# Results of Proficiency Test AZO Dyes in Textile March 2020

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester

Correctors: ing. A.S. Noordman-de Neef & ing. R.J. Starink

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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 from AZO Dyes in Textile. During the annual proficiency testing program 2019/2020 it was decided to continue the proficiency test for the analysis of banned aromatic amines derived from AZO Dyes in Textile.

In this interlaboratory study 162 laboratories in 30 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of this 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 textile samples both positive on banned aromatic amines derived from AZO Dyes of approximately 3 grams each. The first sample is a pink polyester textile labelled #20545 and the second sample is a blue cotton textile labelled #20546. 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, 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 pink polyester was selected which was dyed with with Solvent Red 24 by a third party. A part of this batch was cut in small pieces. After homogenization the batch was divided over 200 subsamples in small bags of approximately 3 grams each and labelled #20545.

The homogeneity of the subsamples was checked by the determination of the sum of o-Aminoazotoluene and o-Toluidine on eight stratified randomly selected subsamples (see for more details chapter 4).

	Sum of o-Aminoazotoluene and o-Toluidine in mg/kg
Sample #20545-1	205.0
sample #20545-2	202.6
sample #20545-3	216.4
sample #20545-4	207.3
sample #20545-5	199.1
sample #20545-6	211.7
sample #20545-7	206.4
sample #20545-8	200.8

Table 1: homogeneity test results of subsamples #20545

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.

	Sum of o-Aminoazotoluene and o-Toluidine in mg/kg
r (observed)	16.0
reference test method	ISO14362-1:17
0.3 * R (ref. test method)	37.8

Table 2: evaluation of the repeatability of subsamples #20545

The calculated repeatability is 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 blue cotton was selected which was dyed with Direct Blue 15. A part of this batch was cut in small pieces. After homogenization the batch was divided over 200 subsamples in small bags of approximately 3 grams each and labelled #20546. The homogeneity of the subsamples was checked by the determination of 3,3'-Dimethoxybenzidine on seven stratified randomly selected subsamples.

	3,3'-Dimethoxybenzidine in mg/kg
Sample #20546-1	98.6
sample #20546-2	97.7
sample #20546-3	104.4
sample #20546-4	96.8
sample #20546-5	102.0
sample #20546-6	99.9
sample #20546-7	97.4

Table 3: homogeneity test results of subsamples #20546

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.

	3,3'-Dimethoxybenzidine in mg/kg
r (observed)	7.8
reference test method	ISO14362-1:17
0.3 * R (ref. test method)	10.5

Table 4: evaluation of the repeatability of subsamples #20546

The calculated repeatability is 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 #20545 and one sample labelled #20546 was sent on March 4, 2020.

# 2.5 ANALYZES

The participants were asked to determine on sample #20545 and on sample #20546 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. 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' 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 that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

### 3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

#### 3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, 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 these with a factor of 2.8.

# 3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported 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, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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  $z_{\text{(target)}}$  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 thirty-six 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 one hundred and twenty-nine participants reported 380 numerical test results. Observed were 2 outlying test results, which is 0.5% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

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

#### 4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the 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 test methods are also in the tables in appendix 1 together with the original data. The abbreviations used in these tables are explained in appendix 5.

For the determination of Aromatic Amines derived from AZO colorants the ISO14362 method is considered to be the official test method. Two versions of ISO14362 were published in 2017. Part 1 of ISO14362 describes a method to detect certain aromatic amines that are banned. Part 3 of ISO14362 describes a method to detect 4-Aminoazobenzene. In both samples, 4-Aminoazobenzene was not present.

Regretfully, not for all listed Aromatic Amines precision data are available in ISO14362-1:17. For sample #20545, only a precision statement is present for o-Toluidine. This has been used for the statistical evaluation of the sum of o-Aminoazotoluene and o-Toluidine. Fortunately, for the component 3,3'-Dimethoxybenzidine, which is present in sample #20546 a precision statement is mentioned.

# Sample #20545

The dye used in sample #20545 can be reduced to o-Aminoazotoluene. Test method ISO14362 describes that o-Aminoazotoluene can be further reduced to o-Toluidine. This may cause uncontrolled variation of levels of o-Aminoazotoluene and o-Toluidine whereas the sum of both analytes should be the constant. Therefore, iis decided to perform the statistical evaluation only on the sum of the two analytes. In appendix 1 the reported test results of o-Aminoazotoluene and o-Toluidine whereas the sum of both analytes are given in one table for convenience reasons. The order of the report form will be adapted as well for coming proficiency tests.

<u>Sum of o-Aminoazotoluene and o-Toluidine</u>: The determination of the sum of these aromatic amines at a concentration level of 90 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 ISO14362-1:2017.

## Sample #20546

3,3'-Dimethoxybenzidine (CASno. 119-90-4): The determination of this aromatic amine at a concentration level of 98 mg/kg was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO14362-1:2017.

#### 4.2 Performance evaluation for the group of Laboratories

A comparison has been made between the reproducibility as declared by the relevant reference test method and 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 (or estimated) from the official test method ISO14362-1 are compared in the next two tables.

Component	unit	n	average	2.8 * sd	R(lit)
Sum of o-Aminoazotoluene and o-Toluidine	mg/kg	122	90.4	89.2	55.2

Table 5: reproducibility of the aromatic amine in textile sample #20545

Component	unit	n	average	2.8 * sd	R(lit)
3,3'-Dimethoxybenzidine	mg/kg	125	98.5	30.9	34.6

Table 6: reproducibility of the aromatic amine in textile sample #20546

Without further statistical calculations, it can be concluded that the group of participating laboratories has no difficulties with the analyzes of 3,3'-Dimethoxybenzidine but have some difficulties with the analyzes of o-Aminoazobenze and o-Toluidine at the investigated concentration levels. See also the discussion in paragraphs 4.1 and 5.

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

	March 2020	March 2019	March 2018	February 2017	February 2016
Number of reporting laboratories	126	165	171	164	161
Number of test results	380	299	496	770	486
Number of statistical outliers	2	11	3	18	15
Percentage statistical outliers	0.5%	3.7%	0.6%	2.3%	3.1%

Table 7: comparison to 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 against the requirements of the respective reference test methods as relative standard deviations (uncertainties). The conclusions are given the following table.

