Results of Proficiency Test
AZO Dyes in Leather
March 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 1997 the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the analysis of banned aromatic amines derived from AZO dyes in Leather. During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency test for the analysis of banned aromatic amines derived from AZO dyes in Leather.

In this interlaboratory study 110 laboratories in 28 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the AZO dyes in Leather proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different leather samples of 3 grams each labelled #21520 and #21521.
The participants were requested 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/IEC17043:2010 (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 of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website 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

A batch of dark blue/black leather was selected which was made positive on 4-Aminoazobenzene by a third party. This batch was cut into small blocks. After homogenization the batch was divided over 160 subsamples in small bags of approximately 3 grams each and labelled #21520.
The homogeneity of the subsamples was checked by determination of 4-Aminoazobenzene using test method EN14362 on ten stratified randomly selected subsamples.

	4-Aminoazobenzene in mg/kg
sample #21520-1	218.9
sample #21520-2	228.3
sample #21520-3	220.3
sample #21520-4	214.3
sample #21520-5	212.1
sample #21520-6	209.6
sample #21520-7	229.6
sample #21520-8	231.5
sample #21520-9	217.7
sample #21520-10	226.9

Table 1: homogeneity test results of subsamples #21520

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2, in the next table.

	4-Aminoazobenzene in mg/kg
r (observed)	21.7
reference test method	ISO17234-2:11
0.3 x R (reference test method)	26.1

Table 2: evaluation of the repeatability of subsamples #21520

The calculated repeatability was in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

The second batch a dark brown leather was selected which was made positive for Benzidine. This batch was cut into small blocks. After homogenization the batch was divided over 160 subsamples in small bags of approximately 3 grams each and labelled #21521.

The batch was used in a previous proficiency test on AZO dyes in leather (as sample #18525 in iis18A04). Therefore, homogeneity of the subsamples was assumed.

2.5 ANALYZES

The participants were asked to determine on sample #21520 only 4-Aminoazobenzene (CASno 60-09-3).
On sample #21521 the participants were asked to determine the following aromatic amines:
4-Aminodiphenyl (CASno. 92-67-1)
Benzidine (CASno. 92-87-5)
4-Chloro-o-toluidine (CASno. 95-69-2)
2-Naphtylamine (CASno. 91-59-8)
2-Amino-4-nitrotoluene (CASno. 99-55-8)
4-Chloraniline (CASno. 106-47-8)
2,4-Diaminoanisol (CASno. 615-05-4)
4,4'-Diaminodiphenylmethane (CASno. 101-77-9)
3,3'-Dichlorobenzidine (CASno. 91-94-1)
3,3'-Dimethoxybenzidine (CASno. 119-90-4)
3,3'-Dimethylbenzidine (Casno. 119-93-7)
3,3'-Dimethyl-4,4'-Diaminodiphenylmethane (CASno. 838-88-0)
p-Cresidine (CASno. 120-71-8)
4,4'-Diamino-3,3'-dichlorodiphenylmethane (CASno. 101-14-4)
4,4'-Diaminodiphenylether (CASno. 101-80-4)
4,4'-Diaminodiphenylsulfide (CASno. 139-65-1)
2,4-Diaminotoluene (CASno. 95-80-7)
2,4,5-Trimethylaniline (CASno. 137-17-7)
o-Anisidine (CASno. 90-04-0)
2,4-Xylidine (CASno. 95-68-1)
2,5-Xylidine (CASno. 95-78-3)
2,6-Xylidine (CASno. 87-62-7)
Total of Xylidines
o-Aminoazotoluene (CASno. 97-56-3)
o-Toluidine (CASno. 95-53-4)
Sum of o-Aminoazotoluene and o-Toluidine

It was requested not to use less than 0.5 grams per determination and not to age and/or dry the samples, nor to determine volatile matter to ensure homogeneity. It was also requested to report if the laboratory was accredited to determine the reported components and to report some analytical details.
It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.
To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test 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 reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test 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

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were 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. If a dataset does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's 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. In this PT, the criterion of ISO13528, paragraph 9.2.1 was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize 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 test 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 reference test method. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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 result is fit-for-use.

The z-scores were calculated in according to:

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

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

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

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

4 EVALUATION

In this interlaboratory study some problems were encountered with the dispatch of the samples due to COVID-19. Therefore, the reporting time on the data entry portal was extended with one week. Ten participants reported the test results after the extended final reporting date and two participants did not report any test results at all. Not all laboratories were able to report al components.
Finally, 108 participants reported 212 numerical test results. Observed were 6 outlying test results, which is 2.8% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

All original data sets given in appendix 1 proved to have a normal Gaussion distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and per component. The test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the reported test results in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.
For the determination of aromatic amines precision data are available and listed in ISO17234-1. For the component Benzidine, which is present in sample #21521, a precision statement is mentioned. For the determination of 4-Aminoazobenzene the ISO17234-2 method is considered to be the official method.

Sample #21520

4-Aminoazobenzene (CAS no. 60-09-3): The determination was problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in not in agreement with the requirements of ISO17234-2:11.

