

# Results of Proficiency Test AZO Dyes in Leather/Footwear February 2023

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 scheme for the determination of banned aromatic amines derived from AZO dyes in Leather/Footwear every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of banned aromatic amines derived from AZO dyes in Leather/Footwear.

In this interlaboratory study 84 laboratories in 27 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/Footwear 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 #23505 and #23506 respectively.

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

For the first sample a batch of leather, one side blue-green/one side black, was selected with a detectable level of banned aromatic amine derived from AZO dyes. After homogenization 120 small plastic bags were filled with approximately 3 grams each and labelled #23505. The batch for sample #23505 was used in a previous proficiency test on AZO Dyes in Leather/Footwear (as sample #17520 in PT iis17A02). Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of leather, one side green/one side black, was selected with a detectable level of banned aromatic amine derived from AZO dyes. After homogenization 120 small plastic bags were filled with approximately 3 grams each and labelled #23506. The batch for sample #23506 was used in a previous proficiency test on AZO Dyes in Leather/Footwear (as sample #20550 in PT iis20A05). Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories two leather samples labelled #23505 and #23506 were sent on January 25, 2023.

#### 2.5 ANALYZES

The participants were requested to determine the following aromatic amines on both samples #23505 and #23506:

4-Aminodiphenyl (CAS No. 92-67-1)

Benzidine (CAS No. 92-87-5)

- 4-Chloro-o-toluidine (CAS No. 95-69-2)
- 2-Naphtylamine (CAS No. 91-59-8)
- 2-Amino-4-nitrotoluene (CAS No. 99-55-8)
- 4-Chloraniline (CAS No. 106-47-8)
- 2,4-Diaminoanisol (CAS No. 615-05-4)
- 4,4'-Diaminodiphenylmethane (CAS No. 101-77-9)
- 3,3'-Dichlorobenzidine (CAS No. 91-94-1)
- 3,3'-Dimethoxybenzidine (CAS No. 119-90-4)
- 3,3'-Dimethylbenzidine (Cas No. 119-93-7)
- 3,3'-Dimethyl-4,4'-Diaminodiphenylmethane (CAS No. 838-88-0)
- p-Cresidine (CAS No. 120-71-8)
- 4,4'-Diamino-3,3'-dichlorodiphenylmethane (CAS No. 101-14-4)
- 4,4'-Diaminodiphenylether (CAS No. 101-80-4)
- 4,4'-Diaminodiphenylsulfide (CAS No. 139-65-1)
- 2,4-Diaminotoluene (CAS No. 95-80-7)
- 2,4,5-Trimethylaniline (CAS No. 137-17-7)
- o-Anisidine (CAS No. 90-04-0)
- 2,4-Xylidine (CAS No. 95-68-1)
- 2,5-Xylidine (CAS No. 95-78-3)
- 2,6-Xylidine (CAS No. 87-62-7)

**Total Xylidines** 

- o-Aminoazotoluene (CAS No. 97-56-3)
- o-Toluidine (CAS No. 95-53-4)

Sum of o-Aminoazotoluene and o-Toluidine

To ensure homogeneity it was requested not to use less than 0.5 gram per determination and not to age and/or dry the samples, nor to determine volatile matter. It was also requested to report if the laboratory was accredited for the determined 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 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 appendices 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. 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 data set 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 F(0.01) for the Rosner's test. Stragglers are marked by F(0.01) for the Dixon's test, by F(0.01) 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 (derived from e.g. ISO or ASTM test methods), 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 according to:

```
z_{\text{(target)}} = \text{(test result - 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 proficiency test no problems were encountered with the dispatch of the samples. Nine participants reported test results after the final reporting date and four other participants did not report any test results. Not all participants were able to report all components requested.

In total 80 participants reported 156 numerical test results. Observed were 4 outlying test results, which is 2.6%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Both data sets 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 tests 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.

Test method ISO17234 is considered to be the official test method for the determination of aromatic amines derived from AZO dyes in Leather/Footwear. Unfortunately, only for a few aromatic amines precision data are mentioned in this test method and if mentioned the precision data is often not for a large concentration range and sometimes not conclusive. As alternative for the aromatic amines not mentioned in the test method iis had used an estimated target reproducibility calculated with the Horwitz equation. Unfortunately, this could give a strict value for the target reproducibility. Therefore, iis decided to use the iis PT data gathered from 2010 to 2021 to estimate a more realistic target reproducibility for the evaluation of the test results. Furthermore, it was decided to use the same target reproducibility for all aromatic amines. The average relative standard deviation over all iis PTs and components for AZO Dyes in Leather/Footwear is 27%. This investigation is summarized in iis memo 2202.

### sample #23505

Benzidine (CAS No. 92-87-5): This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility as derived from its memo 2202.

#### sample #23506

o-Anisidine (CAS No. 90-04-0): This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the target reproducibility as derived from iis memo 2202.

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 are calculated for these aromatic amines. The reported test results are given in appendix 2.

#### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility estimated from the reference 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 the reference method are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
Benzidine	mg/kg	78	42.6	28.3	32.2

Table 1: reproducibility of test on sample #23505

Component	unit	n	average	2.8 * sd	R(target)
o-Anisidine	mg/kg	74	24.6	24.4	18.6

Table 2: reproducibility of test on sample #23506

Without further statistical calculations it can be concluded that the participating laboratories have no difficulties with the determination of Benzidine but have difficulties with the determination of o-Anisidine.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2023 WITH PREVIOUS PTS

	February 2023	February 2022	March 2021	March 2020	March 2019
Number of reporting laboratories	80	96	108	90	117
Number of test results	156	258	212	166	117
Number of statistical outliers	4	3	6	1	3
Percentage of statistical outliers	2.6%	1.2%	2.8%	0.6%	2.6%

Table 3: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTS, see next table.