Parameter	March 2020	March 2019	March 2018	Feb. 2017	Feb. 2016	2004 - 2015	target
4-Aminodiphenyl	n.e.	n.e.	n.e.	n.e.	n.e.	18-36%	16%
Benzidine	n.e.	n.e.	12%	n.e.	17-18%	15-35%	14%
4-Chloro-o-toluidine	n.e.	n.e.	n.e.	n.e.	n.e.	24%	16%
2-Naphtylamine	n.e.	n.e.	n.e.	n.e.	n.e.	27-41%	16%

Parameter	March 2020	March 2019	March 2018	Feb. 2017	Feb. 2016	2004 - 2015	target
4-Chloroaniline	n.e.	n.e.	n.e.	n.e.	n.e.	27%	16%
2,4-Diaminoanisol	n.e.	n.e.	n.e.	n.e.	n.e.	24-52%	16%
4,4'-Diaminodiphenylmethane	n.e.	n.e.	n.e.	n.e.	n.e.	21%	15%
3,3'-Dimethoxybenzidine	11%	11%	12%	17%	16%	16-31%	13%
3,3'-Dimethylbenzidine	n.e.	n.e.	n.e.	36%	n.e.	15-32%	18%
4,4'-Diamino-3,3'- dichlorodiphenylmethane	n.e.	n.e.	n.e.	n.e.	n.e.	20-35%	16%
4,4'-Diaminodiphenylether	n.e.	n.e.	n.e.	n.e.	n.e.	15%	16%
4,4'-Diaminodiphenylsulfide	n.e.	n.e.	n.e.	n.e.	n.e.	18-26%	16%
4,4'-Methyl-bis(2-chloro- aniline)	n.e.	n.e.	n.e.	n.e.	n.e.	43%	22%
o-Aminoazotoluene *)	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	22%
o-Toluidine *)	n.e.	n.e.	n.e.	n.e.	n.e.	19-38%	22%
Sum of o-aminoazotoluene and o-Toluidine *)	35%	n.e.	n.e.	n.e.	n.e.	34%	22%
2,4-Xylidine	n.e.	26%	n.e.	19%	n.e.	n.e.	16%
4-Aminoazobenzene	n.e.	n.e.	30%	n.e.	n.e.	n.e.	27%

Table 8: development of uncertainties of aromatic amines in textile samples over the years

Aromatic amines not mentioned in table 8 are not determined in a PT of iis yet.

In this PT the observed variation expressed as the relative standard deviation RSD of the test results for the sum of o-Aminoazotoluene and o-Toluidine and for 3,3'-Dimethoxybenzidine is in line with the previous PTs.

## 4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT a few questions were asked about accreditation and about sample details such as sample intake, sample preparation and the use of a diatomaceous earth column. From the participants that answered these additional questions, one hundred and ten participants (92%) mentioned that they are accredited for determination of banned AZO Dyes in Textile. Nine participants mentioned that the laboratory is not accredited for the determination of aromatic amines in textiles.

All but one participant used 0.5 grams or more for testing. The samples were used as received by 70% of the participants and further cut or grinded by 30%.

About the use of diatomaceous earth column as prescribed in ISO14362-1 the majority of the participants (79%) reported to have used this column. Twenty-two participants did not use this column and two participants reported to have used a different test method. No effect was observed on the averages or variation between reported test results.

<sup>\*)</sup> In 2015 a sample was also positive for o-Aminoazotoluene and o-Toluidine. The uncertainties were respectively 48% and 70%, while the sum had an uncertainty of 34%.

## 5 DISCUSSION

All reporting participants were able to detect either o-Aminoazotoluene and/or o-Toluidine and/or the sum of both in sample #20545. No other aromatic amines were detected. All reporting participants were able to detect in 3,3'-Dimethoxybenzidine in sample #20546. No other aromatic amines were detected.

When the results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU and with the similar Bluesign® BSSL (Table 9), it was noticed that not all participants would make identical decisions about the acceptability of the textiles for the determined components.

Ecolabel	colabel baby clothes		no direct skin contact		
Bluesign® BSSL	<20 mg/kg	<20 mg/kg	<20 mg/kg		
Oeko-Tex 103	<20 mg/kg	<20 mg/kg	<20 mg/kg		

Table 9: Bluesign® BSSL and Ecolabelling Standards and Requirements for Textiles in EU

All reporting laboratories would have rejected sample #20545 for all categories, except three participants. All reporting laboratories would have rejected sample#20546 for all categories, except one participant.

## 6 CONCLUSION

It can be concluded that the majority of the participants has no problem with detecting the o-Aminoazotoluene and o-Toluidine or 3,3'-Dimethoxybenzidine in the samples.

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-Aminoazotoluene (o-AAT, CAS no. 97-56-3), o-Toluidine (o-Tol, CASno. 95-53-4)

and the sum of o-Aminoazotoluene and o-Toluidine in sample #20545; results in mg/kg

