1	94.8		-0.25	
2366	EN14362-1	88.19		-0.79	
2367	EN14362-1	91.3		-0.53	
2368	EN14362-1	100.2		0.19	
2369	EN14362-1	99		0.10	
2370	EN14362-1	103.4		0.45	
2373	EN14362-1	96.7		-0.09	
2375	EN14362-1	102.45		0.38	
2379	EN14362-1	66.1900		-2.58	
2380	EN14362-1	98.31	C	0.04	
2381	EN14362-1	94.836		-0.24	
2386	EN14362-1	81.7		-1.31	
2389	EN14362-1	108.84		0.90	
2390	EN14362-1	106.256		0.69	
2403	EN14362-1	110.29		1.02	
2410	EN14362-1	102.50		0.38	
2425	EN14362-1	88.54		-0.76	
2426	----	----		----	
2429	EN14362-1	98.2		0.03	
2432	EN14362-1	112.51		1.20	

2442	In house	98.38	0.05
2453	EN14362-1	56.23	-3.39
2456	EN14362-1	91.5	-0.52
2459	EN14362-1	118.53	1.69
2467	EN14362-1	97.37	-0.04
2472	EN14362-1	104.89	0.58
2475	EN14362-1	101.41	0.29
2476	EN14362-1	102.7	0.40
2481	In house	112.2	1.17
2482	EN14362-1	109.63	0.96
2483		69.85	-2.28
2487	EN14362-1	98.51	0.06
2488	EN14362-1	89.18	-0.71
2489	EN14362-1	109.16	0.92
2492	EN14362-1	122.7	2.03
2495	EN14362-1	101.0	0.26
2497	EN14362-1	91.82	-0.49
2499	EN14362-1	182.865	C,R(0.01) 6.93 first reported 149.726
2500	EN14362-1	76.41	-1.75
2511	EN14362-1	74.6	-1.89
2514	EN14362-1	100.00	0.18
2516	EN14362-1	109.6	0.96
2522	EN14362-1	125.70	2.27
2528	EN14362-1	94.89	-0.24
2532	EN14362-1	103.5	0.46
2534	EN14362-1	105.3	0.61
2553	EN14362-1	112.37	1.19
2563	EN14362-1	71.8	-2.12
2565		105.2	0.60
2567	EN14362-1	96	-0.15
2569	EN14362-1	105	0.59
2572	EN14362-1	100.21	0.19
2590	EN14362-1	79.02	-1.53
2596		----	---- test result probably mixed up with test result of 3,3'-dimethylbenzidine
2605	EN14362-1	95.7	-0.17
2609	EN14362-1	102.1	0.35
2612	EN14362-1	66.65	-2.54
2614	EN14362-1	101.8	0.32
2629	EN14362-1	101.54	0.30
2638	EN14362-1	35.454	C,R(0.05) -5.09 first reported 62.832
2643	EN14362-1	110.57	1.04
2644	EN14362-1	74.2	-1.93
2662	EN14362-1	93.7	-0.34
2665	EN14362-1	164.1	R(0.05) 5.40
2674	EN14362-1	101.8	0.32
2678	EN14362-1	79.53	-1.49
2706	In house	46.0	-4.23
2713	EN14362-1	92.65	-0.42
2717	EN14362-1	132.94	2.86
2718	EN14362-1	52.8361	-3.67
2723	EN14362-1	47.01	-4.14
2724		----	----
2727	EN14362-1	124.00	2.13
2734		----	----
2738	In house	108	0.83
2749		----	----
3100	EN14362-1	95.0	-0.23
3109	In house	92.6	-0.43
3110	EN14362-1	104.45	0.54
3116	EN14362-1	100.90	0.25
3117	EN14362-1	91.84	-0.49
3118	EN14362-1	88.80	-0.74
3122	EN14362-1	83.36	-1.18
3146	EN14362-1	93.2	-0.38
3150	EN14362-1	75.3	-1.84
3151	EN14362-1	100.83	0.24
3153	EN14362-1	95.9	-0.16
3154	EN14362-1	183.5	R(0.01) 6.99
3172	EN14362-1	100.22	0.20
3174	EN14362-1	63.06	-2.83
3176	EN14362-1	127.4	2.41
3185	EN14362-1	95.24	-0.21
3186	EN14362-1	114.4	1.35
3191	EN14362-1	98.4	0.05
3192	EN14362-1	122.9	2.04
3197	EN14362-1	100.8	0.24
3199	EN14362-1	94.1	-0.30

3204		83.40	-1.18
3210	In house	94.60	-0.26
3214	EN14362-1	103.68	0.48
3215	EN14362-1	99.28	0.12
3216	EN14362-1	78.83	-1.55
3218	EN14362-1	98	0.01
3220	EN14362-1	74.43	-1.91
3222	EN14362-1	110.1	1.00
3225	EN14362-1	<5	<-7.57 false negative test result?
3228	EN14362-1	99	0.10
3233	EN14362-1	101.57	0.31
3237	In house	101.10	0.27
3248	EN14362-1	122.14	1.98

normality suspect
n 152
outliers 4
mean (n) 97.826
st.dev. (n) 15.4950
R(calc.) 43.386
R(EN14362-1:12) 34.337