Component	February 2023	February 2022	March 2021	March 2020	2019-2005	Target
4-Aminodiphenyl					25-45%	27%
Benzidine	24%	29%	21%	29%	20-66%	27%
3,3'-Dimethoxybenzidine					23%	27%
3,3'-Dimethylbenzidine		16%			24-55%	27%
o-Toluidine					37-63%	27%
o-Ansidine	36%			61%		27%
2,4-Xylidine		21%			16-36%	27%
4-Aminoazobenzene			19%			27%

Table 4: development of the uncertainties over the years

Components not listed have not been tested in an iis PT on AZO dyes in Leather/Footwear

The uncertainties observed in this PT are comparable to or better than the uncertainties observed in previous iis PTs.

Samples #23505 and #23506 were used in previous its PTs as sample #17520 in its17A02 and sample #20550 in its20A05 respectively. The averages found in the PTs for these samples are similar. The calculated reproducibility for Benzidine in the 2023 PT is comparable to the calculated reproducibility in the 2017 PT.

The calculated reproducibility for o-Anisidine improved in the 2023 PT compared to the 2020 PT.

		sa	ample #2350	)5	sa	ample #1752	20
Component	unit	n	average	R(calc)	n	average	R(calc)
Benzidine	mg/kg	78	42.6	28.3	126	51.4	29.0

Table 5: comparison of sample #23505 with #17520

		sa	mple #2350	)6	sa	mple #205	50
Component	unit	n	average	R(calc)	n	average	R(calc)
o-Anisidine	mg/kg	74	24.6	24.4	78	27.3	47.0

Table 6: comparison of sample #23506 with #20550

#### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

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

- A majority (about 95%) of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported component.
- About 30% of the participants used the sample as received and about 70% did further cut or further grind the samples prior to analysis.
- About 55% used around 1 grams or more sample intake and about 45% used a sample intake of about 0.5 grams.

As the majority of the group follow the more or less the same analytical procedures no separate statistical analysis has been performed.

#### 5 DISCUSSION

All reporting participants were able to detect Benzidine in sample #23505 and o-Anisidine in sample #23506. The other aromatic amines that were requested in this PT were not detected by all participants, except for one participant.

When the results of this interlaboratory study were compared to the LEATHER STANDARD by OEKO-TEX® and with the similar BlueSign® system substances list or BSSL (see Table 7) it was noticed that not all participants would make an identical decision about the acceptability of the samples for the determined components.

Almost all reporting participants would have rejected sample #23505 for containing too much Benzidine and two participants would have accepted the sample.

Sample #23506 would have been rejected by fifty-eight of the reporting laboratories for containing too much o-Anisidine and sixteen participants would have accepted the sample.

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

Table 7: BlueSign® BSSL and LEATHER STANDARD by OEKO-TEX®

#### 6 CONCLUSION

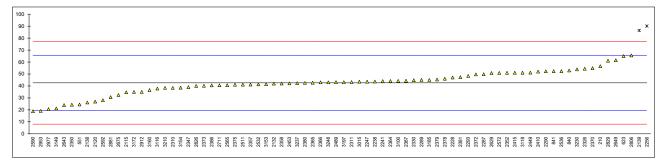
Although it can be concluded that the participants have no problem with the determination of Benzidine and o-Anisidine in the samples of this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation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

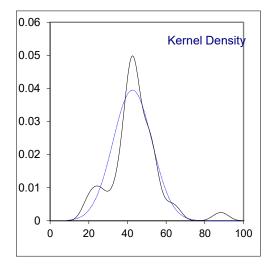
**APPENDIX 1** 

Determination of Benzidine (CAS No. 92-87-5) in sample #23505; results in mg/kg

					sample #23505; results in mg/kg
lab	method	value	mark	` 0,	remarks
210	ISO17234-1	56.53		1.21	
	ISO17234-1	24.4646788		-1.58	
	ISO17234-1	65.017		1.95	
	ISO17234-1	52.9		0.89	
	ISO17234-1	52.40		0.85	
	ISO17234-1	34.75		-0.68	