lab	method	o-AAT	o-Tol	luidine in sample #2 Sum reported	Sum calc by iis	mark	z(targ)
210		ND					
230							
339	In house	24.7	<5		24.7		-3.33
348	In house	32.16	57.11	89.27	89.27		-0.06
362 551	In house	 13.2712	 156.4821	169.7533	 169.7533		4.02
623	In house ISO14362-1	17.20	100.4621	117.92	117.92		1.40
840	EN14362-1	24.1	25.9	50	50		-2.05
841	ISO14362-1	12.5	<5	12.5	12.5		-3.95
2108	EN14362-1		70		70		-1.03
2115	EN14362-1	8.90	45.41		54.31		-1.83
2120	ISO14362-1	12	48		60		-1.54
2121							
2129	EN14362-1	44.05	126	400.74	126		1.81
2137 2139	KS K0147 EN14362-1	11.05 47	91.69 16	102.74 63	102.74 63		0.63 -1.39
2165	EN14362-1	47 		119.7	119.7		1.49
2170	EN14362-1		52.20		52.2		-1.94
2172	EN14362-1	16.1	100.1	116.2	116.2		1.31
2184	EN14362-1			118	118		1.40
2201	EN14362-1	6.78	100.80	107.58	107.58		0.87
2217	ISO14362-1		137.28		137.28		2.38
2218	EN14362-1	 	58.79	40.40	58.79		-1.60
2236	EN14362-1	7.70	11.73	19.43	19.43		-3.60
2238 2241	EN14362-1 EN14362-1	<5 6.37	100.0 99.26	100.0 105.63	100.0 105.63		0.49 0.77
2247	LIN14302-1	0.37					0.77
2255	EN14362-1	n.d	96.1	96.1	96.1		0.29
2256	EN14362-1	23.42 C			23.42		-3.40
2258							
2261	GB/T17592	19.56	90.13		109.69		0.98
2265	EN14362-1	119.5	116.3 C		235.8	R(0.01)	7.37
2266	100440004	45.40	400.00	400.00	400.00		
2271	ISO14362-1	15.40	120.80	136.20	136.20		2.32
2272 2284	EN14362-1 EN14362-1	20.1 16.06	115.02	131.08	20.1 131.08		-3.56 2.06
2286	ISO14362-1	26.00	32.07	58.07 C	58.07		-1.64
2287	EN14362-1	20.53	31.89		52.42		-1.92
2289	ISO14362-1	<5	104	104	104		0.69
2290	ISO14362-1	36.7	71.2	107.9	107.9		0.89
2291		11.0	106.8	117.8 C	117.8		1.39
2293	ISO14362-1	ND	102.87	102.87	102.87		0.63
2295	ISO14362-1		36.0	 	36		-2.76
2297 2310	EN14362-1	nd 	116.1 	nd E 	116.1		1.30
2311							
2313							
2314							
2320							
2330	ISO14362-1	41.77	29.00	70.77	70.77		-0.99
2347	GB/T17592	<5	109		109		0.94
2350	EN14362-1	48.59	7.01		55.6		-1.76
2352	ISO14362-1		112	116.0	112		1.10
2357 2358	ISO14362-1 ISO14362-1	31.78	116.0 59.43	116.0 91.21	116 91.21		1.30 0.04
2364	ISO14362-1	31.70	109.45		109.45		0.04
2365	ISO14362-1	11.97 C	115.26	127.23 C	127.23		1.87
2366	EN14362-1	<5	113	113 C	113		1.15
2367	ISO14362-1	14.03 C	113.93 C		127.96		1.91
2370	EN14362-1	5.21	72.0	77.2	77.2		-0.67
2372	ISO14362-1	5.995	65.104	71.099	71.099		-0.98
2373	EN44262 4		101.42	140.4	101.42		0.56
2375 2378	EN14362-1 ISO14362-1	2.1	110.3 114.8	112.4	112.4 114.8		1.12 1.24
2379	ISO14362-1	18.5151	14.6 14.475 C		32.9901		-2.91
2380	1301-1002-1	16.5151	14.475 C	<del></del>	32.9901		-2.91
2381							
2386	EN14362-1	23.7	40.1	63.8	63.8		-1.35
2390	EN14362-1		73.90		73.9		-0.84
2406	ISO14362-1	37.75	59.20		96.95		0.33
2410	ISO14362-1	39.31	67.12	106.43	106.43		0.81
2425							
2426							

lab	method	o-AAT	o-Tol	Sum reported	Sum calc by iis mark	z(targ)
2429	EN14362-1	ND	112	112 C	112	1.10
2442	ISO14362-1	21.05 C	87.61	108.66 C	108.66	0.93
2449	10014302-1	21.00				
2453	ISO14362-1	14.5	42.0	56.5	56.5	-1.72
2456	ISO14362-1	9.25	64.06 C	73.31 C	73.31 	-0.87 
2459	10044000 480					
2462	ISO14362-1&3	44.44	103.2	103.2	103.2	0.65
2472	ISO14362-1	11.41	97.09	108.50	108.50	0.92
2475	100440004					
2476	ISO14362-1	<5.0	101.840	<5.0 E	101.840	0.58
2482	ISO14362-1	45.2	71.3		116.5	1.32
2489						
2492	EN14362-1		65.3		65.3	-1.27
2494	ISO14362-1	112.5 C	7.82 C	120.32 C	120.32	1.52
2500	EN14362-1	16.21	105.33	121.54	121.54	1.58
2501	ISO14362-1	17.30	97.90	115.20	115.2	1.26
2504	ISO14362-1	n.a.	94.88 C	n.d. E	94.88	0.23
2511	ISO14362-1		34.5717		34.5717	-2.83
2514	EN14362-1		99.14		99.14	0.44
2515	EN14362-1		113.76	113.76	113.76	1.19
2527	EN14362-1		59.9		59.9	-1.55
2528	EN14362-1	14.50	42.00		56.5	-1.72
2532	2.11.1002					
2534						
2538	ISO14362-1	46.94	64.15		111.09	1.05
2549	15014502-1					1.05
2561	100440004	45.00		40440	404.40	0.70
2565	ISO14362-1	15.93	88.26	104.19	104.19	0.70
2567	EN14362-1		100.4		100.4	0.51
2572	ISO14362-1	33.9	69.3	103.2	103.2	0.65
2573	ISO14362-1	14 C	116.9	131 C	131	2.06
2582						
2590						
2591	EN14362-1	15.3	7.0		22.3	-3.45
2602	ISO14362-1	96.5	42.3	138.8	138.8	2.45
2605	ISO14362-1	<5	112.96	112.96	112.96	1.14
2609	ISO14362-1		93.37		93.37	0.15
2618						
2638					<del></del>	
2643	EN14362-1	33.497	36.633	70.130	70.13	-1.03
2644						
2668						
2674	EN14362-1			118	118	1.40
2678	ISO14362-1	15.14	75.7	90.84	90.84	0.02
2689	ISO14362-1	9	102	111	111	1.05
2713				77	77	
	ISO14362-1	21.23	55.86			-0.68
2730						
2743	EN144000 4					4.07
2768	EN14362-1	25.23215	90.1673	115.3995	115.3995	1.27
2789	ISO14362-1		70.05		70.05	-1.03
2798	ISO14362-1		77		77	-0.68
2804	EN14362-1	28.0	68.4	96.4	96.4	0.30
2805	ISO14362-1	102 C	None Det.		102	0.59
2812	EN14362-1	Not Det. C	108.95		108.95	0.94
2823	EN14362-1		36.4490 C		36.4490	-2.73
2827						
2830	ISO14362-1	4.85	88.15 C	101.7 C,E	101.7	0.57
2864	CNS15205	10.99	89.67	100.66	100.66	0.52
2867	ISO14362-1			121.3	121.3	1.57
2870						
2910	EN14362-1	21.2	95.4	116.6	116.6	1.33
2912	E111-1002 1					
3100	ISO14362-1	<5.0	94.77	94.77	94.77	0.22
3110	EN14362-1	52.6	39.0	91.5	91.5	0.06
3116	LIV14302-1				<del></del>	
	100440004					
3118	ISO14362-1	40.00	57.24	57.24 C	57.24	-1.68
3122	EN14362-1	13.88	<5	13.88 C	13.88	-3.88
3153	EN14362-1	<5	92.6	92.6	92.6	0.11
3154	EN14362-1		39.02		39.02	-2.60
3160	ISO14362-1		59.88		59.88	-1.55
3172	ISO14362-1	< 5	36.685	36.685 C	36.685	-2.72
3176	ISO14362-1	36.42			36.42	-2.74
3182	ISO14362-1	28.8	62.6	91.4	91.4	0.05
3185	ISO14362-1	<5	112.73	112.73	112.73	1.13
3190	EN14362-1	27.3	67.4	94.7	94.7	0.22
3191	ISO14362-1		110.67		110.67	1.03
3197	ISO14362-1	<5	102.9	102.9 C	102.9	0.63
3199	ISO14362-1&3	78.9	26.3		105.2	0.75
						-

