

Results of Proficiency Test  
AZO Dyes in Leather  
March 2020

Organized by: Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## 1 INTRODUCTION

Since 1997 the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the determination of banned aromatic amines derived from AZO dyes in Leather. During the annual proficiency testing program 2019/2020 it was decided to continue the proficiency test for the analysis of banned aromatic amines derived AZO dyes in Leather.

In this interlaboratory study 114 laboratories in 27 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](http://www.iisnl.com).

## 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse 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 both positive on banned Aromatic Amines derived from AZO dyes of approximately 3.5 grams each. The first sample is a black/moss green leather labelled #20550 and the second sample is a black/turquoise green leather labelled #20551. 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 can be downloaded from the iis website [www.iisnl.com](http://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

For the first sample a batch of black/green leather made positive on o-Anisidine by a third party was selected. This batch was cut into small blocks. After homogenization the batch was divided over 150 subsamples in small bags of approximately 3.5 grams each and labelled #20550. The homogeneity of the subsamples was checked by determination of o-Anisidine using an in-house test method on eight stratified randomly selected subsamples.

	o-Anisidine in mg/kg
sample #20550-1	31.5
sample #20550-2	34.2
sample #20550-3	31.4
sample #20550-4	29.4
sample #20550-5	30.3
sample #20550-6	29.6
sample #20550-7	32.2
sample #20550-8	30.2

Table 1: homogeneity test results of subsamples #20550

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.

	o-Anisidine in mg/kg
r (observed)	4.4
reference test method	ISO17234-1:2015
0.3 * R (reference test method)	6.0

Table 2: evaluation of the repeatability of subsamples #20550

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

For the second sample a batch of black/turquoise green leather dyed with Direct Red 28 by a third party was selected. This batch was cut into small blocks. After homogenization the batch was divided over 150 subsamples in small bags of approximately 3.5 grams each and labelled #20551. The homogeneity of the subsamples was checked by determination of Benzidine using an in-house test method on eight stratified randomly selected subsamples.

	Benzidine in mg/kg
sample #20551-1	81.6
sample #20551-2	74.1
sample #20551-3	86.9
sample #20551-4	77.8
sample #20551-5	79.4
sample #20551-6	81.7
sample #20551-7	89.1
sample #20551-8	73.9

Table 3: homogeneity test results of subsamples #20551

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.

	Benzidine in mg/kg
r (observed)	15.4
reference test method	ISO17234-1:2015
0.3 * R (reference test method)	14.0

Table 4: evaluation of the repeatability of subsamples #20551

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

To each of the participating laboratories one sample labelled #20550 and one sample labelled #20551 were sent on March 4, 2020.

## 2.5 ANALYZES

The participants were asked to determine on sample #20550 and on sample #20551 the concentrations of 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)
- o-Aminoazotoluene (CASno. 97-56-3)
- 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)  
o-Toluidine (CASno. 95-53-4)  
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  
Sum of o-Aminoazotoluene and o-Toluidine

It was decided not to request p-Aminoazobenzene, CAS no. 60-09-3, because the samples were not positive for this component and to enable this determination more sample amount should be supplied. As it is never easy to obtain sample material it was therefore decided to remove this component from the list so that we can supply a lower sample amount to more participants.

It was requested, to ensure homogeneity, to not use less than 0.5 grams per determination and not to age or dry the samples. 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 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 results cannot be used for meaningful statistical evaluations.

To get comparable 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 method (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](http://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](http://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/](http://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.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. 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 was projected over the Kernel Density Graph for reference.

### 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 general when no literature reproducibility is available, another target may be 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 should be done in order to evaluate whether the reported test results are fit-for-purpose.

The z-scores were calculated in accordance with:

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

The  $z_{\text{(target)}}$  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 serious problems were encountered with the dispatch of the samples. A lot of participants informed iis that they were not able to report test results due to the measures taken to contain the Covid-19 pandemic in their countries. The reporting time on the data entry portal was extended. When the data entry portal was closed after some extra weeks twenty-four participants did not report any test results. For these participants an extra round was prepared on the data entry portal and these test results were not enclosed in this final report but will be evaluated in a later stage compared to this PT report.

In total ninety participants reported 166 numerical test results. Observed was 1 outlying test result, which is 0.6% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal. For the participants that did not report an extra reporting round was created on the data entry portal. Their test results will be evaluated afterwards against the group performance.

All original data sets given in appendix 1 proved to have a normal Gaussian 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 original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

The target reproducibility for o-Anisidine and Benzidine is mentioned in table B.1 of test method ISO17234-1:2015. For o-Anisidine the average over all reported reproducibilities was taken.

#### **Sample #20550**

o-Anisidine (CAS no. 90-04-0): The determination of this aromatic amine at a concentration level of 27 mg/kg was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ISO17234-1:2015.

#### **Sample #20551**

Benzidine (CAS no. 92-87-5): The determination of this aromatic amine at a concentration level of 43 mg/kg was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ISO17234-1:2015.

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 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 reproducibilities as declared by the relevant reference test method and the reproducibilities as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibilities (2.8 \* standard deviation) and the target reproducibilities, derived from the official test method ISO17234-1:2015 (equal to the reproducibility from LMBG 82.02.3:97) are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
o-Anisidine	mg/kg	78	27.3	47.0	17.6

Table 5: reproducibility on sample #20550

Component	unit	n	average	2.8 * sd	R(target)
Benzidine	mg/kg	87	42.9	34.7	24.9

Table 6: reproducibility on sample #20551

Without further statistical calculations, it can be concluded that the group of participating laboratories has a problem with the analysis of o-Anisidine and Benzidine in leather at the given concentration levels.