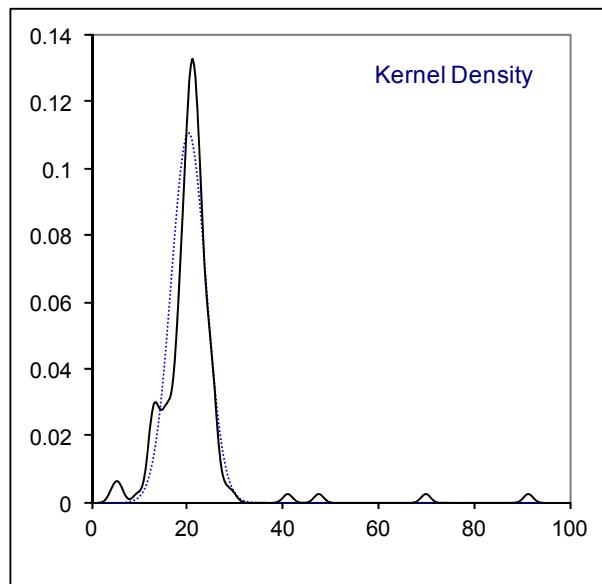
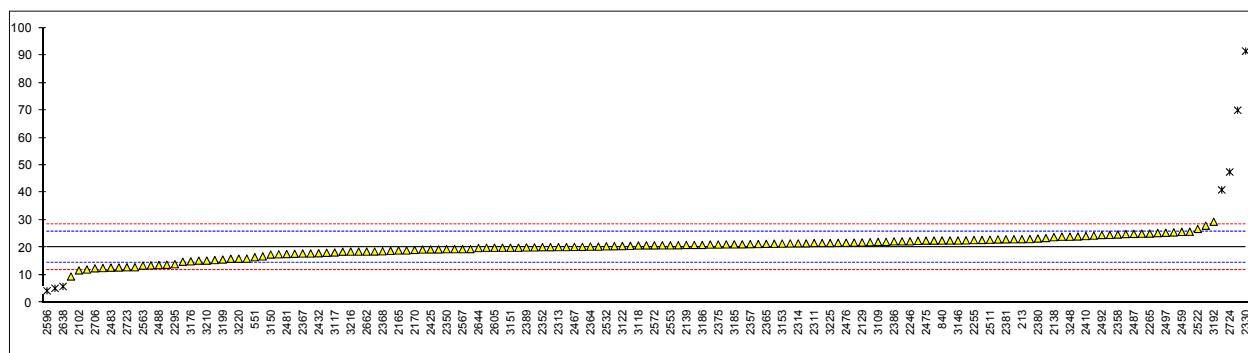
Determination of Benzidine (CASno.92-87-5) in sample #16516; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EN14362-1	5.24	R(0.05)	-5.36	
213	EN14362-1	23.1		1.03	
230	EN14362-1	25.32		1.82	
348	In house	22.99		0.99	
362		----		----	
551	EN14362-1	16.55860349		-1.31	
623	EN14362-1	23.08		1.02	
840	EN14362-1	22.6		0.85	
2102	In house	11.75		-3.03	
2108	EN14362-1	23.5		1.17	
2115	EN14362-1	41.02	R(0.01)	7.44	
2120	EN14362-1	15.3		-1.76	
2129	EN14362-1	21.9		0.60	
2131	EN14362-1	18.55		-0.60	
2132	EN14362-1	22.12		0.68	
2135	EN14362-1	14.9		-1.90	
2138	EN14362-1	23.84		1.30	
2139	EN14362-1	21		0.28	
2146	EN14362-1	24.0		1.35	
2165	EN14362-1	19		-0.44	
2166	EN14362-1	12.89		-2.62	
2170	EN14362-1	19.20		-0.37	
2181	EN14362-1	19.98		-0.09	
2184	EN14362-1	21		0.28	
2190	EN14362-1	ND	C	----	first reported 8
2201	EN14362-1	21.77		0.55	
2232	EN14362-1	18.866		-0.48	
2246	EN14362-1	22.32		0.75	
2247	EN14362-1	21.33		0.40	
2255	EN14362-1	22.78		0.92	
2256	EN14362-1	22.6		0.85	
2265	EN14362-1	25.136		1.76	
2284	EN14362-1	25.1		1.75	
2286	EN14362-1	20.86		0.23	
2287	EN14362-1	22.8		0.92	
2290	EN14362-1	21.23		0.36	
2291	EN14362-1	20.2		-0.01	
2293	EN14362-1	28	C	2.78	first reported 55.8
2295	EN14362-1	14		-2.23	
2296		----		----	
2300	EN14362-1	25.8		2.00	
2310	EN14362-1	22.66		0.87	
2311	EN14362-1	21.7		0.53	
2313	EN14362-1	20.21		0.00	
2314	EN14362-1	21.59		0.49	
2320	EN14362-1	18.6177		-0.57	
2330	EN14362-1	<5	C	<-5.45	possibly sample mix-up; reported this test result for sample #16515
2347		----		----	
2350	EN14362-1	19.45		-0.28	
2352	EN14362-1	20.2		-0.01	
2357	EN14362-1	21.3		0.39	
2358	EN14362-1	24.79		1.64	
2364	EN14362-1	20.4		0.06	
2365	EN14362-1	21.4		0.42	
2366	EN14362-1	21.56		0.48	
2367	EN14362-1	17.9	C	-0.83	first reported n.d.
2368	EN14362-1	18.7		-0.54	
2369	EN14362-1	22		0.64	
2370	EN14362-1	21.15		0.33	
2373	EN14362-1	17.8		-0.87	
2375	EN14362-1	21.15		0.33	
2379	EN14362-1	Not detected		----	
2380	EN14362-1	23.32		1.11	
2381	EN14362-1	23.05		1.01	
2386	EN14362-1	22.3		0.74	
2389	EN14362-1	20.10		-0.04	
2390	EN14362-1	25.558		1.91	
2403	EN14362-1	19.47		-0.27	
2410	EN14362-1	24.25		1.44	
2425	EN14362-1	19.37		-0.30	
2426		----		----	
2429	EN14362-1	21.7		0.53	
2432	EN14362-1	17.97		-0.81	