2120		26.9		-1.37	
	ISO17234-1	41.9437		-0.06	
	ISO17234-1	26.199		-1.43	
	ISO17234-1	86.4	R(0.01)	3.80	
	ISO17234-1	47.05		0.38	
2236					
	ISO17234-1	43.7		0.09	
	ISO17234-1	44.077		0.13	
	ISO17234-1	43.59		0.08	
	ISO17234-1	90.1	R(0.01)	4.12	
2265					
	ISO17234-1	49.8		0.62	
2289	ISO17234-1	45		0.21	
	ISO17234-1	52.3		0.84	
	ISO17234-1	48.4		0.50	
	ISO17234-1	38.4		-0.37	
	ISO17234-1	43.248		0.05	
	ISO17234-1	54.42		1.02	
	ISO17234-1	44.807		0.19	
2347	ISO17234-1	39.03		-0.31	
2350	ISO17234-1	24.2935		-1.59	
	ISO17234-1	51		0.73	
	ISO17234-1	41.2		-0.12	
2358	ISO17234-1	42.00		-0.05	
	ISO17234-1	44.2		0.14	
	ISO17234-1	42.7		0.01	
	ISO17234-1	43		0.03	
	ISO17234-1	44.4		0.15	
2370	ISO17234-1	54.9		1.07	
	ISO17234-1	49.683		0.61	
2373	ISO17234-1	40.19		-0.21	
2375	ISO17234-1	41		-0.14	
2378	GB/T19942	46		0.29	
2379	ISO17234-1	45.392		0.24	
2380	ISO17234-1	42.59		0.00	
2381	ISO17234-1	47.40		0.41	
2386	ISO17234-1	40.473		-0.19	
2410	ISO17234-1	52		0.81	
2415		51.02		0.73	
2449	ISO17234-1	51.16		0.74	
2453	ISO17234-1	42.20		-0.04	
2455					
2489	ISO17234-1	43.2		0.05	
2511	ISO17234-1	41.123		-0.13	
2532	ISO17234-1	41.35		-0.11	
	ISO17234-1	52.418		0.85	
2561					
2565		40.633		-0.17	
2572	ISO17234-1	50.9		0.72	
	ISO17234-1	18.9		-2.06	
	ISO17234-1	28.07		-1.26	
	ISO17234-1	50.8		0.71	
	ISO17234-1	23.926		-1.62	
	ISO17234-1	61.6		1.65	
2675		32.451		-0.88	
	ISO17234-1	40.6		-0.18	
	ISO17234-1	65.542		1.99	
	ISO17234-1	40		-0.23	
	ISO17234-1	61.12		1.61	
2881		30.66		-1.04	
	ISO17234-1	35.05		-0.66	
	ISO17234-1	19.102		-2.04	
	ISO17234-1	20.61		-1.91	
	ISO17234-1	43.54		0.08	
	ISO17234-1	44.259		0.14	
	ISO17234-1	37.8		-0.42	
	ISO17234-1	51.0860		0.73	
	ISO17234-1	21.2		-1.86	
	ISO17234-1	41.5		-0.10	
3,00		: :: <del></del>		3.10	

Ī	lab	method	value	mark	z(targ)	remarks
-	3154	ISO17234-1	38.5		-0.36	
	3160	ISO17234-1	36.53		-0.53	
	3172	ISO17234-1	35.005		-0.66	
	3185	ISO17234-1	45.004		0.21	
	3197	ISO17234-1	43.2		0.05	
	3210		38.39		-0.37	
	3230	In house	53.8904		0.98	
	3237	ISO17234-1	42.35		-0.02	
	3248	ISO17234-1	43		0.03	
		normality	OK			
		n	78			
		outliers	2			
		mean (n)	42.6287			
		st.dev. (n)	10.10048	RSD=24%		
		R(calc.)	28.2813	.102 2170		
		st.dev.(iis memo 2202)	11.50974			
		R(iis memo 2202)	32.2273			

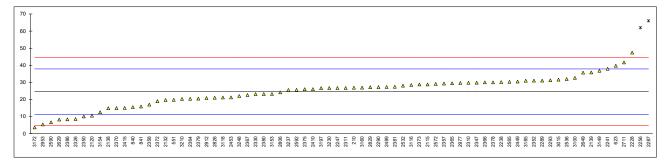


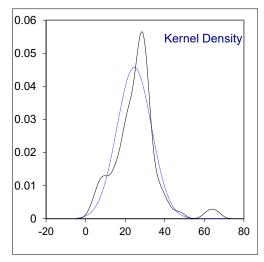


## Determination of o-Anisidine (CAS No. 90-04-0) in sample #23506; results in mg/kg

lab	method	value	mark	z(targ)	remarks
	ISO17234-1	26.83		0.34	
551		19.8019802		-0.72	
	ISO17234-1	39.746		2.28	
	ISO17234-1	15.5		-1.37	
841	ISO17234-1	15.836		-1.32	
2115	ISO17234-1	28.78		0.63	
2120	ISO17234-1	10.5		-2.12	
2132	ISO17234-1	19.6501		-0.74	
	ISO17234-1	14.890		-1.46	
2139	ISO17234-1	35.8		1.69	
2228	ISO17234-1	47.41		3.44	
2236	10017254-1				
	10017004 1				
	ISO17234-1	30.2		0.85	
2241		37.940		2.01	
	ISO17234-1	26.73		0.32	
2256		62.02	R(0.01)	5.64	
2265					
2287	ISO17234-1	66.0	R(0.01)	6.24	
2289	ISO17234-1	31	, ,	0.97	
2290	ISO17234-1	27.2		0.39	
2293		31.24		1.00	
	ISO17234-1	29.74		0.78	
2311					
	ISO17234-1	26.733		0.32	
	ISO17234-1	8.52		-2.42	
	ISO17234-1	23.120		-0.22	
	GB/T19942	29.79		0.78	
	ISO17234-1	10.025		-2.19	
2352	ISO17234-1	31		0.97	
	ISO17234-1	29.3		0.71	
	ISO17234-1	16.98		-1.15	
	ISO17234-1	20.4		-0.63	
	ISO17234-1	29.5		0.74	
	ISO17234-1	30		0.82	
	ISO17234-1	22.6			
				-0.30	
	ISO17234-1	14.93		-1.45	
	ISO17234-1	19		-0.84	
	ISO17234-1	28.76		0.63	
	ISO17234-1	26		0.21	
2378	GB/T19942	30		0.82	
2379	ISO17234-1	20.466		-0.62	
2380	ISO17234-1	23.17		-0.21	
2381	ISO17234-1	27.43		0.43	
	ISO17234-1	8.336		-2.45	
	ISO17234-1	26		0.21	
2415	100172041	15.04		-1.44	
	10017024 1				