## 4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2020 WITH PREVIOUS PTS

	March 2020	March 2019	March 2018	February 2017	February 2016
Number of reporting laboratories	90	117	117	139	113
Number of test results	166	117	116	143	205
Number of statistical outliers	1	3	4	7	3
Percentage of statistical outliers	0.6%	2.6%	3.4%	4.9%	1.4%

Table 7: 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	29%	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	61%	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%

Table 8: development of uncertainties over the years

Components not listed in table 8 have not been tested in an iis PT.

Sample #20551 was used before in Proficiency Test iis17A02 as sample #17520. It is observed that the current PT findings of the leather subsamples containing Benzidine show a somewhat lower average concentration level and a somewhat higher calculated reproducibility.

	unit	#20551			#17520		
		n	average	R(calc)	n	average	R(calc)
Benzidine	mg/kg	87	42.9	34.7	126	51.4	29.0

Table 9: comparison of sample #20551 with sample #17520

#### 4.4 EVALUATION ANALYTICAL DETAILS

For this PT also some analytical details were requested and are listed in appendix 3.

Based on the answers given by the participants the following can be summarized:

- 90% of the participants mentioned that they are accredited for the determination of aromatic amine components.
- 34% of the participants used around 0.5 grams sample intake, 62% used around 1 gram and 4% used more than 1 grams as sample intake.
- 49% of the participants further cut the samples prior to analysis, 42% used the samples as received, 4% further grinded the samples and 5% used another sample preparation method.

The effect of the reported analytical details on the determination of aromatic amines in sample #20550 and #20551 was further investigated on those analytical details where it was possible to distinguish two or more meaningful subgroups. No statistically significant differences were found, see for example a subanalyzes in the next table.

Analytical Details	unit	n	average	RSD(%)
Around 0.5 g sample intake	mg/kg	22	26.7	58
Around 1 g sample intake	mg/kg	43	27.8	62
Further cut	mg/kg	35	26.6	67
Used as received	mg/kg	32	27.3	57

Table 10: effect of analytical details on o-Anisidine in leather sample #20550

## 5 DISCUSSION

Allmost all reporting participants were able to detect o-Anisidine in sample #20550 and Benzidine in sample #20551. Three laboratories did not find o-Anisidine in sample #20550. 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 11), it was noticed that not all participants would make an identical decision about the acceptability of the samples for the determined components. For sample #20550, about half of the reporting laboratories would accept the sample for all categories while the other half would have rejected the sample for all categories. For sample #20551, only six of the reporting laboratories would accept the sample for all categories while all other laboratories would have rejected the sample 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 11: bluesign® BSSL and LEATHER STANDARD by OEKO-TEX

## 6 CONCLUSION

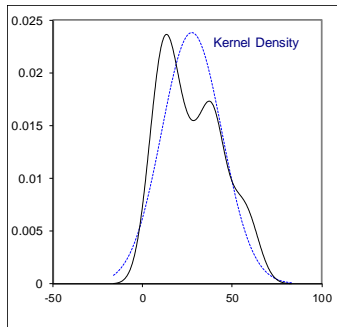
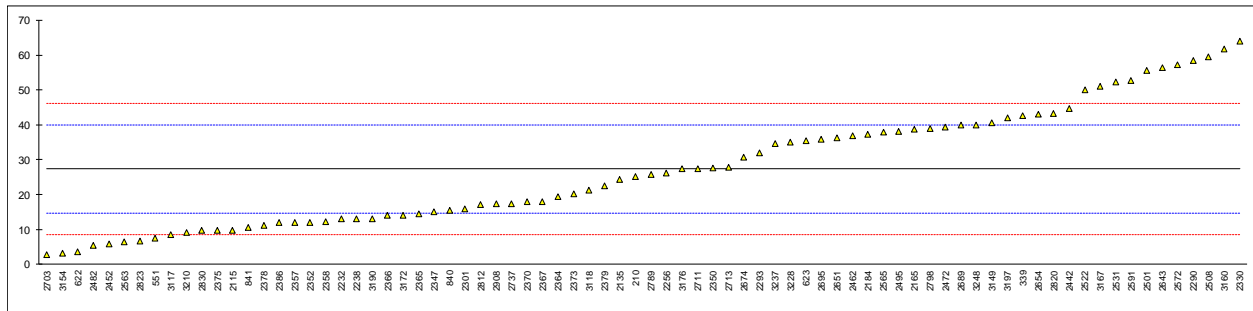
Although it can be concluded that some of the participants have a problem with the determination of o-Anisidine and Benzidine in the samples of this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