2442	In house	24.95	1.69
2453	EN14362-1	12.08	-2.91
2456	EN14362-1	12.6	-2.73
2459	EN14362-1	25.76	1.98
2467	EN14362-1	20.29	0.02
2472	EN14362-1	19.37	-0.30
2475	EN14362-1	22.58	0.84
2476	EN14362-1	21.8	0.57
2481	In house	17.7	-0.90
2482	EN14362-1	17.60	-0.94
2483		12.75	-2.67
2487	EN14362-1	25.03	1.72
2488	EN14362-1	13.69	-2.34
2489	EN14362-1	19.34	-0.31
2492	EN14362-1	24.6	1.57
2495	EN14362-1	24.4	1.50
2497	EN14362-1	25.43	1.86
2499	EN14362-1	12.809	C -2.65 first reported 12.185
2500	EN14362-1	16.03	-1.50
2511		22.9	0.96
2514	EN14362-1	20.68	0.16
2516	EN14362-1	18.6	-0.58
2522	EN14362-1	26.82	2.36
2528	EN14362-1	20.84	0.22
2532	EN14362-1	20.5	0.10
2534	EN14362-1	13.8	-2.30
2553	EN14362-1	20.86	0.23
2563	EN14362-1	13.5	-2.40
2565		22.5	0.82
2567	EN14362-1	19.47	-0.27
2569	EN14362-1	20.4	0.06
2572	EN14362-1	20.84	0.22
2590		----	----
2596	In house	4.30	R(0.05) -5.70
2605	EN14362-1	20.0	-0.08
2609	EN14362-1	22.3	0.74
2612	EN14362-1	< 10	<-3.66 false negative test result?
2614	EN14362-1	20.3	0.03
2629	EN14362-1	18.16	-0.74
2638	EN14362-1	5.889	C,R(0.05) -5.13 first reported 7.497
2643	EN14362-1	22.59	0.85
2644	EN14362-1	19.88	-0.12
2662	EN14362-1	18.6	-0.58
2665	EN14362-1	17.9	-0.83
2674	EN14362-1	20.9	0.24
2678	EN14362-1	21.83	0.58
2706	In house	12.5	-2.76
2713	EN14362-1	13.57	-2.38
2717		----	----
2718	EN14362-1	19.4846	-0.26
2723	EN14362-1	12.88	-2.63
2724	EN14362-1	47.54	C,R(0.01) 9.78 first reported 181.23
2727	EN14362-1	<10	<-3.66 false negative test result?
2734		----	----
2738	In house	16	-1.51
2749		----	----
3100	EN14362-1	20.0	-0.08
3109	In house	22.1	0.67
3110	EN14362-1	21.59	0.49
3116	EN14362-1	20.545	0.12
3117	EN14362-1	18.26	-0.70
3118	EN14362-1	20.81	0.21
3122	EN14362-1	20.61	0.14
3146	EN14362-1	22.6	0.85
3150	EN14362-1	17.5	-0.97
3151	EN14362-1	20.00	-0.08
3153	EN14362-1	21.5	0.46
3154	EN14362-1	70	R(0.01) 17.81
3172	EN14362-1	24.63	1.58
3174	EN14362-1	16.84	-1.21
3176	EN14362-1	15.05	-1.85
3185	EN14362-1	21.22	0.36
3186	EN14362-1	21.0	0.28
3191	EN14362-1	20.1	-0.04
3192	EN14362-1	29.45	3.30
3197	EN14362-1	21.2	0.35
3199	EN14362-1	15.7	-1.62

3204		9.53	-3.83
3210	In house	15.31	-1.76
3214	EN14362-1	24.09	1.38
3215	EN14362-1	23.14	1.04
3216	EN14362-1	18.59	-0.58
3218	EN14362-1	19	-0.44
3220	EN14362-1	16.01	-1.51
3222	EN14362-1	15.5	-1.69
3225	EN14362-1	21.7	0.53
3228	EN14362-1	20	-0.08
3233	EN14362-1	21.44	0.44
3237	In house	20.23	0.00
3248	EN14362-1	24.01	1.36

normality OK
 n 144
 outliers 6
 mean (n) 20.220
 st.dev. (n) 3.6066
 R(calc.) 10.099
 R(EN14362-1:12) 7.825



APPENDIX 2

Summary of other reported aromatic amines in sample #16515

lab	method
2596	112.28 mg/kg 3,3'-dimethylbenzidine, probably mixed-up with test result of 3,3'-dimethoxybenzidine
3204	2.78 mg/kg 4-aminodiphenyl & 4.01 mg/kg dimethylbenzidine & 5.78 mg/kg 4,4'-diaminodiphenylether & 4.2 mg/kg 4,4'-diaminodiphenylsulfide
3216	3.67 mg/kg 4-aminodiphenyl

Summary of other reported aromatic amines in sample #16516

lab	method
2190	1 mg/kg 4-aminodiphenyl
2265	1.687 mg/kg 4-aminodiphenyl
2453	1.36 mg/kg 4-aminodiphenyl
2456	6.7 mg/kg 4-aminodiphenyl
2534	0.9 mg/kg 4-aminodiphenyl
2723	5.3 mg/kg 4-aminodiphenyl
2724	4.32 mg/kg 4-aminodiphenyl
3146	2.18 mg/kg 4-aminodiphenyl
3204	3.00 mg/kg 4-aminodiphenyl
3216	9.76 mg/kg 4-aminodiphenyl

Summary of not detected aromatic amines in sample #16515

4AD = 4-Aminodiphenyl
4CoT = 4-Chloro-o-toluidine
2NA = 2-Naphthylamine
oAAT = o-Aminoazotoluene
ANT = 2-Amino-4-nitrotoluene
4CA = 4-Chloraniline
DAA = 2,4-Diaminoanisole
DADM = 4,4'-Diaminodiphenyl methane
DCB = 3,3'-Dichlorobenzidine
DMB = 3,3'-Dimethylbenzidine
DDDM = 3,3'-Dimethyl-4,4'-Diaminodiphenyl methane