lab	method	o-AAT	o-Tol	Sum reported	Sum calc by iis mark	z(targ)
3210	In house			61.1	61.1	-1.48
3214	ISO14362-1	34.69	78.10	112.79	112.79	1.14
3216						
3218	ISO14362-1		103.58		103.58	0.67
3222						
3228	EN14362-1			123.2	123.2	1.66
3232						
3237	ISO14362-1	30.03	77		107.03	0.84
3248	EN14362-1	7	94	101	101	0.54
3250	ISO14362-1		110.01		110.01	1.00
8008	EN14362-1	37.4	68.9	106.3	106.3	0.81
	normality				OK	
	n	70	109	73	122	
	outliers				1	
	mean (n)				90.385	
	st.dev. (n)				31.8455 RSD = 35	%
	R(calc.)				89.167	
	st.dev.(ISO14362-1:17)				19.7233	
	R(ISO14362-1:17)				55.225	

Lab 2256 first reported for o-AAT: 223.42

Lab 2265 first reported for o-Tol: <5

Lab 2286 first reported for the sum I: <5

Lab 2291 first reported for the sum: ND

Lab 2365 first reported for o-AAT: <5 and for the sum: <5

Lab 2366 first reported for the sum: <5

Lab 2367 first reported for o-AAT: ND and for o-Tol: 102.5

Lab 2379 first reported for o-Tol: Not Detected

Lab 2429 first reported for for the sum: ND

Lab 2442 first reported for o-AAT: ND

Lab 2456 first rep. for o-Tol: 34.06 and for the sum: 43.31

Lab 2494 first rep. for o-AAT: 101.6, for o-Tol: ND and for the sum: 101.6

Lab 2504 first reported for o-Tol: n.d.

Lab 2573 first reported for o-AAT: ND and for the sum: 116.9

Lab 2805 first reported for o-AAT: 120

Lab 2812 first reported for o-AAT: 92.31

Lab 2823 first reported for o-Tol: 36.6958

Lab 2830 first reported for o-Tol: ND and for the sum: ND

Lab 3118 first reported for the sum: ND

Lab 3122 first reported for the sum: <5

Lab 3172 first reported for the sum: <5

Lab 3197 first reported for the sum: <5

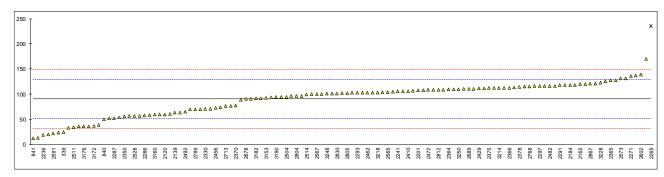
#### Calculation Errors (E) of the sum:

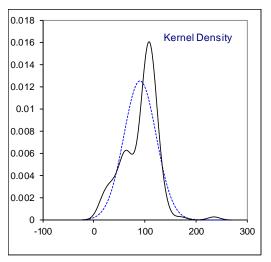
Lab 2297 reported o-Toluidine to be present, but as sum n.d.

Lab 2476 reported o-Toluidine to be present, but as sum <5

Lab 2504 iis calculated: 94.88, o-Toluidine result corrected without correcting the sum?

Lab 2830 iis calculated: 93, o-Toluidine and sum corrected without correcting o-Aminoazotoluene?