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 o-Ansidine (CAS no. 90-04-0) in sample #20550; results in mg/kg**

lab	method	value	mark	z(targ)	remarks
210	ISO17234-1	25.23		-0.33	
230		----		----	
339	In house	42.6		2.43	
348		----		----	
362		----		----	
551	In house	7.5281		-3.15	
622	ISO17234-1	3.60		-3.78	
623	ISO17234-1	35.38		1.28	
840	ISO17234-1	15.5		-1.88	
841	ISO17234-1	10.62		-2.66	
2108	ISO17234-1	<10		----	
2115	ISO17234-1	9.83		-2.78	
2129	ISO17234-1	<5	f-?	<-3.55	possibly a false negative test result?
2132	ISO17234-1	<5	f-?	<-3.55	possibly a false negative test result?
2135	ISO17234-1	24.4		-0.47	
2165	ISO17234-1	38.7		1.81	
2184	ISO17234-1	37.4		1.60	
2213		----		----	
2232	ISO17234-1	12.96		-2.29	
2238	ISO17234-1	13.0		-2.28	
2247		----		----	
2256	ISO17234-1	26.29		-0.17	
2266		----		----	
2290	ISO17234-1	58.5	C	4.96	first reported 80
2293	ISO17234-1	32.05		0.75	
2301	ISO14362-1	15.873		-1.82	
2310		----		----	
2311		----		----	
2330	ISO17234-1	63.96		5.83	
2347	ISO17234-1	15		-1.96	
2350	ISO17234-1	27.62		0.05	
2352	ISO17234-1	12		-2.44	
2357	ISO17234-1	12.0		-2.44	
2358	ISO17234-1	12.11		-2.42	
2364	ISO17234-1	19.34		-1.27	
2365	ISO17234-1	14.45		-2.05	
2366	ISO17234-1	14		-2.12	
2367	ISO17234-1	18.02		-1.48	
2370	ISO17234-1	17.9		-1.50	
2373		20.13		-1.15	
2375	ISO17234-1	9.8		-2.79	
2378	GB/T19942	11.1		-2.58	
2379	ISO17234-1	22.4316		-0.78	
2380		----		----	
2381		----		----	
2386	ISO17234-1	11.9		-2.45	
2390		----		----	
2425		----		----	
2442	ISO17234-1	44.65		2.75	
2449		----		----	
2452	ISO17234-1	5.75		-3.43	
2455		----		----	
2462	ISO17234-1/2	36.8		1.51	
2472	ISO17234-1	39.30		1.90	
2482	ISO17234-1	5.37		-3.49	
2492		----		----	
2495	ISO17234-1	38.19		1.73	
2501	ISO17234-1	55.65	C	4.50	first reported 55.65 in the wrong input field
2508	ISO17234-1	59.43		5.11	
2511		----		----	
2522	GB/T19942	50.03		3.61	
2531	ISO17234-1	52.4		3.99	
2549		----		----	
2561		----		----	
2563	ISO17234-1	6.47		-3.32	
2565	ISO17234-1	37.95		1.69	
2569		----		----	
2572	ISO17234-1	57.3	C	4.77	first reported 78.1
2590		----		----	
2591	ISO17234-1	52.8		4.05	
2643	ISO17234-1	56.452		4.63	
2644		----		----	
2651	ISO17234-1	36.189		1.41	
2654	ISO17234-1	43.10		2.51	
2668		----		----	

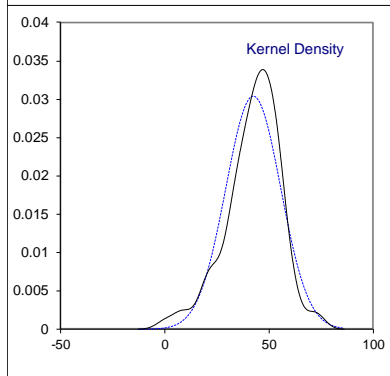
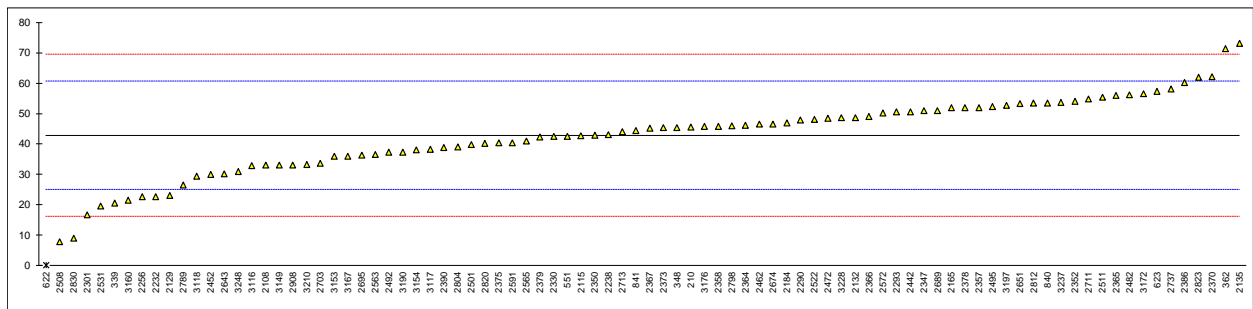
lab	method	value	mark	z(targ)	remarks
2674	ISO17234-1	30.8		0.55	
2689	ISO17234-1	40		2.01	
2695	ISO17234-1	35.8		1.35	
2703	ISO17234-1	2.632		-3.93	
2711	ISO17234-1	27.48		0.02	
2713	ISO17234-1	27.87		0.09	
2723		----		----	
2730		----		----	
2737	GB/T19942	17.4	C	-1.58	first reported 78.11
2743		----		----	
2789	ISO17234-1	25.75		-0.25	
2798	ISO17234-1	39		1.86	
2804	ISO17234-1	<10		----	
2806		----		----	
2812	ISO17234-1	17.21		-1.61	
2820	ISO17234-1	43.2		2.52	
2823	ISO17234-1	6.59554		-3.30	
2829		----		----	
2830	ISO17234-1	9.74	C	-2.80	first reported ND
2870		----		----	
2908	ISO17234-1	17.33		-1.59	
2912		----		----	
3116		----		----	
3117	ISO17234-1	8.55		-2.99	
3118	ISO17234-1	21.33		-0.95	
3149	ISO17234-1	40.6		2.11	
3153	ISO17234-1	<5	f-?	<-3.55	possibly a false negative test result?
3154	EN14362-1	3.24		-3.83	
3160	ISO17234-1	61.87		5.49	
3167	ISO17234-1	51		3.76	
3172	ISO17234-1	14.112	C	-2.10	first reported <5
3176	ISO17234-1	27.38		0.01	
3190	ISO17234-1	13.0		-2.28	
3197	ISO17234-1	42.1		2.35	
3210	In house	9.14		-2.89	
3216		----		----	
3228	ISO17234-1	35.1		1.24	
3237	ISO17234-1	34.69		1.17	
3248	EN14362-1	40		2.01	
normality		OK			
n		78			
outliers		0			
mean (n)		27.3330			
st.dev. (n)		16.77070	RSD=61%		
R(calc.)		46.9580			
st.dev.(ISO17234-1:2015)		6.28659			
R(ISO17234-1:2015)		17.6024			
Compare:					
R(Horwitz)		7.4426			