Lab	4AD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMB	DDDM
110	----	----	----	----	----	----	----	----	----	----	----
213	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----
348	n.d.										
362	----	----	----	----	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----	----	----	----	----
623	n.d.										
840	n.d.										
2102	n.d.										
2108	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----
2120	----	----	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	----	----	----
2131	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2132	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2135	----	----	----	----	----	----	----	----	----	----	----
2138	----	----	----	----	----	----	----	----	----	----	----
2139	----	----	----	----	----	----	----	----	----	----	----
2146	----	----	----	----	----	----	----	----	----	----	----
2165	n.d.										
2166	----	----	----	----	----	----	----	----	----	----	----
2170	----	----	----	----	----	----	----	----	----	----	----
2181	----	----	----	----	----	----	----	----	----	----	----
2184	n.d.										
2190	n.d.										
2201	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2232	----	----	----	----	----	----	----	----	----	----	----
2246	----	----	----	----	----	----	----	----	----	----	----
2247	n.d.										
2255	n.d.										
2256	----	----	----	----	----	----	----	----	----	----	----
2265	----	----	----	----	----	----	----	----	----	----	----
2284	----	----	----	----	----	----	----	----	----	----	----
2286	n.d.										
2287	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10
2290	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2291	n.d.										
2293	----	----	----	----	----	----	----	----	----	----	----
2295	n.d.										
2296	----	----	----	----	----	----	----	----	----	----	----
2300	n.d.										
2310	n.d.										
2311	n.d.										
2313	n.d.										
2314	----	----	----	----	----	----	----	----	----	----	----
2320	n.d.										
2330	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2352	----	----	----	----	----	----	----	----	----	----	----
2357	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2358	n.d.										
2364	----	----	----	----	----	----	----	----	----	----	----
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2367	n.d.										
2368	n.d.										
2369	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2370	n.d.										
2373	----	----	----	----	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----	----	----	----	----

3154	----	----	----	----	----	----	----	----	----	----	----	----	----
3172	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
3174	n.d.												
3176	----	----	----	----	----	----	----	----	----	----	----	----	----
3185	n.d.												
3186	n.d.												
3191	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3192	----	----	----	----	----	----	----	----	----	----	----	----	----
3197	n.d.												
3199	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3204	2.78	<LOD	4.01	4.70									
3210	----	----	----	----	----	----	----	----	----	----	----	----	----
3214	n.d.												
3215	n.d.												
3216	3.67	n.d.											
3218	----	----	----	----	----	----	----	----	----	----	----	----	----
3220	----	----	----	----	----	----	----	----	----	----	----	----	----
3222	----	----	----	----	----	----	----	----	----	----	----	----	----
3225	<5	<5	<5	<5	<5	<5	<5	<5	<5	98.9	<5	<5	<5
3228	n.d.												
3233	----	----	----	----	----	----	----	----	----	----	----	----	----
3237	----	----	----	----	----	----	----	----	----	----	----	----	----
3248	----	----	----	----	----	----	----	----	----	----	----	----	----

Summary of not detected aromatic amines in sample #16515, continued

pC = p-Cresidine**DDM** = 4,4'-Diamino-3,3'-dichlorodiphenyl methane**DDE** = 4,4'-Diaminodiphenyl ether**DDS** = 4,4'-Diaminodiphenyl sulfide**oT** = o-Toluidine**24DAT** = 2,4-Diaminotoluene**TMA** = 2,4,5-Trimethylaniline**oA** = o-Anisidine**4AAT** = 4-Amino-azobenzene**24X** = 2,4-Xylidine**26X** = 2,6-Xylidine

Lab	pC	DDM	DDE	DDS	oT	24DAT	TMA	oA	4AAT	24X	26X
110	----	----	----	----	----	----	----	----	----	----	----
213	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----
348	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
362	----	----	----	----	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----	----	----	----	----
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2102	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2108	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----
2120	----	----	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	----	----	----
2131	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2132	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2135	----	----	----	----	----	----	----	----	----	----	----
2138	----	----	----	----	----	----	----	----	----	----	----
2139	----	----	----	----	----	----	----	----	----	----	----
2146	----	----	----	----	----	----	----	----	----	----	----
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2166	----	----	----	----	----	----	----	----	----	----	----
2170	----	----	----	----	----	----	----	----	----	----	----
2181	----	----	----	----	----	----	----	----	----	----	----
2184	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2190	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2201	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2232	----	----	----	----	----	----	----	----	----	----	----
2246	----	----	----	----	----	----	----	----	----	----	----
2247	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2255	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2256	----	----	----	----	----	----	----	----	----	----	----
2265	----	----	----	----	----	----	----	----	----	----	----
2284	----	----	----	----	----	----	----	----	----	----	----
2286	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2287	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10
2290	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2291	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2293	----	----	----	----	----	----	----	----	----	----	----
2295	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2296	----	----	----	----	----	----	----	----	----	----	----
2300	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2310	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2311	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2313	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2314	----	----	----	----	----	----	----	----	----	----	----
2320	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2330	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2352	----	----	----	----	----	----	----	----	----	----	----
2357	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2364	----	----	----	----	----	----	----	----	----	----	----
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2367	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2368	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2369	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2373	----	----	----	----	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----	----	----	----	----

3154	----	----	----	----	----	----	----	----	----	----	----	----
3172	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
3174	n.d.											
3176	----	----	----	----	----	----	----	----	----	----	----	----
3185	n.d.											
3186	n.d.											
3191	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3192	----	----	----	----	----	----	----	----	----	----	----	----
3197	n.d.											
3199	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3204	<LOD	<LOD	5.78	4.2	<LOD							
3210	----	----	----	----	----	----	----	----	----	----	----	----
3214	n.d.											
3215	n.d.											
3216	n.d.											
3218	----	----	----	----	----	----	----	----	----	----	----	----
3220	----	----	----	----	----	----	----	----	----	----	----	----
3222	----	----	----	----	----	----	----	----	----	----	----	----
3225	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3228	n.d.											
3233	----	----	----	----	----	----	----	----	----	----	----	----
3237	----	----	----	----	----	----	----	----	----	----	----	----
3248	----	----	----	----	----	----	----	----	----	----	----	----