	ISO17234-1	30.45		0.88	
	ISO17234-1	21.19		-0.51	
2455					
	ISO17234-1	27.29		0.41	
2511					
2532	ISO17234-1	28.1		0.53	
2536	ISO17234-1	31.952		1.11	
2561					
2565		30.330		0.87	
2572		29.1		0.68	
	ISO17234-1	6.6		-2.71	
	ISO17234-1	25.62		0.16	
	ISO17234-1	8.2		-2.47	
	15017254-1				
2643	100470044			4.00	
	ISO17234-1	35.7		1.68	
2675		<20			
	ISO17234-1	41.65		2.57	
2806	ISO17234-1	24.133		-0.07	
	ISO17234-1	21		-0.54	
	ISO17234-1	27.18		0.39	
2881					
	ISO17234-1	20.81		-0.57	
	ISO17234-1	5.31		-2.90	
	10017204-1				
2977	10047004 4	29.66		0.77	
	ISO17234-1	31.59		1.06	
	ISO17234-1	32.572		1.20	
	ISO17234-1	28.5		0.59	
3118	ISO17234-1	21.1372		-0.52	
3149	ISO17234-1	36.8		1.84	
	ISO17234-1	23.2		-0.21	

lab	method	value	mark z(	targ)	remarks
3154	ISO17234-1	12.498		-1.82	
3160	ISO17234-1	26.87		0.34	
3172	ISO17234-1	3.6036		-3.16	
3185	ISO17234-1	30.959		0.96	
3197	ISO17234-1	26.6		0.30	
3210	In house	20.27		-0.65	
3230	In house	26.7274		0.32	
3237	ISO17234-1	25.52		0.14	
3248	ISO17234-1	22		-0.39	
	normality n outliers mean (n) st.dev. (n) R(calc.) st.dev.(iis memo 2202) R(iis memo 2202)	OK 74 2 24.5813 8.72664 24.4346 6.63695 18.5835	RSD=36%		





#### **APPENDIX 2**

Other reported aromatic amines; results in mg/kg

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

oAAT = o-Aminoazotoluene (CASno. 97-56-3)

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

SUM = Sum of o-Aminoazotoluene and o-Toluidine

#### sample #23505

lab	4AD	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB
210									
551									
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected				
840	not detected	not detected	not detected	not detected	not detected				
841	not determ.	not determ.	not determ.	not determ.	not determ.				
2115									
2120	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5
2138	ND	ND	ND	ND	ND	ND	ND	ND	ND
2139									
2228	not detected	not detected	not detected	not determ.	not determ.	not detected	not detected	not detected	not detected
2236									
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5
2241	<5	<5	<5	<5	<5	<5	<5	<5	<5
2247	Not detected		Not detected						Not detected
2256									
2265 2287	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2289	<5.0 	<5.0 	<5.0 	<5.0 					<5.0 
2299	<5	<5	<5	<5	<b></b> <5	<b></b> <5	<b></b> <5	<b></b> <5	<5
2293	0	0	0	0	0	0	0	0	0
2310	not detected	•	not detected	-	-	-	-	-	-
2311			Not Detected						
2326	ND	ND	ND	ND	ND	ND	ND	ND	ND
2330			Not Detected						
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2352									
2357									
2358	not detected	not detected	not detected	not detected	not detected				
2364									
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366									
2367	ND	ND	ND	ND	ND	ND	ND	ND	ND
2370	<5	<5	<5	<5	<5	<5	<5	<5	<5
2372	not detected	not detected	not detected	not detected	not detected				

lab	4AD	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB
2373	<5	<5	<5	<5	<5	<5	<5	<5	<5
2375									
2378	<5	<5	<5	<5	<5	<5	<5	<5	<5
2379		Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	
2380	<5	<5	<5	<5	<5	<5	<5	<5	<5
2381									
2386	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2410									
2415	not detected	not detected	not detected	not detected	not detected				
2449									
2453									
2455									
2489	Not detected	Not detected	Not detected	Not detected	Not detected				
2511									
2532	Not detected	Not detected	Not detected	Not detected	Not detected				
2536	Not detected	Not detected	Not detected	Not detected	Not detected				
2561									
2565	<5	<5	<5	<5	<5	<5	<5	<5	<5
2572									
2590									
2592									
2629	<5 mg/kg	<5 mg/kg	<5 mg/kg	<5 mg/kg	<5 mg/kg				