## Determination of Benzidine (CAS no. 92-87-5) in sample #20551; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17234-1	45.51		0.30	
230		----		----	
339	In house	20.6		-2.50	
348	In house	45.36		0.28	
362	EN14362-1	71.4		3.21	
551	In house	42.4630		-0.05	
622	ISO17234-1	0.14	C,R(0.05)	-4.81	first reported 0
623	ISO17234-1	57.25	C	1.62	first reported 83.42
840	ISO17234-1	53.5		1.19	
841	ISO17234-1	44.45		0.18	
2108	ISO17234-1	33		-1.11	
2115	ISO17234-1	42.69		-0.02	
2129	ISO17234-1	23.1		-2.22	
2132	ISO17234-1	48.6444		0.65	
2135	ISO17234-1	73.07		3.39	
2165	ISO17234-1	51.9		1.01	
2184	ISO17234-1	47		0.46	
2213		----		----	
2232	ISO17234-1	22.7		-2.27	
2238	ISO17234-1	43.0		0.01	
2247		----		----	
2256	ISO17234-1	22.67		-2.27	
2266		----		----	
2290	ISO17234-1	47.8		0.55	
2293	ISO17234-1	50.58		0.87	
2301	ISO14362-1	16.664		-2.95	
2310		----		----	
2311		----		----	
2330	ISO17234-1	42.42		-0.05	
2347	GB/T19942	51		0.91	
2350	ISO17234-1	42.85		0.00	
2352	ISO17234-1	54		1.25	
2357	ISO17234-1	52.0		1.03	
2358	ISO17234-1	45.73		0.32	
2364	ISO17234-1	46.11		0.36	
2365	ISO17234-1	55.95		1.47	
2366	ISO17234-1	49		0.69	
2367	ISO17234-1	45.15		0.26	
2370	ISO17234-1	62.2		2.17	
2373		45.32		0.27	
2375	ISO17234-1	40.4		-0.28	
2378	GB/T19942	52.0		1.03	
2379	ISO17234-1	42.2452		-0.07	
2380		----		----	
2381		----		----	
2386	ISO17234-1	60.3		1.96	
2390		38.87		-0.45	
2425		----		----	
2442	ISO17234-1	50.63		0.87	
2449		----		----	
2452	ISO17234-1	30.01		-1.45	
2455		----		----	
2462	ISO17234-1/2	46.5		0.41	
2472	ISO17234-1	48.47		0.63	
2482	ISO17234-1	56.2		1.50	
2492	ISO17234-1	37.4		-0.62	
2495	ISO17234-1	52.30		1.06	
2501	ISO17234-1	39.80		-0.35	
2508	ISO17234-1	7.780	C	-3.95	first reported 6.047
2511	ISO17234-1	55.4570		1.41	
2522	GB/T19942	48.02		0.58	
2531	ISO17234-1	19.62	C	-2.62	first reported 0
2549		----		----	
2561		----		----	
2563	ISO17234-1	36.6		-0.71	
2565	ISO17234-1	41.04		-0.21	
2569		----		----	
2572	ISO17234-1	50.2		0.82	
2590		----		----	
2591	ISO17234-1	40.4		-0.28	
2643	ISO17234-1	30.237		-1.42	
2644		----		----	
2651	ISO17234-1	53.238		1.16	
2654	ISO17234-1	Not detected		----	
2668		----		----	