Summary of not detected aromatic amines in sample #16516

4AD = 4-Aminodiphenyl
4CoT = 4-Chloro-o-toluidine
2NA = 2-Naphthylamine
oAAT = o-Aminoazotoluene
ANT = 2-Amino-4-nitrotoluene
4CA = 4-Chloraniline
DAA = 2,4-Diaminoanisole
DADM = 4,4'-Diaminodiphenyl methane
DCB = 3,3'-Dichlorobenzidine
DMoxB = 3,3'-Dimethoxybenzidine
DMB = 3,3'-Dimethylbenzidine

Lab	4AD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB
110	----	----	----	----	----	----	----	----	----	----	----
213	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----
348	n.d.										
362	----	----	----	----	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----	----	----	----	----
623	n.d.										
840	n.d.										
2102	n.d.										
2108	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----
2120	----	----	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	----	----	----
2131	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	----
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2135	----	----	----	----	----	----	----	----	----	----	----
2138	----	----	----	----	----	----	----	----	----	----	----
2139	----	----	----	----	----	----	----	----	----	----	----
2146	----	----	----	----	----	----	----	----	----	----	----
2165	n.d.										
2166	----	----	----	----	----	----	----	----	----	----	----
2170	----	----	----	----	----	----	----	----	----	----	----
2181	----	----	----	----	----	----	----	----	----	----	----
2184	n.d.										
2190	1	n.d.									
2201	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2232	----	----	----	----	----	----	----	----	----	----	----
2246	----	----	----	----	----	----	----	----	----	----	----
2247	n.d.										
2255	nd										
2256	----	----	----	----	----	----	----	----	----	----	----
2265	1.687	----	----	----	----	----	----	----	----	----	----
2284	----	----	----	----	----	----	----	----	----	----	----
2286	n.d.										
2287	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10
2290	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2291	n.d.										
2293	----	----	----	----	----	----	----	----	----	----	----
2295	n.d.										
2296	----	----	----	----	----	----	----	----	----	----	----
2300	n.d.										
2310	n.d.										
2311	n.d.										
2313	n.d.										
2314	----	----	----	----	----	----	----	----	----	----	----
2320	n.d.										
2330	<5	<5	<5	<5	<5	<5	<5	<5	<5	100.8	<5
2347	----	----	----	----	----	----	----	----	----	----	----
2350	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2352	----	----	----	----	----	----	----	----	----	----	----
2357	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2358	n.d..										
2364	----	----	----	----	----	----	----	----	----	----	----
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2367	n.d.										
2368	n.d.										
2369	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2370	n.d.										
2373	----	----	----	----	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----	----	----	----	----

2379	n.d.										
2380	----	----	----	----	----	----	----	----	----	----	----
2381	----	----	----	----	----	----	----	----	----	----	----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2389	n.d.										
2390	n.d.										
2403	----	----	----	----	----	----	----	----	----	----	----
2410	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2425	----	----	----	----	----	----	----	----	----	----	----
2429	n.d.										
2432	----	----	----	----	----	----	----	----	----	----	----
2442	----	----	----	----	----	----	----	----	----	----	----
2453	1.36	----	----	----	----	----	----	----	3.03	----	----
2456	6.7	----	----	----	----	----	----	----	----	----	----
2459	n.d.										
2467	----	----	----	----	----	----	----	----	----	----	----
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2475	----	----	----	----	----	----	----	----	----	----	----
2476	n.d.										
2481	<5	n.d.	<5	n.d.	n.d.						
2482	----	----	----	----	----	----	----	----	----	----	----
2483	n.d..										
2487	----	----	----	----	----	----	----	----	----	----	----
2488	----	----	----	----	----	----	----	----	----	----	----
2489	n.d.										
2492	----	----	----	----	----	----	----	----	----	----	----
2495	----	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2497	----	----	----	----	----	----	----	----	----	----	----
2499	----	----	----	----	----	----	----	----	----	----	----
2500	n.d..										
2511	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2514	----	----	----	----	----	----	----	----	----	----	----
2516	----	----	----	----	----	----	----	----	----	----	----
2522	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2528	----	----	----	----	----	----	----	----	----	----	----
2532	n.d.										
2534	0.9	n.d.									
2553	n.d.										
2563	----	----	----	----	----	----	----	----	----	----	----
2565	----	----	----	----	----	----	----	----	----	----	----
2567	n.d.										
2569	n.d.										
2572	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2590	----	----	----	----	----	----	----	----	----	----	----
2596	----	----	----	----	----	----	----	----	----	----	----
2605	n.d.										
2609	----	----	----	----	----	----	----	----	----	----	----
2612	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
2614	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2629	----	----	----	----	----	----	----	----	----	----	----
2638	----	----	----	----	----	----	----	----	----	----	----
2643	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2644	n.d.										
2662	n.d.										
2665	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2674	n.d.										
2678	n.d.										
2706	----	----	----	----	----	----	----	----	----	----	----
2713	----	----	----	----	----	----	----	----	----	----	----
2717	----	----	----	----	----	----	----	----	----	----	----
2718	----	----	----	----	----	----	----	----	----	----	----
2723	5.30	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2724	4.32	----	----	----	----	----	----	----	----	----	----
2727	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2734	----	----	----	----	----	----	----	----	----	----	----
2738	n.d.										
3100	n.d.										
3109	----	----	----	----	----	----	----	----	----	----	----
3110	n.d.										
3116	----	----	----	----	----	----	----	----	----	----	----
3117	----	----	----	----	----	----	----	----	----	----	----
3118	n.d.										
3122	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3146	2.18	----	----	----	----	----	----	----	----	----	----
3150	----	----	----	----	----	----	----	----	----	----	----
3151	----	----	----	----	----	----	----	----	----	----	----
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5