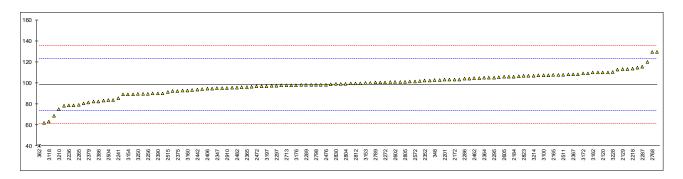


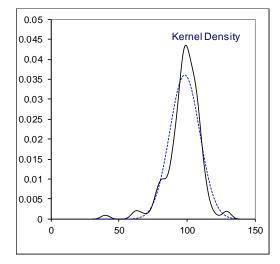
# Determination of 3,3'-Dimethoxybenzidine (CASno. 119-90-4) in sample #20546; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO14362-1	107.31		0.71	
230					
339	In house	110.0		0.93	
348	In house	102.62		0.33	
362	EN14362-1	40.0	R(0.01)	-4.74	
551	In house	113.2691		1.20	
623	ISO14362-1	61.81		-2.97	
840	EN14362-1	103.0		0.37	
841	ISO14362-1	92.64		-0.47	
2108	EN14362-1	120 88.94		1.74	
2115 2120	EN14362-1 ISO14362-1	110		-0.77 0.93	
2121	13014302-1			0.93	
2129	EN14362-1	113		1.18	
2137	KS K0147	99.53		0.08	
2139	EN14362-1	78		-1.66	
2165	EN14362-1	107.5		0.73	
2170	EN14362-1	98.99		0.04	
2172	EN14362-1	103		0.37	
2184	EN14362-1	106		0.61	
2201	EN14362-1	102.90		0.36	
2217	ISO14362-1	100.99		0.20	
2218	EN14362-1	113.35		1.20	
2236	ISO14362-1	78.41		-1.63	
2238	EN14362-1	98.0		-0.04	
2241	EN14362-1	85.21		-1.08	
2247					
2255	EN14362-1	97.8		-0.06	
2256	EN14362-1	89.45		-0.73	
2258					
2261	GB/T17592	89.32		-0.74	
2265	EN14362-1	79.3		-1.55	
2266					
2271	ISO14362-1	104.60		0.49	
2272		100.6		0.17	
2284		96.23		-0.18	
2286	ISO14362-1	103.81		0.43	
2287	EN14362-1	115.44		1.37	
2289	ISO14362-1	98		-0.04	
2290	ISO14362-1	105.9		0.60	
2291		98.4		-0.01	
2293	ISO14362-1	104.93		0.52	
2295		105		0.53	
2297	EN14362-1	97.3		-0.10	
2310					
2311					
2313					
2314					
2320 2330	EN14262 1	60 16		-2.43	
2330 2347	EN14362-1 ISO14362-1	68.46 95		-2.43 -0.28	
2350	EN14362-1	80.38		-0.26 -1.47	
2352	ISO14362-1	102		0.28	
2357	ISO14362-1	110.0		0.28	
2358	ISO14362-1	96.89		-0.13	
2364	ISO14362-1	104.89		0.52	
2365	ISO14362-1	95.97		-0.20	
2366	EN14362-1	92		-0.53	
2367	ISO14362-1	108.07		0.78	
2370	EN14362-1	93.9		-0.37	
2372	ISO14362-1	97.663		-0.07	
2373		107.04		0.69	
2375	EN14362-1	92.4		-0.49	
2378	ISO14362-1	104		0.45	
2379	ISO14362-1	81.3562		-1.39	
2380					
2381					
2386		82.4		-1.30	
2390		89.96		-0.69	
2406	ISO14362-1	94.47		-0.33	
2410	ISO14362-1	107.64		0.74	
2425					
2426					

	•	<del>.</del>		- (, )	
2429	method EN14362-1	value 108	mark	<b>z(targ)</b> 0.77	remarks
2442	ISO14362-1	93.75		-0.38	
2449					
2453	ISO14362-1	89.9		-0.70	
2456	ISO14362-1	83.78 		-1.19 	
2459 2462	ISO14362-1&3	104.5		0.49	
2472	ISO14362-1	96.84		-0.13	
2475					
2476	ISO14362-1	98.230		-0.02	
2482	ISO14362-1	95.4		-0.25	
2489 2492	EN14362-1	90.1		-0.68	
2494	ISO14362-1	129.6		2.52	
2500	EN14362-1	108.11		0.78	
2501	EN14362-1	78.65		-1.61	
2504	ISO14362-1	83.496		-1.21	
2511 2514	ISO14362-1 EN14362-1	107.7589 101.70		0.75 0.26	
2515	EN14362-1	91.13		-0.60	
2527	EN14362-1	112.5		1.13	
2528	EN14362-1	106.83		0.68	
2532					
2534 2538	ISO14362-1	95.78		-0.22	
2549	10014302-1				
2561					
2565	ISO14362-1	103.16		0.38	
2567	EN14362-1	97.9		-0.05	
2572 2573	ISO14362-1 ISO14362-1	101.3 98.2		0.23 -0.02	
2582	13014302-1	90.2		-0.02	
2590					
2591	EN14362-1	82.2		-1.32	
2602	ISO14362-1	100.9		0.20	
2605 2609	ISO14362-1 ISO14362-1	105.76 100.49		0.59 0.16	
2618	13014302-1	100.49		0.10	
2638					
2643	EN14362-1	95.304		-0.26	
2644					
2668 2674	EN14362-1	109		0.85	
2678	ISO14362-1	89.26	С	-0.75	first reported: 178.11
2689	ISO14362-1	95	Ū	-0.28	mot openious in our i
2713	ISO14362-1	97.74		-0.06	
2730					
2743 2768	EN14362-1	129.3225		2.50	
2789	ISO14362-1	100.17		0.14	
2798	ISO14362-1	98		-0.04	
2804	EN14362-1	99.0		0.04	
2805	ISO14362-1	101		0.20	
2812 2823	EN14362-1 EN14362-1	99.32 106.5958		0.07 0.66	
2827	_11110021				
2830	EN14362-1	98.80	С	0.03	first reported: 22.12
2864	CNS15205	94.60		-0.32	
2867	ISO14362-1	114.4		1.29	
2870 2910	EN14362-1	95.0		-0.28	
2912	LIV14302-1			-0.20	
3100	GB/T17592	107.19		0.70	
3110	EN14362-1	93.3		-0.42	
3116	EN14362-1	100		0.12	
3118 3122	ISO14362-1 EN14362-1	63.01 97.14		-2.87 -0.11	
3153	EN14362-1 EN14362-1	99.9		0.11	
3154		89.14		-0.76	
3160	ISO14362-1	92.77		-0.46	
3172	ISO14362-1	108.92		0.84	
3176 3182	EN14362-1 ISO14362-1	97.80 109.8		-0.06 0.92	
3185	ISO14362-1	109.6		0.92	
3190	EN14362-1	100.7		0.18	
3191	ISO14362-1	105.33		0.55	
3197	ISO14362-1	96.9		-0.13	
3199	ISO14362-1	83.1		-1.25	

lab	method	value	mark :	z(targ)	remarks
3210	In house	75.2		-1.89	
3214	ISO14362-1	106.83		0.68	
3216					
3218	ISO14362-1	106.18		0.62	
3222					
3228	EN14362-1	110.2		0.95	
3232					
3237	ISO14362-1	99.3		0.07	
3248	EN14362-1	102		0.28	
3250	ISO14362-1	89.28		-0.75	
8008	EN14362-1	101.1		0.21	
	normality	suspect			
	n	125			
	outliers	1			
	mean (n)	98.491			
	st.dev. (n)	11.0346	RSD = 11%		
	R(calc.)	30.897			
	st.dev.(ISO14362-1:17)	12.3465			
	R(ISO14362-1:17)	34.570			





# **APPENDIX 2 Summary of other reported aromatic amines**

#### Abbreviations of amine names

4AD = 4-Aminodiphenyl (CASNo. 92-67-1)

BD = 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'-Diaminodiphenyl methane (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'-Diaminodiphenyl methane (CASNo. 838-88-0)

pC = p-Cresidine (CASno. 120-71-8)

DDM = 4,4'-Diamino-3,3'-dichlorodiphenyl methane (CASno. 101-14-4)