lab	method	value	mark	z(target)	remarks
2674	ISO17234-1	46.6		0.42	
2689	ISO17234-1	51		0.91	
2695	ISO17234-1	36.35		-0.73	
2703	ISO17234-1	33.656		-1.04	
2711	ISO17234-1	54.74		1.33	
2713	ISO17234-1	43.99		0.12	
2723		----		----	
2730		----		----	
2737	GB/T19942	58.16		1.72	
2743		----		----	
2789	ISO17234-1	26.58		-1.83	
2798	ISO17234-1	46		0.35	
2804	ISO17234-1	39.0		-0.44	
2806		----		----	
2812	ISO17234-1	53.42		1.18	
2820	ISO17234-1	40.1		-0.31	
2823	ISO17234-1	61.9576		2.14	
2829		----		----	
2830	ISO17234-1	9.03	C	-3.81	first reported ND
2870		----		----	
2908	ISO17234-1	33.11		-1.10	
2912		----		----	
3116	ISO17234-1	32.97		-1.11	
3117	ISO17234-1	38.35		-0.51	
3118	ISO17234-1	29.50		-1.50	
3149	ISO17234-1	33.0		-1.11	
3153	ISO17234-1	36.0		-0.77	
3154	EN14362-1	38.04		-0.54	
3160	ISO17234-1	21.56		-2.40	
3167	ISO17234-1	36		-0.77	
3172	ISO17234-1	56.467		1.53	
3176	ISO17234-1	45.69		0.32	
3190	ISO17234-1	37.4		-0.62	
3197	ISO17234-1	52.7		1.10	
3210	In house	33.3		-1.08	
3216		----		----	
3228	ISO17234-1	48.6		0.64	
3237		53.60		1.21	
3248	EN14362-1	31		-1.34	
normality		OK			
n		87			
outliers		1			
mean (n)		42.8812			
st.dev. (n)		12.40056	RSD=29%		
R(calc.)		34.7216			
st.dev.(ISO17234-1:2015)		8.89504			
R(ISO17234-1:2015)		24.9061			
Compare					
R(Horwitz)		10.9110			





**APPENDIX 2 Other reported aromatic amines****Abbreviations**

4AD	= 4-Aminodiphenyl (CASno. 92-67-1)
B	= Benzidine (CASno. 92-87-5)
4CoT	= 4-Chloro-o-toluidine (CASno. 95-69-2)
2NA	= 2-Naphtylamine (CASno. 91-59-8)
oAAT	= o-Aminoazotoluene (CASno. 97-56-3)
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)
oT	= o-Toluidine (CASno. 95-53-4)
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
SUM	= Sum of o-Aminoazotoluene and o-Toluidine

**Sample #20550; abbreviations explained above**

lab	4AD	B	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM
210	----	----	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----	----	----
339	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	----
348	----	----	----	----	----	----	----	----	----	----	----	----	----
362	----	----	----	----	----	----	----	----	----	----	----	----	----
551	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.
622	0	0	0	0	0	0	0	0	0	0	0	0	0
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	<5	<5	<5	<5
2108	----	----	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	----	----	----	----	----
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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.	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.	n.d.
2213	----	----	----	----	----	----	----	----	----	----	----	----	----
2232	----	----	----	----	----	----	----	----	----	----	----	----	----
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2247	----	----	----	----	----	----	----	----	----	----	----	----	----
2256	----	----	----	----	----	----	----	----	----	----	----	----	----
2266	----	----	----	----	----	----	----	----	----	----	----	----	----
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	< 5.0
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.
2301	----	----	----	----	----	----	----	----	----	----	----	----	----
2310	----	----	----	----	----	----	----	----	----	----	----	----	----
2311	----	----	----	----	----	----	----	----	----	----	----	----	----
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	<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	<5	<5



Sample #20550 -continued; abbreviations explained on start of appendix 2

lab	pC	DDM	DDE	DDS	oT	24DAT	TMA	24X	25X	26X	TX	SUM
210	----	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----	----
339	<5	----	----	----	<5	<5	<5	----	----	----	----	----
348	----	----	----	----	----	----	----	----	----	----	----	----
362	----	----	----	----	----	----	----	----	----	----	----	----
551	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	----	n.d.
622	0	0	0	0	0	0	0	0	0	0	0	0
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
2108	----	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	----	----	----	----
2132	<5	<5	<5	<5	<5	<5	<5	<5	n.a.	<5	n.a.	n.a.
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.
2213	----	----	----	----	----	----	----	----	----	----	----	----
2232	----	----	----	----	----	----	----	----	----	----	----	----
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2247	----	----	----	----	----	----	----	----	----	----	----	----
2256	----	----	----	----	----	----	----	----	----	----	----	----
2266	----	----	----	----	----	----	----	----	----	----	----	----
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
2293	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	n.d.	n.d.
2301	----	----	----	----	----	----	----	----	----	----	----	----
2310	----	----	----	----	----	----	----	----	----	----	----	----
2311	----	----	----	----	----	----	----	----	----	----	----	----
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
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	----	----	----	----	----	----	----	----	----	----	----	----
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.	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
2373	----	----	----	----	----	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----	----	----	----	----	----
2378	----	----	----	----	----	----	----	----	----	----	----	----
2379	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	----
2380	----	----	----	----	----	----	----	----	----	----	----	----
2381	----	----	----	----	----	----	----	----	----	----	----	----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<15	<5
2390	----	----	----	----	----	----	----	----	----	----	----	----
2425	----	----	----	----	----	----	----	----	----	----	----	----
2442	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	----	----	----
2449	----	----	----	----	----	----	----	----	----	----	----	----
2452	----	----	----	----	----	----	----	----	----	----	----	----
2455	----	----	----	----	----	----	----	----	----	----	----	----
2462	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	----	----
2472	<5	<5	<5	<5	<5	<5	<5	<5	----	<5	----	<5
2482	----	----	----	----	----	----	----	----	----	----	----	----
2492	----	----	----	----	----	----	----	----	----	----	----	----
2495	<5	<5	<5	<5	<5	<5	<5	<5	<5	----	----	----
2501	----	----	----	----	----	----	----	----	----	----	----	----
2508	----	----	----	----	----	----	----	----	----	----	----	----
2511	----	----	----	----	----	----	----	----	----	----	----	----
2522	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	n.a.	<5.0	n.a.	<5.0
2531	0	0	0	0	0	0	0	0	----	0	----	0
2549	----	----	----	----	----	----	----	----	----	----	----	----
2561	----	----	----	----	----	----	----	----	----	----	----	----
2563	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2565	<5	<5	<5	<5	<5	<5	<5	<5	----	<5	----	<5
2569	----	----	----	----	----	----	----	----	----	----	----	----
2572	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2590	----	----	----	----	----	----	----	----	----	----	----	----
2591	----	----	----	----	----	----	----	----	----	----	----	----
2643	----	----	----	----	----	----	----	----	----	----	----	----
2644	----	----	----	----	----	----	----	----	----	----	----	----
2651	----	----	----	----	----	----	----	----	----	----	----	----
2654	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	----	----
2668	----	----	----	----	----	----	----	----	----	----	----	----