3154	----	----	----	----	----	----	----	----	----	----	----	----	----
3172	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
3174	n.d.												
3176	----	----	----	----	----	----	----	----	----	----	----	----	----
3185	n.d.												
3186	n.d.												
3191	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3192	----	----	----	----	----	----	----	----	----	----	----	----	----
3197	n.d.												
3199	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3204	3.00	<LOD											
3210	----	----	----	----	----	----	----	----	----	----	----	----	----
3214	n.d.												
3215	n.d.												
3216	9.76	n.d											
3218	----	----	----	----	----	----	----	----	----	----	----	----	----
3220	----	----	----	----	----	----	----	----	----	----	----	----	----
3222	----	----	----	----	----	----	----	----	----	----	----	----	----
3225	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3228	n.d.												
3233	----	----	----	----	----	----	----	----	----	----	----	----	----
3237	----	----	----	----	----	----	----	----	----	----	----	----	----
3248	----	----	----	----	----	----	----	----	----	----	----	----	----

Summary of not detected aromatic amines in sample #16516, continued

DDDM = 3,3'-Dimethyl-4,4'-Diaminodiphenyl methane**pC = p-Cresidine****DDM = 4,4'-Diamino-3,3'-dichlorodiphenyl methane****DDE = 4,4'-Diaminodiphenyl ether****DDS = 4,4'-Diaminodiphenyl sulfide****oT = o-Toluidine****24DAT = 2,4-Diaminotoluene****TMA = 2,4,5-Trimethylaniline****oA = o-Anisidine****4AAT = 4-Amino-azobenzene****24X = 2,4-Xylidine****26X = 2,6-Xylidine**

Lab	DDDM	pC	DDM	DDE	DDS	oT	24DAT	TMA	oA	4AAT	24X	26X
110	----	----	----	----	----	----	----	----	----	----	----	----
213	----	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----	----
348	n.d.											
362	----	----	----	----	----	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----	----	----	----	----	----
623	n.d.											
840	n.d.											
2102	n.d.											
2108	----	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----	----
2120	----	----	----	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	----	----	----	----
2131	----	----	----	----	----	----	----	----	----	----	----	----
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2135	----	----	----	----	----	----	----	----	----	----	----	----
2138	----	----	----	----	----	----	----	----	----	----	----	----
2139	----	----	----	----	----	----	----	----	----	----	----	----
2146	----	----	----	----	----	----	----	----	----	----	----	----
2165	n.d.											
2166	----	----	----	----	----	----	----	----	----	----	----	----
2170	----	----	----	----	----	----	----	----	----	----	----	----
2181	----	----	----	----	----	----	----	----	----	----	----	----
2184	n.d.											
2190	n.d.											
2201	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2232	----	----	----	----	----	----	----	----	----	----	----	----
2246	----	----	----	----	----	----	----	----	----	----	----	----
2247	n.d.											
2255	nd											
2256	----	----	----	----	----	----	----	----	----	----	----	----
2265	----	----	----	----	----	----	----	----	----	----	----	----
2284	----	----	----	----	----	----	----	----	----	----	----	----
2286	n.d.											
2287	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10	≤10
2290	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2291	n.d.											
2293	----	----	----	----	----	----	----	----	----	----	----	----
2295	n.d.											
2296	----	----	----	----	----	----	----	----	----	----	----	----
2300	n.d.											
2310	n.d.											
2311	n.d.											
2313	n.d.											
2314	----	----	----	----	----	----	----	----	----	----	----	----
2320	n.d.											
2330	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2347	----	----	----	----	----	----	----	----	----	----	----	----
2350	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2352	----	----	----	----	----	----	----	----	----	----	----	----
2357	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2358	n.d..											
2364	----	----	----	----	----	----	----	----	----	----	----	----
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2367	n.d.											
2368	n.d.											
2369	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2370	n.d.											
2373	----	----	----	----	----	----	----	----	----	----	----	----

3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	----	----	----	----	----	----	----	----	----	----	----	----	----
3172	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
3174	n.d.												
3176	----	----	----	----	----	----	----	----	----	----	----	----	----
3185	n.d.												
3186	n.d.												
3191	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3192	----	----	----	----	----	----	----	----	----	----	----	----	----
3197	n.d.												
3199	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3204	<LOD	<LOD	<LOD	5.11	<LOD								
3210	----	----	----	----	----	----	----	----	----	----	----	----	----
3214	n.d.												
3215	n.d.												
3216	n.d.	n.a	n.d	n.d	n.d.								
3218	----	----	----	----	----	----	----	----	----	----	----	----	----
3220	----	----	----	----	----	----	----	----	----	----	----	----	----
3222	----	----	----	----	----	----	----	----	----	----	----	----	----
3225	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3228	n.d.												
3233	----	----	----	----	----	----	----	----	----	----	----	----	----
3237	----	----	----	----	----	----	----	----	----	----	----	----	----
3248	----	----	----	----	----	----	----	----	----	----	----	----	----