2643									
2649									
2675	<20	<20	<20	<20	<20	<20	35.582	<20	<20
2711	<5	<5	<5	<5	<5	<5	<5	<5	6.6
2806	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2826		Not detected						Not detected	
2829	not detected	not detected	not detected		not detected	not detected	not detected		
2881						36.47			
2912									
2953									
2977	<5,0 <5	<5,0 <5	<5,0 <5	<5,0 <5	<5,0 <5	<5,0 <5	<5,0 <5	<5,0 <5	<5,0
3015			<5 <5	<5 <5	<5 <5			<5 <5	<5 <5
3100	<5	<5	< 5			<5	<5	< 5	
3116									
3118	<5	<5	<5	<5	<5	<5	<5	<5	<5
3149	 < 5	 < 5	 < 5	 < 5					
3153	-	-	-	-	-	-	-	-	< 5
3154	not detected	not detected	not detected not detected		not detected				
3160 3172	not detected < 1	< 1	< 1	< 1	not detected < 1				
3185	< i	<51 <51	< 1 <5	< 1 <5	< 1 <5	< 1 <5	< 5	< i	< 1 < 5
3197	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5
3210	~0	~0	~0	~0	~0	~0	~5	~0	<b>~</b> J
3230	not detected	not detected	not detected	not detected	not detected				
3237	not detected	not detected	not detected	not detected	not detected				
3248									
3240									

sample #23505 -continued: abbreviations components explained at start of appendix 2

	le #23505 -c		•					
lab	DMB	DDDM	рС	DDM	DDE	DDS	24DAT	TMA
210 551								
623	Not Detected							
840	not detected							
841					not determined			
2115	 - 5							
2120 2132	< 5 <5							
2132	ND							
2139								
2228	not detected							
2236								
2238 2241	<5 <5							
2247	Not detected							
2256								
2265								
2287	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2289 2290	 <5	 <5	 <5	 <5	 <5	 <5	<b></b> <5	 <5
2293	0	0	0	0	0	0	0	0
2310	not detected							
2311	Not Detected							
2326 2330	ND Not Detected							
2347	<5	<5	<5	<5	<5	<5	<5	<5
2350	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2352								
2357								
2358 2364	not detected							
2365	<5	<5	<5	<5	<5	<5	<5	<5
2366								
2367	ND							
2370	<5	<5	<5	<5	<5	<5	<5	<5
2372 2373	not detected <5	not detected <5	not detected <5	not detected <5	not detected <5	not detected <5	not detected <5	not detected <5
2375								
2378	<5	<5	<5	<5	<5	<5	<5	<5
2379	Not detected							
2380	<5	<5	<5	<5	<5	<5	<5	<5
2381 2386	 < 5	< 5	 < 5	 < 5				
2410								
2415	not detected							
2449								
2453 2455								
2489	Not detected							
2511								
2532	Not detected							
2536 2561	Not detected							
2565	 <5	 <5	 <5	 <5	<5	<5	 <5	<b></b> <5
2572								
2590								
2592 2629	 <5 mg/kg	 <5 malka	 <5 mg/kg	 <5 ma/ka	 <5 mg/kg	 <5 mg/kg	 <5 mg/kg	 <5 mg/kg
2643	<5 mg/kg	<5 mg/kg 						
2649								
2675	<20	<20	<20	<20	<20	<20	<20	<20
2711	<5	<5	<5	<5	<5	<5	<5	<5
2806 2826	< 5 Not detected							
2829	not detected							
2881								
2912								
2953 2977	 <5,0	<5.0	 <5,0	<5,0	<5,0	<5,0	<5,0	 <5,0
3015	<5,0 <5							
3100	<5	<5	<5	<5	<5	<5	<5	<5
3116								
3118	<5	<5	<5	<5	<5	<5	<5	<5
3149 3153	 < 5							
3154	not detected							
5.0→	40.00.00	30.00.00	40.00.00	40.00.00	40.00.00	40.00.00	40.00.00	30.00.00

lab	DMB	DDDM	pC	DDM	DDE	DDS	24DAT	TMA
3160	not detected							
3172	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
3185	<5	<5	<5	<5	<5	<5	<5	<5
3197	<5	<5	<5	<5	<5	<5	<5	<5
3210								
3230	not detected							
3237								
3248								

sample #23505 -continued; abbreviations components explained at start of appendix 2

	•	-	obreviations	-		•		CLIM
lab 210	oA	24X	25X	26X	TX	oAAT	оТ	SUM
210 551								
623	Not Detected							
840	not detected							
841	not determ.							
2115								
2120	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2132	<5	<5	N.A.	<5	N.A.	<5	<5	N.A.
2138	ND							
2139								
2228 2236	not detected	no detected	not determ.	not detected	not determ.	not determ.	not detected	not determ.