DDE = 4,4'-Diaminodiphenyl ether (CASno. 101-80-4)

DDS = 4,4'-Diaminodiphenyl sulphide (CASno. 139-65-1)

oTol = 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 = 2,5Xylidine (CASno. 95-78-3)

26X = 2,6-Xylidine (CASno. 87-62-7)

TX = Total of Xylidine

# Summary of other reported aromatic amines in sample #20545, see abbreviations above

Lab	4AD	BD	4CoT	2NA	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						1124						
551	N.D.											
623	ND											
840	Not Det											
841	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2108												
2115												
2120	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2121												
2129												
2137												
2139												
2165	ND											
2170												
2172												
2184	n.d.											
2201	ND[<5]											
2217												
2218												
2236												
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2241	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2247												

Lab	4AD	BD	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM
2255	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2256												
2258												
2261												
2265	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2266												
2271 2272	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 
2284	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2286	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2287												
2289	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2290	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
2291	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2293	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2295												
2297	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
2310												
2311												
2313 2314												
2320												
2330	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350												
2352												
2357												
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2364												
2365	<5	<5	<5	<5	<5	< <u>5</u>	<5	< <u>5</u>	<5	<5	<5	< <u>5</u>
2366	<5 ND	<5 ND	<5 ND	<5 ND	<5 ND	<5 ND	<5 ND	<5 ND	<5 ND	<5 ND	<5 ND	<5 ND
2367	ND .E	ND .F	ND .F	ND .E	ND .F	ND	ND .F	ND .E	ND -5	ND -5	ND -5	ND .E
2370 2372	<5 n.d.	<5	<5	<5	<5 n.d.	<5 n.d.	<5	<5 n.d.	<5 n.d.	<5 n.d.	<5 n.d.	<5
2373	11.u.	n.d. 	n.d. 	n.d. 	11.u.	11.u.	n.d. 	11.u.	11.u.	11.u.	11.u.	n.d. 
2375												
2378												
2379	Not Det	Not Det	Not Det	Not Det	Not Det	Not Det	Not Det	Not Det	Not Det	Not Det	Not Det	Not Det
2380												
2381												
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2390												
2406												
2410												
2425 2426												
2429	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2442	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2449												
2453												
2456												
2459												
2462	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2475 2476	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2476	<5.0	<5.0	<5.0	<5.0	<5.0	<0.0	<0.0	<0.0	<0.0	<0.0	<5.0	<3.0
2489												
2492												
2494	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2500	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2501												
2504	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2511												
2514												
2515 2527												
2527 2528												
2532												
2534												
2538												
2549												
2561												
2565	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2567	<5	<5	<5	<5		<5	<5	<5	<5	<5	<5	<5
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
2573	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2582												

Lab	4AD	BD	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM
2590												
2591												
2602												
2605	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2609												
2618												
2638												
2643												
2644												
2668												
2674	n.d.											
2678 2689	0 ND	0	0	0	0 ND	0	0	0	0 ND	0	0	0
2713	ND <5	ND <5	ND <5	ND <5	<5	ND <5	ND <5	ND <5	<5	ND <5	ND <5	ND <5
2730												
2743												
2768	n.d											
2789												
2798												
2804	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2805	Not Det											
2812												
2823			28.5535*					45.2278*				
2827												
2830	ND											
2864	ND											
2867	n.d.											
2870	n d	n d	 n d	n d	n d	n d	n d	 n d	n d	n d	n d	n d
2910 2912	n.d. 											
3100	<5.0	<5.0	< 5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
3110												
3116												
3118	ND											
3122	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154												
3160												
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176	N.D	N.D.	N.D.						N.D	N.D.		N.D.
3182	N.D.											
3185	<5 	<5	<5 	<5	<5 	<5 						
3190 3191												
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3199	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3210												
3214	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3216												
3218												
3222												
3228	N.D.											
3232												
3237												
3248												
3250												
8008												

<sup>\*)</sup> Lab 2823 first reported for 4CoT: 29.7269 and for DADM: 67.8218

# Summary of aromatic amines in sample #20545 continued

Lab	рС	DDM	DDE	DDS	24DAT	TMA	οA	24X	25X	26X	TX
210											
230											
339	<5				<5	<5	<5				
348	<5	<5	<5	<5	<5	<5	<5	<5		<5	
362											
551	N.D.		N.D.								
623	ND										
840	not det										
841	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2108											
2115											

		2014			0.45.45			0.434	051/	201/	<b>T</b> V
2120	<b>pC</b> < 5	<b>DDM</b> < 5	<b>DDE</b> < 5	<b>DDS</b> < 5	<b>24DAT</b> < 5	<b>TMA</b> < 5	<b>oA</b> < 5	<b>24X</b> < 5	25X	<b>26X</b> < 5	TX 
2121											
2129											
2137											
2139											
2165	ND	ND	ND	ND	ND	ND	ND	ND		ND	
2170											
2172	 n d	 n d	 n d	 n d		 n d					
2184 2201	n.d. ND[<5]	n.d. ND[<5]	n.d. ND[<5]	n.d. ND[<5]	n.d. ND[<5]	n.d. ND[<5]	n.d. ND[<5]	n.d. ND[<5]	 ND[<5]	n.d. ND[<5]	ND[<5]
2217											
2218											
2236											
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2241	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0		<5.0	<5.0
2247 2255	 n d	 n d	 n d	 n d	 n d	 n d	 n d				
2256	n.d 	n.d 	n.d 	n.d 	n.d 	n.d 	n.d 	n.d 	n.d 	n.d 	n.d 
2258											
2261											
2265	< 5	< 5	< 5	< 5	< 5	< 5	< 5				< 5
2266											
2271	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2272											
2284 2286	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5
2287											
2289	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2290	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
2291	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND*
2293	ND	ND	ND	ND	ND	ND	ND	ND		ND	ND
2295											
2297 2310	nd 	nd 	nd 	nd 	nd 	nd 	nd 	nd 	nd 	nd 	nd 
2310											
2313											
2314											
2320											
2330	ND	ND	ND	ND	ND	ND	ND	ND	NA	ND	ND
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	
2350											
2352 2357											
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2364											
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2367	ND	ND	ND	ND	ND	ND	ND	ND		ND	
2370 2372	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2372	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 
2375											
2378											
2379	not det	not det	not det	not det	not det	not det					
2380											
2381											
2386 2390	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<15 
2406											
2410											
2425											
2426											
2429	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2442	ND	ND 	ND	ND	ND 	ND 	ND	ND 			
2449 2453											
2456											Traces
2459											
2462	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	
2472	<5	<5	<5	<5	<5	<5	<5	<5		<5	
2475											
2476	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0 	<5.0	<5.0	<5.0	<5.0
2482 2489											
2409											
2494	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2500	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2501											