APPENDIX 3**Analytical details**

Lab	was extraction with chlorobenzene CB performed?	How far was the extract evaporated?	evaporation temp in °C and time in min.	time from start of reduction to start of HPLC/GC analysis in hours	remarks
110	did not use CB	to a certain quantity	40°C, < 10 min	< 2	
213	CB extraction was done				
230	CB extraction was done	to complete dryness	25 ?	3	
348	did not use CB	to a certain quantity	n/a	1.5	
362	did not use CB	to a certain quantity	50°C, 10min	16	
551	CB extraction was done	to a certain quantity	--	--	
623	did not use CB	to a certain quantity	40°C, 15 min	One day	
840	did not use CB	to a certain quantity	50°C, 15 min	2	
2102	did not use CB	to a certain quantity	Not used	1.5	
2108	did not use CB	to a certain quantity	50°C, 30 min	3	
2115	did not use CB	to a certain quantity	50°C, 3 min	12	
2120	did not use CB	to a certain quantity	40°C, 20 min	1.5	
2129	did not use CB	to a certain quantity	40°C, 20 min	5-6	
2131	did not use CB	to a certain quantity	--	--	
2132	did not use CB	to a certain quantity	49°C, 5 min	1.2	
2135	did not use CB	to complete dryness	50°C, 7 min	8	TurboVap
2138	did not use CB		40-45°C, 10min	1 day	
2139	did not use CB	to a certain quantity	no	4	
2146	did not use CB	to a certain quantity	36-37°C, 15-20 min	5	
2165	did not use CB	to a certain quantity	< 50°C, 15 min.	<12	
2166	did not use CB	to complete dryness	60°C, 90 min	4	büchi syncore polyvap
2170	did not use CB	to a certain quantity	35°C, 10 min	5	
2181	did not use CB	to a certain quantity	35°C, 6 min	2.5	
2184	did not use CB	to a certain quantity	< 50°C, 15 min	<12	
2190	CB extraction was done	to a certain quantity	--		
2201	CB extraction was done	to complete dryness	40°C,6 min	Daily	
2232	did not use CB	to a certain quantity	40°C, 2 min	3	
2246	did not use CB	to a certain quantity	49°C, 5 min	1.2	
2247	did not use CB	to a certain quantity	40°C, 10 min	2.5	
2255	did not use CB	to a certain quantity	45°C, 7-8 min	3.5	
2256	did not use CB	to a certain quantity	40°C, 4 min	6	
2265	did not use CB	to a certain quantity	50°C,15min	5	
2284	did not use CB	to a certain quantity	45°C, 5 min	2	
2286	CB extraction was done	to complete dryness	40°C, 20 min	2-4	
2287	did not use CB	to a certain quantity	40°C, 10 min	1.5	
2290	did not use CB	to a certain quantity	60°C, 60 min	7	
2291	CB extraction was done	to a certain quantity	42°C, 15 min	3	
2293	CB extraction was done	to complete dryness	60°C, 20 min	6	
2295	did not use CB	to a certain quantity	70°C, 60 min	3	
2296	did not use CB	to a certain quantity	46°C, 60 min	1 day	
2300					
2310	did not use CB	to a certain quantity	45°C, < 5 min	<2	
2311	did not use CB	to a certain quantity	30°C, 5 min	2	
2313	did not use CB	to a certain quantity	40°C, 10 min	2	
2314	did not use CB	to a certain quantity	45°C, < 5 min	<2	
2320	did not use CB	to a certain quantity	n/a	1.3	
2330	CB extraction was done	to complete dryness	20 min	1.5	
2347	did not use CB	to a certain quantity	40°C, 20 min	3 days	
2350	did not use CB	to a certain quantity	NONE	1 day	
2352	did not use CB	to a certain quantity	50°C,3-5min	<1.3	
2357	did not use CB	to a certain quantity	35°C, 10 min	1.5	
2358	did not use CB	to a certain quantity	23°C,15min	0.75	
2364	did not use CB	to a certain quantity	45°C,4min	2.5	
2365	did not use CB	to a certain quantity	40°C, 20 min	0.5	

2366	did not use CB	to a certain quantity	45°C, 15 min	<8	
2367	did not use CB	to a certain quantity	40°C, 5 min	1.8	
2368	did not use CB	to a certain quantity	45°C, 5 min	1.7	
2369	did not use CB	to a certain quantity	40°C, 15-20 min	2.5	
2370	did not use CB	to a certain quantity	40°C, 5 min	3	
2373	did not use CB	to a certain quantity	45°C, 5 min	1.8	
2375	other solvent was used	to a certain quantity	-	1.5	
2379	did not use CB	to a certain quantity	40°C, 5 min	3 days	
2380	other solvent was used	to a certain quantity	< 60 °C, 3-5 min	4	
2381	CB extraction was done	to a certain quantity	< 60 °C, 3-5 min	4	
2386	did not use CB	to a certain quantity	30°C, 15-20 min	4-6	
2389	did not use CB	to a certain quantity	n/a	3	
2390	did not use CB	to a certain quantity		1	Screening method is used
2403					
2410	did not use CB	to a certain quantity	<50°C, 5 min	<24	
2425	other solvent was used	to a certain quantity	50°C	2	
2426					
2429	did not use CB	to a certain quantity	40°C, 10 min	3	
2432	did not use CB	to a certain quantity		1.5	
2442	CB extraction was done	to a certain quantity	45°C, 5 min	2	Screening method is used
2453	did not use CB	to a certain quantity	55°C	1	
2456	did not use CB	to a certain quantity	40°C	2	
2459	did not use CB	to a certain quantity	45°C, 5 min	5	
2467	did not use CB	to a certain quantity	50°C till ready	1.5	
2472	did not use CB	to a certain quantity	38°C, 10 min	2	
2475	did not use CB	to a certain quantity	45°C, 5-10 min	<2	
2476	did not use CB	to a certain quantity	40°C, 10 min	3	
2481	CB extraction was done	to a certain quantity	34°C - 8 min	7	
2482	did not use CB	to a certain quantity	< 50°C, 20 min	6	
2483	did not use CB	to a certain quantity	37°C, 15 min	2 days.	
2487	did not use CB	to a certain quantity	40°C, 10 min	2	
2488	did not use CB	to a certain quantity	40°C	4	
2489	did not use CB	to a certain quantity	45°C, 3-4 min	2	
2492	other solvent was used	to a certain quantity	35 °C, 30 min	<1	
2495	did not use CB	to a certain quantity	40°C, 7 min	4	
2497	did not use CB	to a certain quantity	50°C, 15 min	8	
2499	did not use CB	to a certain quantity	40 °C, 5 min	8	
2500	CB extraction was done	to a certain quantity	40 °C,10 min	4	
2511	did not use CB	to a certain quantity	35°C, 20 min	4-5	
2514	did not use CB	to a certain quantity	50°C, 5-6 min	0.5	
2516	did not use CB	to a certain quantity	35°C, 7 min	4	
2522	did not use CB	to a certain quantity	40°C, 10 min	2.5	
2528	did not use CB	to a certain quantity	40°C	6	
2532	did not use CB	to a certain quantity	40°C, 10 min	3	
2534	did not use CB	to complete dryness	30°C, 4 min.	3	
2553	did not use CB	to a certain quantity	40°C, 2-3 min	1.5	
2563	did not use CB	to a certain quantity	-	6	
2565	did not use CB	to a certain quantity	35°C, 15 min	about half a day	
2567	did not use CB	to a certain quantity	48°C, 3-4 min	-	
2569	did not use CB	to a certain quantity	40°C, 10 min	3	
2572	did not use CB	to complete dryness	60°C, 1 hour	7	
2590	did not use CB	to a certain quantity	40°C	2.4	
2596	other solvent was used	to a certain quantity	35°C, 5 min	2.5	
2605	did not use CB	to a certain quantity	35°C, 8 min	3	
2609	did not use CB	to a certain quantity	40°C, 5 min	2	
2612	CB extraction was done	to complete dryness	70°C, 30 min	4	
2614	did not use CB	to a certain quantity	40°C, <5 min	<3	
2629	did not use CB	to a certain quantity	40°C, 10 min	2	
2638	did not use CB	to a certain quantity	46°C 7 min	1.5	
2643	did not use CB	to a certain quantity	45°C,15 min	3	
2644	did not use CB	to a certain quantity	45°C, 6 min	1..5	
2662	did not use CB	to a certain quantity	35°C, 10 min	2	