lab	pC	DDM	DDE	DDS	oT	24DAT	TMA	24X	25X	26X	TX	SUM
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	----	----
2689	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	n.d.	n.d.
2695	----	----	----	----	----	----	----	----	----	----	----	----
2703	----	----	----	----	----	----	----	----	----	----	----	----
2711	----	----	----	----	----	----	----	----	----	----	----	----
2713	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2723	----	----	----	----	----	----	----	----	----	----	----	----
2730	----	----	----	----	----	----	----	----	----	----	----	----
2737	----	----	----	----	----	----	----	----	----	----	----	----
2743	----	----	----	----	----	----	----	----	----	----	----	----
2789	----	----	----	----	----	----	----	----	----	----	----	----
2798	----	----	----	----	----	----	----	----	----	----	----	----
2804	<10	<10	<10	<10	<10	<10	<10	----	----	----	----	<10
2806	----	----	----	----	----	----	----	----	----	----	----	----
2812	----	----	----	----	----	----	----	----	----	----	----	----
2820	----	----	----	----	----	----	----	----	----	----	----	----
2823	----	----	----	----	<b>41.5245</b>	----	----	----	----	----	----	----
2829	----	----	----	----	----	----	----	----	----	----	----	----
2830	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	----	----	----	----	----	----	----	----	----	----	----	----
2908	----	----	----	----	----	----	----	----	----	----	----	----
2912	----	----	----	----	----	----	----	----	----	----	----	----
3116	----	----	----	----	----	----	----	----	----	----	----	----
3117	----	----	----	----	----	----	----	----	----	----	----	----
3118	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3149	----	----	----	----	----	----	----	----	----	----	----	----
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	----	----	----	----	----	----	----	----	----	----	----	----
3160	----	----	----	----	----	----	----	----	----	----	----	----
3167	----	----	----	----	----	----	----	----	----	----	----	----
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176	----	----	----	----	----	----	----	----	----	----	----	----
3190	----	----	----	----	----	----	----	----	----	----	----	----
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3210	----	----	----	----	----	----	----	----	----	----	----	----
3216	----	----	----	----	----	----	----	----	----	----	----	----
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	----	n.d.
3237	----	----	----	----	----	----	----	----	----	----	----	----
3248	----	----	----	----	----	----	----	----	----	----	----	----

Lab 2823 first reported 85.6648. The original and revised test results are possibly false positives

Sample #20551; abbreviations explained on start of appendix 2

lab	4AD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM
210	----	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----	----
339	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	----
348	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
362	----	----	----	----	----	----	----	----	----	----	----	----
551	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
622	0	0	0	0	0	0	0	0	0	0	0	0
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
2108	----	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	----	----	----	----
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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.
2213	----	----	----	----	----	----	----	----	----	----	----	----
2232	----	----	----	----	----	----	----	----	----	----	----	----
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2247	----	----	----	----	----	----	----	----	----	----	----	----
2256	----	----	----	----	----	----	----	----	----	----	----	----
2266	----	----	----	----	----	----	----	----	----	----	----	----
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
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.
2301	----	----	----	----	----	----	----	----	----	----	----	----
2310	----	----	----	----	----	----	----	----	----	----	----	----
2311	----	----	----	----	----	----	----	----	----	----	----	----
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	----	----	----	----	----	----	----	----	----	----	----	----



lab	4AD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM
3210	----	----	----	----	----	----	----	----	----	----	----	----
3216	----	----	----	----	----	----	----	----	----	----	----	----
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	----	----	----	----	----	----	----	----	----	----	----	----
3248	----	----	----	----	----	----	----	----	----	----	----	----