Lob	C	DDM	DDE	DDC	24DAT	TNAA	- ^ ^	247	2EV	acv	TV
2504	pC n.d	DDM n.d	DDE	DDS	24DAT	TMA	oA n d	24X	25X	26X	TX n.d
	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2511											
2514											
2515											
2527											
2528											
2532											
2534											
2538											
2549											
2561											
2565	<5	<5	<5	<5	<5	<5	<5	<5		<5	
2567	<5	<5	<5	<5	<5	<5	<5	<5		<5	
2572	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2573	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2582											
2590											
2591											
2602											
2605	<5	<5	<5	<5	<5	<5	<5	<5		<5	
2609											
2618											
2638											
2643											
2644											
2668											
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.				
2678	0	0	0	0	0	0	0	0	0	0	0
2689	ND	ND	ND	ND	ND	ND	ND	ND		ND	ND
2713	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	
2730											
2743											
2768	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2789											
2798											
2804	<10	<10	<10	<10	<10	<10	<10				
2805	not det	not det	not det	not det	not det	not det	not det	not det		not det	
2812											
2823											
2827											
2830	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2864	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2867	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.			
2870											
2910	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	
2912											
3100	<5.0	< 5.0	<5.0	<5.0	< 5.0	< 5.0	<5.0	< 5.0		< 5.0	
3110											
3116											
3118	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3122	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154											
3160											
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176											
3182	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
3185	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3190											
3191											
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3199	<10	<10	<10	<10	<10	<10	<10	<10		<10	
3210											
3214	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3216											
3218											
3222											
3228	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.		N.D.	
3232											
3237											
3248											
3250											
8008											

<sup>\*)</sup> Lab 2291 first reported for TX 117.8

Summary of other reported aromatic amines in sample #20546, see abbreviations above

					•	_	•	546, see	_	-	•	
Lab	4AD	BD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	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.											
623	ND											
840	not det											
841	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2108												
2115 2120	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2121												
2129												
2137												
2139												
2165	ND											
2170												
2172 2184	n.d.											
2201	ND[<5]											
2217												
2218												
2236												
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2241	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2247	 n d											
2255 2256	n.d 											
2258												
2261												
2265	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2266												
2271	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2272												
2284 2286	<5 <5											
2287												
2289	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2290	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
2291	ND											
2293	ND											
2295	 nd		 nd	 nd	 nd		 nd	 n d	 nd	 nd		
2297 2310	nd 											
2311												
2313												
2314												
2320												
2330	ND -	ND	ND -	ND	ND -	ND -	ND	ND	ND -	ND _	ND _	ND
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350 2352												
2357												
2358	n.d.											
2364												
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	<5 ND											
2367	ND -E	ND 45	ND 15	ND 15	ND 45	ND 45	ND -E	ND 45	ND -F	ND -E	ND -5	ND 15
2370 2372	<5 n.d.											
2373												
2375												
2378												
2379	not det											
2380												
2381	 -5											
2386 2390	<5 											
2406												
2410												
2425												
2426												
2429	ND											
2442	ND											
2449												

Lab	4AD	BD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMB	DDDM
2453												
2456					Traces							
2459 2462	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2475												
2476	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2482												
2489 2492												
2494	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2500	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2501												
2504	n.d.	n.d.	n.d.	n.d.	n.a. 	n.a. 	n.d.	n.d.	n.d. 	n.d. 	n.d. 	n.d. 
2511 2514												
2515												
2527												
2528												
2532 2534												
2538												
2549												
2561												
2565	<5 -5	<5	<5 .5	<5 -5	<5	<5	<5 .5	<5 -5	<5 -5	<5 .5	<5 -5	<5 -5
2567 2572	<5 <5.0	<5 <5.0	<5 <5.0	<5 <5.0	<5.0	<5.0	<5 <5.0	<5 <5.0	<5 <5.0	<5 <5.0	<5 <5.0	<5 <5.0
2573	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2582												
2590												
2591 2602												
2605	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2609												
2618												
2638												
2643 2644												
2668												
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2678	0	0	0	0	0	0	0	0	0	0	0	0
2689 2713	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5
2713												
2743												
2768	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2789 2798												
2804	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2805	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2812												
2823												
2827 2830	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2864		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2867	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	 n d	 n d	 n d	 n d	 n d	 n d	 n d	 n d	 n d	 n d	 n d	 n d
2910 2912	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 
3100	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
3110												
3116	ND.	AUD.	ND.	ND.	ND.	ND.	ND.	ND.	ND.	ND.	ND.	ND.
3118 3122	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5	ND <5
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154												
3160												
3172 3176	< 5 	< 5 	< 5 	< 5 	< 5 	< 5 	< 5 	< 5 	< 5 	< 5 	< 5 	< 5 
3176	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D
3185	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3190												
3191												
3197 3199	<5 <10	<5 <10	<5 <10	<5 <10	<5 <10	<5 <10	<5 <10	<5 <10	<5 <10	<5 <10	<5 <10	<5 <10
3210			-10									
0210												
3214 3216	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 

Lab	4AD	BD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMB	DDDM
3218												
3222												
3228	N.D.											
3232												
3237												
3248												
3250												
8008												

# Summary of aromatic amines in sample #20546 continued

											-	oAAT+	
Lab 210	рС	DDM	DDE	DDS	oTol	24DAT	TMA	οA	24X	25X	26X	оТ	TX
210 230													
339	<5				<5	<5	<5	<5					
348	<5	<5	<5	<5	<5	<5	<5	<5	<5		<5	<5	
362													
551	N.D.	ND	N.D.	N.D.	ND								
623 840	ND not det												
841	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2108													
2115													
2120	< 5	< 5	< 5	< 5	< 5	< 5	< 5 	< 5	< 5		< 5		
2121 2129													
2137													
2139													
2165	ND		ND	ND									
2170 2172													
2172	n.d.		n.d.	n.d.									
2201	ND[<5]												
2217													
2218													
2236 2238	 -5	 -5	<5	<5	<5	<5	 -5	<5	<5	<5	<5	 -5	<5
2236	<5 <5.0	<5 <5.0	<5.0	<5.0	<5.0	<5.0	<5 <5.0	<5.0	<5.0	<0 	<5.0	<5 <5.0	<5.0
2247													
2255	n.d												
2256													
2258 2261													
2265	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5					< 5
2266													
2271	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2272 2284	<5	<5	<5	<5	<5	<5	<5		<5	<5	<5	<5	<5
2286	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5
2287													
2289	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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
2291 2293	ND ND	ND 	ND ND	ND ND	98.4 ND								
2295													
2297	nd												
2310													
2311													
2313 2314													
2320													
2330	ND	NA	ND	ND	ND								
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5		
2350													
2352 2357													
2358	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 2370	ND <5	<5	ND <5	<5	<5								
2372	n.d.												
- <del>-</del>													