2665	did not use CB	to a certain quantity	20-25°C, 10 min	4	
2674	did not use CB	to a certain quantity	45°C, 10 min	<12	
2678	did not use CB	to a certain quantity	45°C, 10 min	6	
2706	did not use CB	to a certain quantity		4-6	no MTBE used
2713	did not use CB	to a certain quantity	40°C, 5-7 min	5	
2717	CB extraction was done	to complete dryness	40°C, 10 min	2.5	
2718	did not use CB	to a certain quantity	skipped	2	
2723					
2724	did not use CB	to complete dryness	40°C, 10 min	3	
2727	other solvent was used	to a certain quantity	50°C, 3 min	6	
2734					
2738	other solvent was used	to a certain quantity	45°C, 5-7 min	4.5	
2749					
3100	did not use CB	to a certain quantity	35°C, 5-6 min	1.5-2	with storage at -20 °C
3109	did not use CB	to a certain quantity	40°C, 4-6 min	1-2 days	
3110	did not use CB	to a certain quantity	50°C, 10 min	2	
3116	did not use CB	to a certain quantity	40°C, 15 mins	<4	
3117	did not use CB	to a certain quantity	34°C, 4-5 min	1-1.5	
3118	did not use CB	to a certain quantity	35° C	2	
3122	did not use CB	to a certain quantity	45°C, 5 min	4	
3146					
3150	did not use CB	to a certain quantity	45°C, 7 min	1	
3151	did not use CB	to a certain quantity	40°C, 15 min	1.5	
3153	did not use CB	to a certain quantity	35 °C, 7 min	7	
3154	did not use CB	to a certain quantity	50°C, 5 min	2	
3172	did not use CB	to a certain quantity	45°C, 40 min	2	
3174	did not use CB	to a certain quantity	40°C, 20 min	12	
3176	did not use CB	to a certain quantity	50°C, 15 min	1.5	
3185	did not use CB	to a certain quantity	35°C, 3-5 min	<1	
3186	did not use CB	to complete dryness	40°C, 15 min	4-5	
3191	did not use CB	to a certain quantity	45°C, 5 min	In 24 hrs.	
3192	did not use CB	to complete dryness	50°C	5	
3197	did not use CB	to a certain quantity	40°C, 10 min	3	
3199	did not use CB	to a certain quantity	40°C, 10 min	1	
3204	CB extraction was done	to a certain quantity	50°C	three days	
3210	did not use CB	to a certain quantity	40°C, 10 min	2	
3214	did not use CB	to a certain quantity	35°C, 10 min	8	
3215	did not use CB	to a certain quantity	40°C, 20 min	1	
3216	did not use CB	to a certain quantity	40°C, 10 min	7 days	
3218	did not use CB	to a certain quantity	40°C, 5 min	3	
3220	did not use CB	to a certain quantity	30°C , 10 min	3	
3222	did not use CB	to complete dryness	48°C, 10 min	3	
3225	did not use CB	to a certain quantity	-	1 day	
3228	did not use CB	to a certain quantity		<12	
3233	did not use CB	to a certain quantity	45°C, 10 min	2.5	
3237	did not use CB	to a certain quantity	45°C, 8 min.	2	
3248	CB extraction was done	to a certain quantity	40°C, 10 min	3	

APPENDIX 4**Number of participants per country**

1 lab in AUSTRIA
6 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
2 labs in CAMBODIA
1 lab in CROATIA
1 lab in EGYPT
1 lab in FINLAND
5 labs in FRANCE
16 labs in GERMANY
1 lab in GUATEMALA
10 labs in HONG KONG
12 labs in INDIA
2 labs in INDONESIA
11 labs in ITALY
4 labs in JAPAN
5 labs in KOREA
1 lab in MAURITIUS
1 lab in MOROCCO
34 labs in P.R. of CHINA
5 labs in PAKISTAN
1 lab in POLAND
3 labs in PORTUGAL
1 lab in ROMANIA
2 labs in SINGAPORE
3 labs in SPAIN
2 labs in SRI LANKA
6 labs in SWITZERLAND
3 labs in TAIWAN R.O.C.
2 labs in THAILAND
1 lab in THE NETHERLANDS
2 labs in TUNISIA
7 labs in TURKEY
2 labs in U.S.A.
1 lab in UNITED KINGDOM
6 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:

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