Sample #20551 -continued; abbreviations explained on start of appendix 2

lab	pC	DDM	DDE	DDS	oT	24DAT	TMA	oA	24X	25X	26X	TX	SUM
210	----	----	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----	----	----
339	<5	----	----	----	<5	<5	<5	<5	----	----	----	----	----
348	<5	<5	<5	<5	<5	<5	<5	<5	<5	----	<5	----	<5
362	----	----	----	----	----	----	----	----	----	----	----	----	----
551	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	----	n.d.
622	0	0	0	0	0	0	0	0	0	0	0	0	0
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	<5	<5	<5	<5
2108	----	----	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	----	----	----	----	----
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	n.a.	<5	n.a.	n.a.
2135	----	----	----	----	----	----	----	----	----	----	----	----	----
2165	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.
2213	----	----	----	----	----	----	----	----	----	----	----	----	----
2232	----	----	----	----	----	----	----	----	----	----	----	----	----
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2247	----	----	----	----	----	----	----	----	----	----	----	----	----
2256	----	----	----	----	----	----	----	----	----	----	----	----	----
2266	----	----	----	----	----	----	----	----	----	----	----	----	----
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	< 5.0
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.
2301	----	----	----	----	----	----	----	----	----	----	----	----	----
2310	----	----	----	----	----	----	----	----	----	----	----	----	----
2311	----	----	----	----	----	----	----	----	----	----	----	----	----
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	<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	<5	<5
2366	<5	<5	<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.	----	----
2370	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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.	----	----
2380	----	----	----	----	----	----	----	----	----	----	----	----	----
2381	----	----	----	----	----	----	----	----	----	----	----	----	----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<15	<5
2390	----	----	----	----	----	----	----	----	----	----	----	----	----
2425	----	----	----	----	----	----	----	----	----	----	----	----	----
2442	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	----	----	----
2449	----	----	----	----	----	----	----	----	----	----	----	----	----
2452	----	----	----	----	----	----	----	----	----	----	----	----	----
2455	----	----	----	----	----	----	----	----	----	----	----	----	----
2462	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	----	----
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	----	<5	----	<5
2482	----	----	----	----	----	----	----	----	----	----	----	----	----
2492	----	----	----	----	----	----	----	----	----	----	----	----	----
2495	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	----	----	----
2501	----	----	----	----	----	----	----	----	----	----	----	----	----
2508	----	----	----	----	----	----	----	----	----	----	----	----	----
2511	----	----	----	----	----	----	----	----	----	----	----	----	----
2522	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	n.a.	<5.0	n.a.	<5.0
2531	0	0	0	0	0	<5.00	0	0	0	----	0	----	0
2549	----	----	----	----	----	----	----	----	----	----	----	----	----
2561	----	----	----	----	----	----	----	----	----	----	----	----	----
2563	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.
2565	<5	<5	<5	<5	<5	<5	<5	<5	<5	----	<5	----	<5
2569	----	----	----	----	----	----	----	----	----	----	----	----	----
2572	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0

lab	pC	DDM	DDE	DDS	oT	24DAT	TMA	oA	24X	25X	26X	TX	SUM
2590	----	----	----	----	----	----	----	----	----	----	----	----	----
2591	----	----	----	----	----	----	----	----	----	----	----	----	----
2643	----	----	----	----	----	----	----	----	----	----	----	----	----
2644	----	----	----	----	----	----	----	----	----	----	----	----	----
2651	----	----	----	----	----	----	----	----	----	----	----	----	----
2654	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	----	----
2668	----	----	----	----	----	----	----	----	----	----	----	----	----
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	----	----
2689	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	----	----	----	----	----	----	----	----	----	----	----	----	----
2703	----	----	----	----	----	----	----	0.058	----	----	----	----	----
2711	----	----	----	----	----	----	----	----	----	----	----	----	----
2713	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2723	----	----	----	----	----	----	----	----	----	----	----	----	----
2730	----	----	----	----	----	----	----	----	----	----	----	----	----
2737	----	----	----	----	----	----	----	----	----	----	----	----	----
2743	----	----	----	----	----	----	----	----	----	----	----	----	----
2789	----	----	----	----	----	----	----	----	----	----	----	----	----
2798	----	----	----	----	----	----	----	----	----	----	----	----	----
2804	<10	<10	<10	<10	<10	<10	<10	<10	----	----	----	----	<10
2806	----	----	----	----	----	----	----	----	----	----	----	----	----
2812	----	----	----	----	----	----	----	----	----	----	----	----	----
2820	----	----	----	----	----	----	----	----	----	----	----	----	----
2823	----	----	----	----	----	----	----	----	----	----	----	----	----
2829	----	----	----	----	----	----	----	----	----	----	----	----	----
2830	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	----	----	----	----	----	----	----	----	----	----	----	----	----
2908	----	----	----	----	----	----	----	----	----	----	----	----	----
2912	----	----	----	----	----	----	----	----	----	----	----	----	----
3116	----	----	----	----	----	----	----	----	----	----	----	----	----
3117	----	----	----	----	----	----	----	----	----	----	----	----	----
3118	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.
3149	----	----	----	----	----	----	----	----	----	----	----	----	----
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	----	----	----	----	----	----	----	----	----	----	----	----	----
3160	----	----	----	----	----	----	----	----	----	----	----	----	----
3167	----	----	----	----	----	----	----	----	----	----	----	----	----
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176	----	----	----	----	----	----	----	----	----	----	----	----	----
3190	----	----	----	----	----	----	----	----	----	----	----	----	----
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3210	----	----	----	----	----	----	----	----	----	----	----	----	----
3216	----	----	----	----	----	----	----	----	----	----	----	----	----
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	----	n.d.
3237	----	----	----	----	----	----	----	----	----	----	----	----	----
3248	----	----	----	----	----	----	----	----	----	----	----	----	----