Sample #21521

Benzidine (CAS no. 92-87-5): The determination of was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO17234-1:20.

The majority of the participants agreed on a concentration near or below the limit of detection for all other aromatic amines mentioned in paragraph 2.5. Therefore, no z-scores were calculated for these aromatic amines. The reported test results of these components are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(lit)
4-Aminoazobenzene	mg/kg	101	271.2	144.1	106.7

Table 3: reproducibility on sample #21520

Component	unit	n	average	2.8 * sd	R(lit)
Benzidine	mg/kg	105	64.1	37.2	37.2

Table 4: reproducibility on sample #21521

Without further statistical calculations, it can be concluded that the group of participating laboratories has problems with the analysis of 4-Aminoazobenzene and has no problems with the analysis of Benzidine in leather at the observed concentration levels.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2021 WITH PREVIOUS PTS

	March 2021	March 2020	March 2019	March 2018	February 2016
Number of reporting laboratories	108	90	117	117	113
Number of test results	212	166	117	116	205
Number of statistical outliers	6	1	3	4	3
Percentage of statistical outliers	2.8%	0.6%	2.6%	3.4%	1.4%

Table 5: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determination of the proficiency test was compared, expressed as relative standard deviation (RSD) of the PTs in the next table.

Component	March 2020	March 2019	March 2018	February 2017	2016-2005	Target
4-Aminodiphenyl	n.e.	n.e.	n.e.	n.e.	25 - 45%	15 - 33%
Benzidine	21%	n.e.	20%	20%	20 - 66%	15 - 25%
3,3'-Dimethoxybenzidine	n.e.	23%	n.e.	n.e.	n.e.	15%
3,3'-Dimethylbenzidine	n.e.	n.e.	n.e.	n.e.	24 - 55%	17 - 24%
o-Toluidine	n.e.	n.e.	n.e.	n.e.	37 - 63%	30 - 37%
o-Ansidine	n.e.	n.e.	n.e.	n.e.	n.e.	15 – 37%
2,4-Xylidine	n.e.	n.e.	n.e.	n.e.	16 - 36%	15 - 37%
4-Aminoazobenzene	19%	n.e.	n.e.	n.e.	n.e.	14%

Table 6: development of uncertainties over the years

Components not listed in table 6 have not been tested in an iis AZO dyes in Leather PT.

Sample #21521 was used before in Proficiency Test iis18A04 as sample #18525. It is observed that the current PT findings of the leather subsamples containing Benzidine show a significant lower average concentration level.

	unit	#21521			#18525		
		n	average	R(calc)	n	average	R(calc)
Benzidine	mg/kg	105	64.1	37.2	112	77.8	44.5

Table 7: comparison of sample #21521 with sample #18525

4.4 EVALUATION ANALYTICAL DETAILS

For this PT also some analytical details were requested and listed in appendix 3.
Based on the answers given by the participants the following can be summarized:

- 75% of the participants mentioned that they are accredited for the determination of aromatic amine components.

#21520

- 40% of the participants used around 0.5 grams sample intake, 40% used around 1 gram and 7% used more than 1 grams as sample intake.
- 42% of the participants further cut the samples prior to analysis, 57% used the samples as received and 1% grinded the sample.

#21521

- 42% of the participants used around 0.5 grams sample intake, 51% used around 1 gram and 7% used more than 1 grams as sample intake.
- 72% of the participants further cut the samples prior to analysis, 27% used the samples as received and 1% used another sample preparation method.

The effect of sample intake is not significant in sample #21520.

5 DISCUSSION

Allmost all reporting participants were able to detect 4-Aminoazobenzene in sample #21520 and Benzidine in sample #21521. No other aromatic amines were detected.

When the results of this interlaboratory study were compared to the LEATHER STANDARD by OEKO-TEX and with the similar bluesign® systems substances list or BSSL (Table 8), it was noticed that not all participants would make an identical decision about the acceptability of the samples for the determined components.
Two of the reporting laboratories would accept the sample #21520 for all categories while all other laboratories would have rejected the sample #21520 for all categories.
Two of the reporting laboratories would accept the sample #21521 for all categories while all other laboratories would have rejected the sample #21521 for all categories.

Ecolabel	baby clothes	in direct skin contact	no direct skin contact
bluesign® BSSL	<20 mg/kg	<20 mg/kg	<20 mg/kg
Leather by OEKO-TEX	<20 mg/kg	<20 mg/kg	<20 mg/kg