2238	<5	<5	<5	 <5	 <5	 <5	<5	<5
2241	<5	<5	<5	<5	<5	<5	<5	<5
2247	Not detected							
2256								
2265								
2287	<5.0	<5.0		<5.0		<5.0	<5.0	<10
2289								
2290	<5 0							
2293 2310	0 not detected							
2311	Not Detected							
2326	ND							
2330	Not Detected	Not Detected	Not Analyzed	Not Detected				
2347	<5	<5	out of capability	<5	out of capability	<5	<5	<5
2350	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2352								
2357								
2358 2364	not detected							
2365	<5	<5	<5	<5	<5	<5	<5	<5
2366								
2367	ND				ND	ND	ND	ND
2370	<5	<5	<5	<5	<5	<5	<5	<5
2372	not detected							
2373	<5	<5	not applicable	<5	<5	<5	<5	<5
2375								
2378	<5	<5	no capacity	<5	no capacity	<5	<5	<5
2379	Not detected	Not detected	Not Analyzed	Not detected	Not Analyzed	Not detected	Not detected	Not detected
2380 2381	<5 							
2386	< 5	< 5		< 5	< 10	< 5	< 5	< 10
2410								
2415	not detected							
2449								
2453								
2455		 Ni-6 d-66d		Not detected				
2489	Not detected							
2511 2532	Not detected	Not detected		Not detected				
2536	Not detected							
2561								
2565	<5	<5		<5		<5	<5	<5
2572								
2590								
2592 2629	 <5 mg/kg							
2643	~5 mg/kg 	<5 mg/kg	~5 mg/kg	~5 mg/kg	~5 mg/kg 	~5 mg/kg	~5 mg/kg 	<5 mg/kg
2649								
2675	<20	22.546	not determined	<20	not determined	<20	121.421	121.421
2711	42	<5	<5	<5	<5	<5	<5	<5
2806	< 5	< 5		< 5		< 5	< 5	< 5
2826	Not detected	Not detected	Not analyzed	Not detected	Not analyzed	Not detected	Not detected	Not detected
2829	not detected							
2881 2912								
2953								
2977	<5.0	<5,0	<5.0	<5.0	<5.0	<5,0	<5.0	<5,0
3015	<5	<5	<5	<5 <5	<5	<5	<5 <5	<5
3100	<5	<5	<5	<5	<5	<5	<5	<5
3116								
3118	<5	<5	<5	<5	<5	<5	<5	<5
3149								
3153	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3154	not detected	not detected	not detected	not detected		not detected	not detected	

lab	οA	24X	25X	26X	TX	oAAT	оТ	SUM
3160	not detected							
3172	< 1	< 1		< 1		< 1	< 1	
3185	<5	<5		<5		<5	<5	<5
3197	<5	<5	<5	<5	<5	<5	<5	<5
3210								
3230	not detected	not analysed	not analysed	not detected	not analysed	not detected	not detected	not detected
3237								
3248								

sample #23506; abbreviations components explained at start of appendix 2

	le #23506;	•			•				
lab 210	4AD	В	4CoT	2NA	ANT	4CA	DAA	DADM	DCB
210 551									
623	Not Detected								
840	not detected					not detected		not detected	
841	not determ.								
2115									
2120	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5
2138 2139	ND	ND	ND	ND	ND	ND 	ND 	ND 	ND
2228	not detected	not detected	not detected	not detected	not determ.	not determ.	not detected	not detected	not detected
2236									
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5
2241	<5	<5	<5	<5	<5	<5	<5	<5	<5
2247	Not detected								
2256 2265									
2287	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2289									
2290	<5	<5	<5	<5	<5	<5	<5	<5	<5
2293	0	0	0	0	0	0	0	0	0
2310								not detected	
2311 2326	Not Detected ND			Not Detected ND		Not Detected ND		Not Detected	Not Detected ND
2320		ND Not Detected	ND Not Detected		ND Not Detected		ND Not Detected	ND Not Detected	
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2352									
2357	not detected								
2358 2364	not detected								
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366									
2367	ND								
2370	<5	<5	<5	<5	<5	<5	<5	<5	<5
2372 2373	not detected <5	not detected <5	not detected <5	not detected <5	not detected	not detected <5	not detected <5	not detected <5	not detected
2375									
2378	<5	<5	<5	<5	<5	<5	<5	<5	<5
2379	Not detected								
2380	<5	<5	<5	<5	<5	<5	<5	<5	<5
2381 2386	 < 5	< 5	< 5	< 5	< 5	< 5	< 5	 < 5	 < 5
2410									
2415	not detected								
2449									
2453 2455									
2433	Not detected								
2511									
2532	Not detected								
2536								Not detected	
2561 2565	 <5	<b></b> <5	<b></b> <5	<b></b> <5	<b></b> <5	 <5	 <5	 <5	<b></b> <5
2572	<5 								
2590									
2592									
2629	< 5mg/kg								
2643 2649									
2675	<20	<20	<20	<20	<20	<20	<20	<20	<20
2711	<5	<5	<5	<5	<5	<5	<5	<5	<5
2806	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2826								Not detected	
2829		not detected	not detected	not detected			not detected		
2881 2912	21.20								30.80
2953									
2977	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0
3015	<5	<5	<5	<5	<5	<5	<5	<5	<5
3100	<5	<5	<5	<5	<5	<5	<5	<5	<5
3116 3118	 <5								
3149	<5 	<b></b>	<5 						
3153	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3154	not detected								

lab	4AD	В	4CoT	2NA	ANT	4CA	DAA	DADM	DCB
3160	not detected								
3172	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
3185	<5	<5	<5	<5	<5	<5	<5	<5	<5
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5
3210									
3230	not detected								
3237									
3248									

sample #23506 -continued; abbreviations components explained at start of appendix 2

	*	•	bbreviations		•		•	0.45.47
lab	DMoxB	DMB	DDDM	pC	DDM	DDE	DDS	24DAT
210 551								
	Not Detected							
840	not detected							
841	not determ.							