## APPENDIX 3 Analytical details

lab	ISO/IEC17025 accredited	sample intake (g)	sample pre-treatment	remarks
210	Yes	---	---	---
230	---	---	---	---
339	No	---	---	---
348	No	1	Further Cut	---
362	Yes	1.0	Used as received	---
551	Yes	1.0	Further Cut	---
622	Yes	whole sample	Further Cut	---
623	Yes	1	Further Cut	---
840	Yes	0.5	Further Cut	---
841	Yes	0.5	Further Cut	---
2108	Yes	0,5	Further Cut	---
2115	Yes	0.5	Used as received	---
2129	Yes	0.5	Used as received	---
2132	Yes	0.5	Further Cut	---
2135	Yes	0,8	Used as received	---
2165	Yes	1	Used as received	---
2184	Yes	1	Used as received	---
2213	---	---	---	---
2232	Yes	1	Further Cut	---
2238	Yes	1	Used as received	---
2247	---	---	---	---
2256	Yes	1.0032&1.0043	Further Grinded	---
2266	---	---	---	---
2290	---	---	---	---
2293	No	1.000	Used as received	---
2301	Yes	1	Further Cut	---
2310	---	---	---	---
2311	---	---	---	---
2330	Yes	1	Used as received	---
2347	Yes	---	---	other method:GB/T 19942-2005
2350	---	---	Used as received	---
2352	Yes	0.5	Further Cut	---
2357	---	---	---	---
2358	---	---	---	---
2364	Yes	1.0041&1.0019	Other	#20550 as received. #20551 didi further cut.
2365	Yes	0.5	Further Cut	---
2366	Yes	1.0	Further Cut	---
2367	Yes	0.5006&0.5002	Other	#20550 used as received #20551 cut 5*5mm
2370	Yes	0.5	Further Cut	---
2373	Yes	0.5	Other	#20550 Used as received #20551 Further Cut
2375	Yes	1,0	Further Cut	---
2378	Yes	1	Used as received	---
2379	Yes	1	Further Cut	---
2380	---	---	---	---
2381	---	---	---	---
2386	Yes	0,5	Further Cut	---
2390	---	---	---	---
2425	---	---	---	---
2442	Yes	1.0073&1.0036	Further Cut	---
2449	---	---	---	---
2452	---	---	---	---
2455	---	---	---	---
2462	Yes	1	Further Cut	---
2472	Yes	1	Further Cut	---
2482	Yes	0,5	Used as received	---
2492	Yes	0.5	Used as received	---
2495	Yes	0.5	Used as received	---
2501	No	1.0014	Further Cut	---
2508	Yes	0.5 & 1	Further Cut	0.5 sample1, 0.5 & 1 sample2 due to low concentration analyte
2511	---	---	---	---
2522	Yes	1	Further Cut	TEST METHOD GB/T 19942-2005
2531	Yes	1	Used as received	---
2549	---	---	---	---
2561	---	---	---	---
2563	Yes	1	Used as received	---
2565	---	---	---	---
2569	---	---	---	---
2572	---	---	---	---
2590	---	---	---	---
2591	No	1	Further Cut	---
2643	Yes	0.4995	Used as received	---
2644	---	---	---	---
2651	Yes	0.5 per sample	Used as received	---
2654	Yes	1	Further Cut	---



lab	ISO/IEC17025 accredited	sample intake (g)	sample pre-treatment	remarks
2668	---	---	---	---
2674	Yes	1.0	Used as received	---
2689	Yes	0.5	Further Cut	---
2695	Yes	2	Further Grinded	---
2703	Yes	1.0	Further Cut	---
2711	No	1.006	Further Cut	Careful that your system assigns same weight to both samples
2713	Yes	0.75	Further Cut	---
2723	---	---	---	---
2730	---	---	---	---
2737	Yes	0.5	Further Cut	Method GB/T 19942-2019
2743	---	---	---	---
2789	Yes	1	Used as received	---
2798	Yes	0.5	Used as received	---
2804	No	1	Used as received	---
2806	---	---	---	---
2812	Yes	1	Used as received	---
2820	Yes	1	Used as received	---
2823	Yes	1.0060&1.0025	Further Cut	---
2829	No	1,0	Further Cut	---
2830	Yes	1	Used as received	---
2870	---	---	---	---
2908	Yes	1	Further Cut	we founded also the aniline in 20550 with a result: 63.8 mg/kg
2912	---	---	---	---
3116	Yes	1	Used as received	---
3117	Yes	1.001 & 0.995	Other	#20550:Used as received #20551:Further Cut
3118	Yes	0.5000	Used as received	---
3149	Yes	1	Used as received	---
3153	Yes	0.5	Further Grinded	---
3154	Yes	1	Used as received	---
3160	Yes	0.9	Further Cut	---
3167	Yes	1.00	Further Cut	---
3172	Yes	2	Further Cut	---
3176	Yes	0,5	Further Cut	---
3190	Yes	1	Used as received	---
3197	Yes	0,5	Further Cut	---
3210	---	1	Further Cut	---
3216	---	---	---	---
3228	Yes	1	Used as received	---
3237	Yes	0,5	Used as received	---
3248	Yes	0.5	Used as received	---

## APPENDIX 4

### Number of participants per country

4 labs in BANGLADESH  
1 lab in BRAZIL  
1 lab in BULGARIA  
1 lab in CAMBODIA  
4 labs in FRANCE  
9 labs in GERMANY  
1 lab in GUATEMALA  
8 labs in HONG KONG  
8 labs in INDIA  
4 labs in INDONESIA  
13 labs in ITALY  
1 lab in JAPAN  
1 lab in MAURITIUS  
4 labs in MOROCCO  
25 labs in P.R. of CHINA  
2 labs in PAKISTAN  
1 lab in SINGAPORE  
2 labs in SOUTH KOREA  
5 labs in SPAIN  
1 lab in SWITZERLAND  
1 lab in TAIWAN  
1 lab in THAILAND  
2 labs in TUNISIA  
6 labs in TURKEY  
1 lab in U.S.A.  
3 labs in UNITED KINGDOM  
4 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
f-?	= possibly a false negative test result?
f+?	= possibly a false positive test result?
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected

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