Table 8: bluesign® BSSL and LEATHER STANDARD by OEKO-TEX

6 CONCLUSION

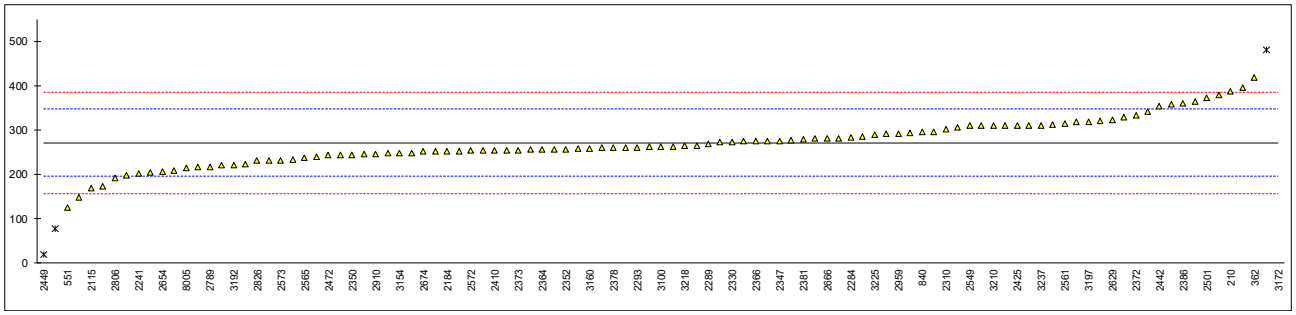
Although, it can be concluded that the majority participants have no problem with the determination of 4-Aminoazobenzene and Benzidine in the samples of this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.
Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

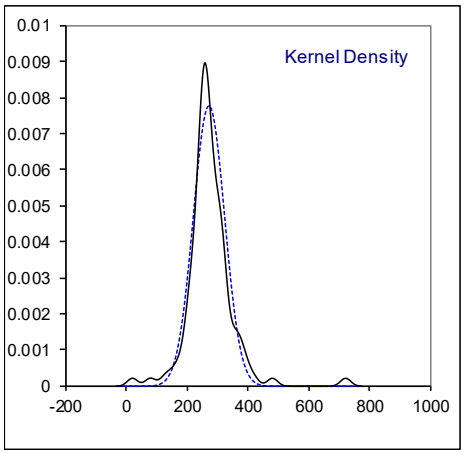
APPENDIX 1

Determination of 4-Aminoazobenzene (CAS no. 60-09-3) in sample #21520; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17234-2	386.8		3.03	
230	INH-201	309.94415		1.02	
339		-----		-----	
348	In house	341.9		1.86	
362	ISO17234-2	418		3.85	
551	ISO17234-2	125.698		-3.82	
623	ISO17234-2	233.933		-0.98	
840	ISO17234-2	295		0.63	
841	ISO17234-2	252.44		-0.49	
2115		168.6		-2.69	
2129	ISO17234-2	363.5		2.42	
2132	ISO17234-2	207.4		-1.67	
2135	ISO17234-2	378.2		2.81	
2165	ISO17234-2	247.3		-0.63	
2184	ISO17234-2	252		-0.50	
2201	ISO17234-2	257.47		-0.36	
2238	ISO17234-2	223.0		-1.26	
2241	ISO17234-2	202.833		-1.79	
2247	ISO17234-2	309.85		1.01	
2250	ISO17234-2	240		-0.82	
2256		286		0.39	
2265		-----		-----	
2284	ISO17234-2	283.64		0.33	
2289	ISO17234-2	268		-0.08	
2290	ISO17234-2	255.8		-0.40	
2293	ISO17234-2	260.94		-0.27	
2310	ISO17234-2	301.2		0.79	
2311	ISO17234-2	264.61		-0.17	
2330	ISO17234-2	273.462		0.06	
2347	GB/T33392	275		0.10	
2350	ISO17234-2	244.326		-0.70	
2352	ISO17234-2	256		-0.40	
2357	ISO17234-2	274.3		0.08	
2358	ISO17234-2	253.88		-0.45	
2364	ISO17234-2	255.8		-0.40	
2365	ISO17234-2	274.4		0.08	
2366	ISO17234-2	274.3		0.08	
2367	ISO17234-2	255.47		-0.41	
2370	ISO17234-2	295.3		0.63	
2372	ISO17234-2	333.6		1.64	
2373	ISO17234-2	254.7		-0.43	
2375	ISO17234-2	292.7		0.56	
2378	GB/T33392	260		-0.29	
2379	ISO17234-2	251.8748		-0.51	
2380	ISO17234-2	277.00		0.15	
2381	ISO17234-2	279.16		0.21	
2386	ISO17234-2	360.6		2.35	
2390	ISO17234-2	281.90		0.28	
2410	ISO17234-2	254		-0.45	
2415	ISO17234-2	358.50		2.29	
2425	In house	310.0		1.02	
2429	ISO17234-2	260.4		-0.28	
2442	ISO17234-2	352.82		2.14	
2449	ISO17234-2	19.63	R(0.01)	-6.60	
2452	ISO17234-2	243.774		-0.72	
2453	ISO17234-2	220.410		-1.33	
2455		-----		-----	
2472	ISO17234-2	242.50		-0.75	
2476	ISO17234-2	312.7		1.09	
2501	ISO17234-2	372.69		2.66	
2511	ISO17234-2	480	R(0.05)	5.48	
2532	ISO17234-2	318		1.23	
2538	ISO17234-2	78.32	R(0.05)	-5.06	
2549	ISO17234-2	309.2		1.00	
2561	ISO17234-2	313.46		1.11	
2565	ISO17234-2	236.590		-0.91	
2572	ISO17234-2	253.3		-0.47	
2573	ISO17234-2	231.3		-1.05	
2582	ISO17234-2	173.71		-2.56	
2590	ISO17234-2	280.065		0.23	
2629	ISO17234-2	322.7		1.35	
2643	ISO17234-2	147.174		-3.25	
2654	ISO17234-2	206.800		-1.69	
2666	ISO17234-2	280.50		0.24	
2668	ISO17234-2	306.65		0.93	