2115								
2120	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2132	<5	<5	<5	<5	<5	<5	<5	<5
2138	ND							
2139 2228	not detected							
2236	not detected		not detected	not detected				
2238	<5	<5	<5	<5	<5	<5	<5	<5
2241	<5	<5	<5	<5	<5	<5	<5	<5
2247	Not detected							
2256								
2265 2287	 <5.0	 <5.0	<5.0	<5.0	 <5.0	 <5.0	<5.0	<5.0
2289	<5.0 							
2290	<5	<5	<5	<5	<5	<5	<5	<5
2293	0	0	0	0	0	0	0	0
2310	not detected							
2311		Not Detected						
2326 2330	ND Not Detected							
2330	Not Detected <5							
2350	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2352								
2357								
2358	not detected							
2364	<5	<5	<5	<5	<5	<5	<5	<5
2365 2366								
2367	ND							
2370	<5	<5	<5	<5	<5	<5	<5	<5
2372	not detected							
2373	<5	<5	<5	<5	<5	<5	<5	<5
2375	 /F		 / F					
2378 2379	<5 Not detected							
2380	<5	<5	<5	<5	<5	<5	<5	<5
2381								
2386	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2410								
2415 2449	not detected							
2449								
2455								
2489	Not detected							
2511								
2532	Not detected							
2536 2561	Not detected							
2565	<5	<5	<5	<5	<5	<5	<5	<5
2572								
2590								
2592								
2629 2643	< 5mg/kg 	< 5mg/kg	< 5mg/kg 	< 5mg/kg 				
2649								
2675	71.265	<20	<20	<20	<20	<20	<20	<20
2711	<5	<5	<5	<5	<5	<5	<5	<5
2806	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2826	Not detected							
2829 2881	not detected	not detected	not detected	not detected 25.30	not detected	not detected 26.20	not detected	not detected
2912				23.30		20.20		
2953								
2977	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0
3015	<5	<5	<5	<5	<5	<5	<5	<5
3100	<5	<5	<5	<5	<5	<5	<5	<5
3116 3118	 <5							
3149	~5 		<u></u>					
3153	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3154	not detected							

lab	DMoxB	DMB	DDDM	рС	DDM	DDE	DDS	24DAT
3160	not detected							
3172	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
3185	<5	<5	<5	<5	<5	<5	<5	<5
3197	<5	<5	<5	<5	<5	<5	<5	<5
3210								
3230	not detected							
3237								
3248								

sample #23506 -continued; abbreviations components explained at start of appendix 2

	*	•	abbreviations		•	•	•	0.00
lab	TMA	24X	25X	26X	TX	oAAT	оТ	SUM
210 551								
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
841	not determ.	not determ.	not determ.	not determ.	not determ.	not determ.	not determ.	not determ.
2115								
2120	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2132	<5 NB	<5	N.A.	<5 NB	N.A.	<5 NB	<5 NB	N.A.
2138	ND 	ND	ND	ND 	ND	ND	ND 	ND
2139 2228	not detected	not detected	not determ.	not detected	not determ.	not determ.	not detected	not determ.
2236								
2238	<5	<5	<5	<5	<5	<5	<5	<5
2241	<5	<5	<5	<5	<5	<5	<5	<5
2247	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2256								
2265 2287	 <5.0	<5.0		<5.0		<5.0	<5.0	 <10
2289								
2290	<5	<5	<5	<5	<5	<5	<5	<5
2293	0	0	0	0	0	0	0	0
2310	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2326 2330	ND Not Detected	ND Not Detected	ND Not Analyzed	ND Not Detected	ND Not Detected	ND Not Detected	ND Not Detected	ND Not Detected
2347	<5	<5	Not Analyzed out of capability		out of capability		<5	<5
2350	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2352								
2357								
2358 2364	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2365	<5	<5	<5	<5	<5	<5	<5	<5
2366								
2367	ND				ND	ND	ND	ND
2370	<5	<5	<5	<5	<5	<5	<5	<5
2372	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2373	<5	<5	not applicable	<5	<5	<5	<5	<5
2375								
2378	<5	<5	no capacity	<5	no capacity	<5	<5	<5
2379	Not detected	Not detected	Not Analyzed	Not detected	Not Analyzed	Not detected	Not detected	Not detected
2380	<5	<5	<5	<5	<5	<5	<5	<5
2381 2386	 < 5	 < 5		 < 5	 < 10	< 5	 < 5	 < 10
2410								
2415	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2449								
2453 2455								
2489	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2511								
2532	Not detected	Not detected		Not detected	Not detected	Not detected	Not detected	Not detected
2536 2561	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2565	<5	 <5		<5		<5	<b></b> <5	<5
2572								
2590								
2592	 < 5ma/ka				 - Emali		 < Emalka	 < Emalka
2629 2643	< 5mg/kg 	< 5mg/kg 	< 5mg/kg 	< 5mg/kg 	< 5mg/kg 	< 5mg/kg 	< 5mg/kg 	< 5mg/kg 
2649								
2675	<20	<20	not determ.	<20	<20	<20	<20	<20
2711	<5	<5	<5	<5	<5	<5	<5	<5
2806 2826	< 5 Not detected	< 5 Not detected	Not analyzed	< 5 Not detected	Not analyzed	< 5 Not detected	< 5 Not detected	Not detected
2829	Not detected not detected	Not detected not detected	Not analyzed not detected	Not detected not detected	not detected	Not detected not detected	Not detected not detected	Not detected not detected
2881								
2912								
2953	 <5.0	 <5.0	 <5.0	 <5.0	 -5 0	 <5.0	 <5.0	 <5.0
2977 3015	<5,0 <5	<5,0 <5	<5,0 <5	<5,0 <5	<5,0 <5	<5,0 <5	<5,0 <5	<5,0 <5
3100	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5
3116								
3118	<5	<5	<5	<5	<5	<5	<5	<5
3149								
3153	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5

lab	TMA	24X	25X	26X	TX	oAAT	оТ	SUM
3154	not detected	not detected	not detected	not detected		not detected	not detected	
3160	not detected							
3172	< 1	< 1		< 1		< 1	< 1	
3185	<5	<5		<5		<5	<5	<5
3197	<5	<5	<5	<5	<5	<5	<5	<5
3210								
3230	not detected	not analysed	not analysed	not detected	not analysed	not detected	not detected	not detected
3237								
3248								

## **APPENDIX 3 Analytical details**