lab	method	value	mark	z(targ)	remarks
2674	ISO17234-2	251		-0.53	
2695	ISO17234-2	not detected		-----	Possibly a false negative test result?
2703	ISO17234-2	244.719		-0.69	
2711	ISO17234-2	272.69		0.04	
2737	ISO17234-2	310.65		1.04	
2773	ISO17234-2	320		1.28	
2789	ISO17234-2	216.460		-1.44	
2806	ISO17234-2	192.5		-2.06	
2823		-----		-----	
2826	ISO17234-2	230.67		-1.06	
2829	ISO17234-2	394.4		3.23	
2870	ISO17234-2	292		0.55	
2910	ISO17234-2	245		-0.69	
2959	ISO17234-2	292		0.55	
3100	ISO17234-2	262.159		-0.24	
3116	ISO17234-2	215.60		-1.46	
3153	ISO17234-2	231.2		-1.05	
3154	ISO17234-2	247.88		-0.61	
3160	ISO17234-2	258.98		-0.32	
3172	ISO17234-2	721.32	C,R(0.01)	11.81	First reported 492.9215
3176	ISO17234-2	259.59		-0.30	
3185	ISO17234-2	262.200		-0.24	
3192	ISO17234-2	221.0477		-1.31	
3197	ISO17234-2	318.1		1.23	
3210	In house	309.93		1.02	
3218	ISO17234-2	263.294		-0.21	
3220	ISO17234-2	198.343		-1.91	
3225	ISO17234-2	290.13		0.50	
3228	ISO17234-2	254		-0.45	
3237	ISO17234-2	311		1.04	
3246	ISO17234-2	248.47		-0.60	
3248	ISO17234-2	205		-1.74	
3250	ISO17234-2	328.25		1.50	
8005	ISO17234-2	214.36		-1.49	
8030	ISO17234-2	261.7463		-0.25	
	normality	OK			
	n	101			
	outliers	4			
	mean (n)	271.1727			
	st.dev. (n)	51.48082	RSD = 19%		
	R(calc.)	144.1463			
	st.dev.(ISO17234-2:11 (HPLC))	38.12304			
	R(ISO17234-2:11 (HPLC))	106.7445			Compare R(Horwitz) = 52.2738

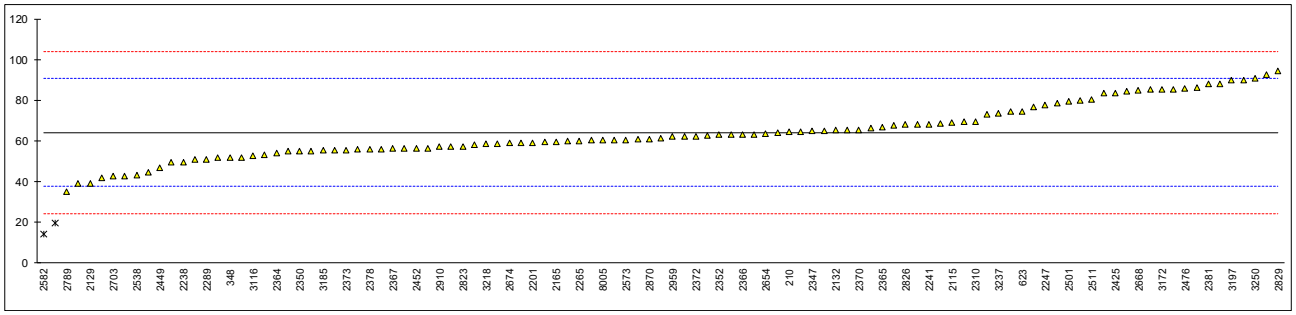


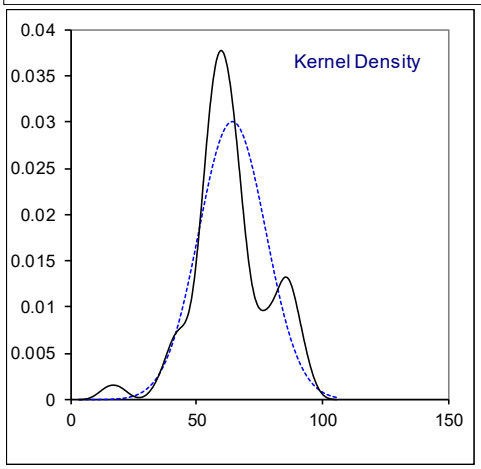


Determination of Benzidine (CAS no. 92-87-5) in sample #21521; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17234-1	64.4		0.02	
230	INH-201	51.788891		-0.93	
339		-----		-----	
348	In house	51.8		-0.93	
362		-----		-----	
551	ISO17234-1	41.825		-1.68	
623	ISO17234-1	74.498		0.78	
840	ISO17234-1	61		-0.23	
841	ISO17234-1	61.26		-0.22	
2115	ISO17234-1	68.8		0.35	
2129	ISO17234-1	39.0		-1.89	
2132	ISO17234-1	65.2		0.08	
2135	ISO17234-1	50.78		-1.00	
2165	ISO17234-1	59.7		-0.33	
2184	ISO17234-1	56		-0.61	
2201	ISO17234-1	59.09		-0.38	
2238	ISO17234-1	49.6		-1.09	
2241	ISO17234-1	68.282		0.31	
2247	ISO17234-1	77.83		1.03	
2250	ISO17234-1	65		0.07	
2256		53		-0.84	
2265	ISO17234-1	60.1		-0.30	
2284	ISO17234-1	55.26		-0.67	
2289	ISO17234-1	51		-0.99	
2290	ISO17234-1	62.1		-0.15	
2293	ISO17234-1	56.228		-0.59	
2310	ISO17234-1	69.5		0.40	
2311	ISO17234-1	62.748		-0.10	
2330	ISO17234-1	66.433		0.17	
2347	GB/T33392	65		0.07	
2350	ISO17234-1	55.028		-0.68	
2352	ISO17234-1	63		-0.08	
2357	ISO17234-1	63.2		-0.07	
2358	ISO17234-1	64.66		0.04	
2364	ISO17234-1	54.0		-0.76	
2365	ISO17234-1	66.6		0.19	
2366	ISO17234-1	63.1		-0.08	
2367	ISO17234-1	56.22		-0.59	
2370	ISO17234-1	65.3		0.09	
2372	ISO17234-1	62.4		-0.13	
2373	ISO17234-1	55.6		-0.64	
2375	ISO17234-1	78.4		1.07	
2378	ISO17234-1	56		-0.61	
2379	ISO17234-1	87.9910		1.79	
2380	ISO17234-1	90.00		1.95	
2381	ISO17234-1	87.87		1.79	
2386	ISO17234-1	83.3		1.44	
2390	ISO17234-1	54.78		-0.70	
2410	ISO17234-1	63		-0.08	
2415	ISO17234-1	65.30		0.09	
2425	In house	83.5		1.46	
2429	ISO17234-1	56.3		-0.59	
2442	ISO17234-1	84.63		1.54	
2449	ISO17234-1	46.98		-1.29	
2452	ISO17234-1	56.274		-0.59	
2453	ISO17234-1	51.970		-0.91	
2455		-----		-----	
2472	ISO17234-1	67.50		0.25	
2476	ISO17234-1	85.8		1.63	
2501	ISO17234-1	79.50		1.16	
2511	ISO17234-1	80.311		1.22	
2532	ISO17234-1	92.5		2.13	
2538	ISO17234-1	43.15		-1.58	
2549	ISO17234-1	85.2		1.58	
2561	ISO17234-1	57.055		-0.53	
2565	ISO17234-1	55.632		-0.64	
2572	ISO17234-1	60.4		-0.28	
2573	ISO17234-1	60.4		-0.28	
2582	ISO17234-1	14.27	DG(0.05)	-3.75	
2590	ISO17234-1	74.451		0.78	
2629	ISO17234-1	76.8		0.95	
2643	ISO17234-1	42.677		-1.61	
2654	ISO17234-1	63.360		-0.06	
2666	ISO17234-1	59.91		-0.32	
2668	ISO17234-1	85.06		1.57	

lab	method	value	mark	z(targ)	remarks
2674	ISO17234-1	59		-0.38	
2695	ISO17234-1	19.5	DG(0.05)	-3.35	
2703	INH-034	42.62		-1.62	
2711	ISO17234-1	59.00		-0.38	
2737	ISO17234-1	68.235		0.31	
2773	ISO17234-1	85.5		1.61	
2789	ISO17234-1	34.97	C	-2.19	First reported 22.455
2806	ISO17234-1	44.3		-1.49	
2823	ISO17234-1	57.299		-0.51	
2826	ISO17234-1	67.93		0.29	
2829	ISO17234-1	94.48		2.28	
2870	ISO17234-1	61		-0.23	
2910	ISO17234-1	57		-0.54	
2959		62		-0.16	
3100	ISO17234-1	55.078		-0.68	
3116	ISO17234-1	52.731		-0.86	
3153	ISO17234-1	59.4		-0.35	
3154	ISO17234-1	68.58		0.34	
3160	ISO17234-1	58.62		-0.41	
3172	ISO17234-1	85.4458		1.60	
3176	ISO17234-1	86.03		1.65	
3185	ISO17234-1	55.236		-0.67	
3192	ISO17234-1	73.0415		0.67	
3197	ISO17234-1	89.8		1.93	
3210	In house	49.41		-1.11	
3218	ISO17234-1	58.408		-0.43	
3220	ISO17234-1	38.977		-1.89	
3225	ISO17234-1	69.4		0.40	
3228	ISO17234-1	58		-0.46	
3237	ISO17234-1	73.5		0.71	
3246	ISO17234-1	60.25		-0.29	
3248	ISO17234-1	64		-0.01	
3250	ISO17234-1	90.91		2.01	
8005	ISO17234-1	60.373		-0.28	
8030	ISO17234-1	79.7887		1.18	
	normality	OK			
	n	105			
	outliers	2			
	mean (n)	64.1204			
	st.dev. (n)	13.28371	RSD = 21%		
	R(calc.)	37.1944			
	st.dev.(ISO17234-1:20)	13.30077			
	R(ISO17234-1:20)	37.2422			Compare R(Horwitz) = 15.3566