lab	ISO/IEC17025 accredited	sample intake (g)	sample pre-treatment
210	Yes	Further cut	1g
551	Yes	Further cut	1g
623	Yes	Further cut	1
840	Yes	Further cut	0.5
841	Yes	Further cut	0.5 grams
2115	Yes	Used as received	0.5 g
2120 2132	No Yes	Used as received Further cut	1 g
2132	Yes	Used as received	1g about 0.5g
2130	Yes	Further cut	0.5 g
2228	Yes	#23505 further cut, #23506 as received	0.5 g
2236			0.5 g
2238	Yes	Used as received	0.5g
2241	Yes	Further cut	0.5g.
2247	Yes	Further cut	2gm
2256	Yes	Further cut	0.5016g & 0.5015g
2265			
2287	Yes	Further cut	0.5g
2289	Yes	Further cut	1.0g
2290	Yes		Ç
2293	Yes	Further cut	1 gram
2310	Yes	Further cut	1
2311	Yes	Further cut	0.5
2326	Yes	Further cut	A- 23505 = 0.5081 G B - 23506 = 0.5048 G
2330	Yes	Further cut	1 g
2347	Yes	Further cut	1g
2350	Yes	Further cut	Benzidine 1.0085 g o-Anisidine 0.9685 g
2352	Yes	Further cut	0.5g
2357		<del></del>	
2358	Yes	Further cut	1
2364	Yes	Used as received	#23505:m=0.4967g, #23506:m=0.5011g
2365	Yes	Further cut	0.5g
2366	Yes	Further cut	0.5g
2367	Yes	Used as received	0.50
2370	Yes	Further cut	0.5g
2372 2373	Yes Yes	Used as received Further cut	1g
2375	Yes	Further cut	0.5g 1 gram
2378	Yes	Further cut	1g
2379	Yes	Further cut	1 gram
2380	Yes	Further cut	1.0 g
2381	Yes	#23505 Further cut, #23505 as received	0.50 gm per trial.
2386	Yes	#23505 Further cut, #23505 as received	0.5 g
2410	Yes	Used as received	0.5 g
2415	Yes	Further cut	0.5
2449	Yes	Further cut	1.0 gram
2453	No	Further grinded	±1g
2455			
2489	Yes	Further cut	0.5025g/0.5016g
2511	Yes	Further cut	1 gram
2532	Yes	Further cut	0.5 gram
2536	Yes	Used as received	1.0015
2561	<del></del>		
2565	Yes	Used as received	1g
2572	Yes	Lland on received	10
2590	Yes	Used as received	1g
2592 2629	Yes Yes	Used as received Further cut	1 gr 1.0 gram
2629	Yes	Used as received	0.5~1.0 g
2649	Yes	Further grinded	2 grams
2675	Yes	Further cut	1g
2711	No	Further cut	19
2806	Yes	Used as received	CIRCA 1 grammo
2826	Yes	Used as received	0.5
2829	No	Further cut	1 gr
2881			×
2912	Yes	Used as received	0.5 g
2953	Yes	Further cut	1
2977	No	Used as received	1g
3015	Yes	Used as received	1.0
3100	Yes	Further cut	1.0076g
3116	Yes	Used as received	1g
3118	Yes	Further cut	0.5
3149	 V	 	0.5
3153	Yes	Further cut	0.5 gram

lab	ISO/IEC17025 accredited	sample intake (g)	sample pre-treatment
3154	Yes	Used as received	1 g
3160	Yes	Further cut	0,75
3172	Yes		
3185	Yes	#23505 Further cut, #23505 as received	1g
3197	Yes	Further cut	0,5g
3210	Yes	Further cut	1
3230	Yes	Further cut	1 g
3237	Yes	Further cut	0,5 gram
3248	Yes	Used as received	1g

#### **APPENDIX 4**

## Number of participants per country

- 4 labs in BANGLADESH
- 1 lab in BRAZIL
- 1 lab in CAMBODIA
- 1 lab in FRANCE
- 5 labs in GERMANY
- 1 lab in GUATEMALA
- 6 labs in HONG KONG
- 5 labs in INDIA
- 2 labs in INDONESIA
- 10 labs in ITALY
- 2 labs in JAPAN
- 5 labs in KOREA, Republic of
- 1 lab in MAURITIUS
- 1 lab in MEXICO
- 1 lab in MOROCCO
- 16 labs in P.R. of CHINA
- 2 labs in PAKISTAN
- 1 lab in POLAND
- 2 labs in PORTUGAL
- 1 lab in SPAIN
- 2 labs in TAIWAN
- 1 lab in THAILAND
- 1 lab in TUNISIA
- 3 labs in TURKEY
- 2 labs in U.S.A.
- 1 lab in UNITED KINGDOM
- 6 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 D(0.01) = outlier in Grubbs' outlier test D(0.05) = straggler in Grubbs' outlier test D(0.05) = outlier in Double Grubbs' outlier test D(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

E = calculation difference between reported test result and result calculated by iis

W = test result withdrawn on request of participant ex = test result excluded from statistical evaluation

n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

f+? = possibly a false positive test result? f-? = possibly a false negative test result?

#### Literature

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