APPENDIX 2 Other reported aromatic amines

Abbreviations

4AD	= 4-Aminodiphenyl (CASno. 92-67-1)
4CoT	= 4-Chloro-o-toluidine (CASno. 95-69-2)
2NA	= 2-Naphtylamine (CASno. 91-59-8)
ANT	= 2-Amino-4-nitrotoluene (CASno. 99-55-8)
4CA	= 4-Chloraniline (CASno. 106-47-8)
DAA	= 2,4-Diaminoanisol (CASno. 615-05-4)
DADM	= 4,4'-Diaminodiphenylmethane (CASno. 101-77-9)
DCB	= 3,3'-Dichlorobenzidine (CASno. 91-94-1)
DMoxB	= 3,3'-Dimethoxybenzidine (CASno. 119-90-4)
DMB	= 3,3'-Dimethylbenzidine (Casno. 119-93-7)
DDDM	= 3,3'-Dimethyl-4,4'-Diaminodiphenylmethane (CASno. 838-88-0)
pC	= p-Cresidine (CASno. 120-71-8)
DDM	= 4,4'-Diamino-3,3'-dichlorodiphenylmethane (CASno. 101-14-4)
DDE	= 4,4'-Diaminodiphenylether (CASno. 101-80-4)
DDS	= 4,4'-Diaminodiphenylsulfide (CASno. 139-65-1)
24DAT	= 2,4-Diaminotoluene (CASno. 95-80-7)
TMA	= 2,4,5-Trimethylaniline (CASno. 137-17-7)
oA	= o-Anisidine (CASno. 90-04-0)
24X	= 2,4-Xylidine (CASno. 95-68-1)
25X	= 2,5-Xylidine (CASno. 95-78-3)
26X	= 2,6-Xylidine (CASno. 87-62-7)
TX	= Total of Xylidines
oAAT	= o-Aminoazotoluene (CASno. 97-56-3)
oAT	= o-Aminoazotoluene
oT	= o-Toluidine (CASno. 95-53-4)
SUM	= Sum of o-Aminoazotoluene and o-Toluidine

Sample #21521; abbreviations explaned above

lab	4AD	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM	pC
210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
230	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
348	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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.	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.	n.d.
841	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2115	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2129	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2132	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2135	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2184	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2201	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2241	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2247	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2250	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2256	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2265	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2284	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2289	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2290	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2293	n.d.	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.	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.	n.d.
2330	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2364	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

lab	4AD	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM	pC
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	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2370	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2373	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2379	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2380	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2381	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2390	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2410	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2415	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2425	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2429	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2442	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2449	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2452	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2453	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2476	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2501	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2532	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2538	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2549	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2565	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2572	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2573	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2582	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2590	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2629	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2643	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2654	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2666	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2668	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2695	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2703	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2711	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2737	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2773	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2789	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2806	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
2823	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2826	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2829	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2870	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2910	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2959	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3100	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3160	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3185	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3192	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3218	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3220	n.d.	-----	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3225	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3246	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3248	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3250	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
8005	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
8030	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Sample #21521 -continued; abbreviations explaned on start of appendix 2

lab	DDM	DDE	DDS	24DAT	TMA	oA	24X	25X	26X	TX	oAAT	oT	SUM
210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
230	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
348	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	<5
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.	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.	n.d.	n.d.
841	<5	<5	<5	<5	<5	<5	<5	<5	<5	<15	<5	<5	<10
2115	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2129	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2132	<10	<10	<10	<10	<10	<10	<10	n.d.	<10	n.d.	<10	<10	n.d.
2135	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	-----
2184	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	-----
2201	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2241	<5	<5	<5	<5	<5	<5	<5	-----	<5	<5	<5	<5	<5
2247	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2250	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2256	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2265	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	-----
2284	<5	<5	<5	<5	<5	<5	<5	NA	<5	NA	<5	<5	<5
2289	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2290	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2293	n.d.	n.d.	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.	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.	n.d.	n.d.
2330	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2347	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	-----
2350	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	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	<5	n.d.
2367	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2370	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2373	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2379	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2380	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2381	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2390	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2410	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2415	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2425	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2429	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2442	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	n.d.
2449	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2452	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2453	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2472	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	-----
2476	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2501	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2532	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2538	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2549	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2565	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2572	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2573	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2582	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2590	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2629	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2643	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2654	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2666	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2668	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

lab	DDM	DDE	DDS	24DAT	TMA	oA	24X	25X	26X	TX	oAAT	oT	SUM
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	n.d.
2695	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2703	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2711	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2737	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2773	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2789	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2806	<30	<30	<30	<30	<30	<30	<30	-----	<30	-----	<30	<30	-----
2823	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2826	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2829	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2870	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2910	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	-----
2959	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3100	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	-----	<5.0	-----	<5.0	<5.0	<5.0
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3160	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	-----	< 5	< 5	-----
3176	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3185	< 5	< 5	< 5	< 5	< 5	< 5	< 5	-----	< 5	-----	< 5	< 5	< 5
3192	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3218	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3220	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3225	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	<10
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	-----
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3246	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3248	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3250	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
8005	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
8030	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	n.d.

APPENDIX 3 Analytical details
Sample #21520

lab	ISO/IEC17025 accredited	sample intake (g)	sample pre-treatment	remarks
210	Yes		---	
230	---		---	
339	---		---	
348	Yes	1	Further cut	
362	No	1.0245	Used as received	
551	---		---	
623	Yes	1	Further cut	
840	Yes	0.5	Further cut	
841	Yes	1	Further cut	
2115	---		---	
2129	Yes	0,5	Used as received	
2132	Yes	0.5	Used as received	
2135	Yes	0,9	Used as received	
2165	No	2	Used as received	
2184	Yes	2	Used as received	
2201	Yes	0.5	Further cut	
2238	Yes	1	Used as received	
2241	Yes	0.5.	Further cut	
2247	Yes	1-2	Further cut	
2250	Yes	0,5	Used as received	
2256	---		---	
2265	---		---	
2284	Yes	0.5	Further cut	
2289	Yes	1	Further cut	
2290	Yes		---	
2293	Yes	1.0	Used as received	
2310	Yes	0.5	Used as received	
2311	Yes	0.5	Used as received	
2330	Yes	0.50	Further cut	
2347	No	1.0	---	
2350	Yes	1.0222	Further cut	
2352	Yes	0.5001	Further cut	
2357	---		---	
2358	Yes	1.0	Further cut	
2364	Yes	0.5026	Used as received	
2365	Yes	1	Used as received	
2366	Yes	0.5	Further cut	
2367	No	0.5020	Used as received	
2370	Yes	0.5	Further cut	
2372	No	1	Used as received	
2373	Yes	0.5	Used as received	
2375	Yes	1	Further cut	
2378	Yes	1	Used as received	
2379	No	0.5	Further cut	
2380	Yes	1.00	Used as received	
2381	Yes	.5	Used as received	
2386	Yes	0,5	Used as received	
2390	Yes	1	Used as received	
2410	Yes	0.5	Used as received	
2415	Yes	0.5	Further cut	
2425	Yes	0.5	Further cut	
2429	---		---	
2442	Yes	1.0024	Further cut	
2449	---		---	
2452	Yes	0.5	Used as received	
2453	No	±1	Used as received	
2455	---		---	
2472	Yes	3	Used as received	
2476	---		---	
2501	Yes	1	Used as received	
2511	---		---	
2532	Yes	0.5003	Used as received	
2538	Yes	0,5	Used as received	
2549	Yes	0.5	Used as received	
2561	Yes	1	Used as received	
2565	Yes	1	Used as received	
2572	Yes		---	
2573	Yes	0.5	Used as received	
2582	Yes	1.0074	Used as received	
2590	Yes	1	Further cut	
2629	Yes	1.0	Further cut	
2643	Yes	0.5	Further grinded	
2654	No	1	Used as received	
2666	Yes	1,0087	Further cut	
2668	Yes	0.5	Further cut	

lab	ISO/IEC17025 accredited	sample intake (g)	sample pre-treatment	remarks
2674	No	2	Further cut	
2695	Yes	2	Used as received	
2703	---		---	
2711	No	1.028	Used as received	
2737	Yes	1	Further cut	
2773	Yes	1.0005	Further cut	
2789	Yes	1	Used as received	
2806	Yes		---	
2823	---		---	
2826	Yes	0.5	Used as received	
2829	No	1.0	Further cut	
2870	Yes	1	Further cut	
2910	Yes	0.5	Further cut	
2959	No		Used as received	
3100	Yes	0.806	Used as received	
3116	Yes	1	Used as received	
3153	Yes	0.5	Further cut	
3154	Yes		---	
3160	Yes	0,75	Used as received	
3172	---		---	
3176	Yes	0,5	Used as received	
3185	Yes	1	Used as received	
3192	Yes	1	Used as received	
3197	Yes	0,5	Further cut	
3210	Yes	0.5	Used as received	
3218	Yes	0.5	Used as received	
3220	Yes	1	Used as received	
3225	Yes	0.5	Further cut	
3228	Yes	3	Used as received	
3237	Yes	0,5	Further cut	
3246	Yes	1.00	Further cut	
3248	Yes	0.5	Further cut	
3250	Yes	1	Used as received	
8005	Yes	1	Used as received	
8030	No	0.5	Further cut	

Sample #21521

lab	ISO/IEC17025 accredited	sample intake (g)	sample pre-treatment	remarks
210	---		---	
230	---		---	
339	---		---	
348	Yes	1	Further cut	
362	---		---	
551	---		---	
623	Yes	1	Further cut	
840	Yes	0.5	Further cut	
841	Yes	1	Further cut	
2115	---		---	
2129	Yes	0,5	Used as received	
2132	Yes	1	Further cut	
2135	---		---	
2165	No	2	Used as received	
2184	Yes	2	Used as received	
2201	Yes	0.5	Further cut	
2238	Yes	1	Used as received	
2241	Yes	0.5	Further cut	
2247	---		---	
2250	Yes	0,5	Used as received	
2256	---		---	
2265	No	1	Further cut	
2284	Yes	0.5	Further cut	
2289	Yes	1	Further cut	
2290	Yes		---	
2293	Yes	1.0	Used as received	
2310	Yes	1.0	Further cut	
2311	Yes	0.5	Used as received	
2330	Yes	0.50	Further cut	
2347	No	1.0g	---	
2350	Yes	0.5270	Further cut	
2352	Yes	0.5021	Further cut	
2357	---		---	
2358	Yes	1.0	Further cut	
2364	Yes	0.5090	Further cut	
2365	Yes	1	Further cut	
2366	Yes	0.5	Further cut	
2367	Yes	0.5084	Further cut	
2370	Yes	0.5	Further cut	
2372	No	1	Used as received	
2373	Yes	0.5	Other	
2375	Yes	1	Further cut	
2378	Yes	1	Further cut	
2379	Yes	0.5	Further cut	
2380	Yes	1.00	Further cut	
2381	Yes	.5	Further cut	
2386	Yes	0,5	Further cut	
2390	Yes	1	Used as received	
2410	Yes	0.5	Further cut	
2415	Yes	0.5	Further cut	
2425	Yes	0.5	Further cut	
2429	---		---	
2442	Yes	1.0056	Further cut	
2449	Yes	1.0	Used as received	
2452	Yes	0.5	Further cut	
2453	No	±1	Further cut	
2455	---		---	
2472	Yes	3	Used as received	
2476	---		---	
2501	Yes	1	Further cut	
2511	---		---	
2532	Yes	0.5001	Further cut	
2538	No	1	Further cut	
2549	Yes	0.5	Used as received	
2561	Yes	1	Used as received	
2565	Yes	1	Further cut	
2572	Yes		---	
2573	Yes	0.5	Further cut	
2582	Yes	1.0077	Further cut	
2590	Yes	1	Further cut	
2629	---		---	
2643	Yes	0.5	Used as received	
2654	Yes	1	Further cut	
2666	Yes	1,0065	Further cut	
2668	Yes	0.5	Further cut	
2674	Yes	2	Further cut	

lab	ISO/IEC17025 accredited	sample intake (g)	sample pre-treatment	remarks
2695	Yes	2	Used as received	
2703	---		---	
2711	No	0.968	Further cut	
2737	Yes	1	Further cut	
2773	Yes	1.0005	Further cut	
2789	Yes	1	Used as received	
2806	Yes		---	
2823	Yes	1.0032	Further cut	
2826	Yes	1	Used as received	
2829	No	1.0	Further cut	
2870	Yes	1	Further cut	
2910	Yes	0.5	Further cut	
2959	No		Used as received	
3100	Yes	0.516	Further cut	
3116	Yes	1	Used as received	
3153	Yes	0.5	Further cut	
3154	Yes		---	
3160	Yes	0,75	Further cut	
3172	---		---	
3176	Yes	0,5	Used as received	
3185	Yes	1	Further cut	
3192	Yes	1	Further cut	
3197	Yes	0,5	Further cut	
3210	Yes	1	Further cut	
3218	Yes	0.5	Further cut	
3220	Yes	1	Used as received	
3225	Yes	0.5	Further cut	
3228	Yes	2	Further cut	
3237	Yes	0,5	Further cut	
3246	Yes	1.00	Used as received	
3248	Yes	0.5	Further cut	
3250	Yes	1	Used as received	
8005	No	1	Used as received	
8030	Yes	0.5	Further cut	

APPENDIX 4

Number of participants per country

4 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
2 labs in CAMBODIA
1 lab in EGYPT
2 labs in FRANCE
8 labs in GERMANY
1 lab in GUATEMALA
9 labs in HONG KONG
9 labs in INDIA
1 lab in INDONESIA
8 labs in ITALY
1 lab in JAPAN
1 lab in MAURITIUS
2 labs in MOROCCO
28 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in PORTUGAL
3 labs in SOUTH KOREA
3 labs in SPAIN
1 lab in SRI LANKA
2 labs in TAIWAN
2 labs in THAILAND
2 labs in TUNISIA
4 labs in TURKEY
1 lab in U.S.A.
3 labs in UNITED KINGDOM
7 labs in VIETNAM

APPENDIX 5

Abbreviations

C = final test result after checking of first reported suspect test 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
W = test result withdrawn on request of participant
ex = test result excluded from statistical evaluation
fr. = first reported
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected

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