	•	-	-	-	-	-	-	-	-	-	•	oAAT+	
Lab	рС	DDM	DDE	DDS	oTol	24DAT	TMA	οA	24X	25X	26X	оТ	TX
2373 2375													
2378													
2379	Not Det	Not Det											
2380													
2381	 -5	 -5	 -5	 -15									
2386 2390	<5 	<5 	<5 	<15 									
2406													
2410													
2425													
2426 2429	ND	ND	ND	ND									
2442	ND												
2449													
2453													
2456 2459								Traces					
2459	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5		
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5		<5	<5	
2475													
2476	<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
2482 2489													
2492													
2494	ND	ND	ND	ND									
2500	N.D.	N.D.	N.D.	N.D.									
2501 2504	n.d.	n.d.	n.d.	n.d.									
2511	11.u. 	11.u.	11.u.	11.u.	11.u.	11.u.	11.u. 	11.u. 	11.u. 	11.u. 	11.u.	11.u.	
2514													
2515													
2527 2528													
2532													
2534													
2538													
2549													
2561 2565	<5	<5	<5	<5	<5	<5	<5	<5	<5		<5	<5	
2567	<5	<5	<5	<5	<5	<5	<5	<5	<5		<5		
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
2573	ND	ND	ND	ND									
2582 2590													
2591													
2602													
2605	<5	<5	<5	<5	<5	<5	<5	<5	<5		<5	<5	
2609 2618													
2638													
2643													
2644													
2668	 n d												
2674 2678	n.d. 0	0	0	0	0	0							
2689	ND		ND	ND	ND								
2713	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2730													
2743 2768	n.d	n.d	n.d	n.d									
2789													
2798													
2804	<10	<10	<10	<10	<10	<10	<10	<10				<10	
2805 2812	Not Det		Not Det										
2823													
2827													
2830	ND	ND	ND	ND									
2864 2867	ND n.d	ND 	ND 	ND n.d	ND								
2867 2870	n.d. 			n.d. 									
2910	n.d.		n.d.	n.d.									
2912													
3100	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0		<5.0	<5.0	
3110 3116													
0110													

	-		-	-	•	<u>.</u>	•	•	-	•	-	oAAT+	
Lab	рC	DDM	DDE	DDS	oTol	24DAT	TMA	οA	24X	25X	26X	οТ	TX
3118	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3122	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154													
3160													
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176													
3182	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
3185	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3190													
3191													
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3199	<10	<10	<10	<10	<10	<10	<10	<10	<10		<10		
3210													
3214	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3216													
3218													
3222													
3228	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.		N.D.	N.D.	
3232													
3237													
3248													
3250													
8008													

**APPENDIX 3 Analytical details** 

APPE	NDIX 3 Ana	alytical deta	ils	
		Sample		
	Laboratory	intake in	Sample	
lab	accredited	grams	preparation	Test method followed
210	Yes			
230	 No			<del></del>
339	No	1 ~	 Curthor Cut	a different test method
348 362	only o-Tol.	1 g	Further Cut	a different test method
	Yes Yes	1.0g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
551 623	Yes	1.0g 1 gram	Further Cut Further Cut	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
840	Yes	0.5	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
841	Yes	0.5 gram	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2108	Yes	0,25 g	Further Cut	
2115	Yes	0,23 g 0.5 g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2120	Yes	1 g	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2121		' 9		
2129	Yes	0.1	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2137	Yes		Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2139	Yes	1.003g	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2165	Yes	1	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2170	Yes	0.6 grams	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2172	Yes	1	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2184	Yes	1 gram	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2201	Yes	0.50 g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2217	Yes	1.0	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2218	Yes	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2236	Yes	~1gram	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2238	Yes	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2241	Yes	0.5g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2247				
2255	Yes	0.5	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2256	Yes	1	Further Grinded	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2258		à		I''
2261	Yes	1g	Used as received	a different test method; GB/T 17592-2011
2265	Yes	0,5g	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2266	 V	0.5-	Lland on manning d	ICO142CO 4 Agreeu E and did NOT use the distance agree and solution
2271	Yes	0.5g	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2272 2284	Yes Yes	1gram	Used as received Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2286	No	0.5g 0.5g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2287	No	0.5g 0.5g	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2289	Yes	1.0g	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2290		1.09		
2291	Yes		Further Grinded	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2293	No	0.72 g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2295	Yes	1 gram	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2297	Yes	1.0g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2310		Ü		
2311				
2313				
2314				<del></del>
2320				
2330	Yes	0.5 grams	Further Cut	ISO 14362-1: 2017 did not use the diatomaceous earth column
2347	Yes			
2350			Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2352	Yes	0.5g	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2357				
2358		#00F4F. 0 Fm	#00F4Ff	<del></del>
0064	Voc	#20545: 0.5g	#20545further cut.	ICO11262 1 chapter 10 1 and used the distance accuse earth column
2364	Yes	#20546: 1.0g	#20546 as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2365	Yes	0.5grams	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2366 2367	Yes Yes	1.0g 0.5g	Used as received Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2370	Yes	0.5 g	Further Cut	ISO14362-1 Chapter 10.4 and used the diatomaceous earth column
2370	No	1g	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2373	Yes	0.5g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2375	Yes	0,75 g	Further Cut	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2378	Yes	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2379	Yes	19	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2380		•		
2381				
2386	Yes	0,5	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2390	Yes	1.0 gram	Further Cut	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2406	Yes	1 gram	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2410	Yes	0.5 g	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2425				
2426				

		Sample		
	Laboratory	intake in	Sample	
lab	accredited	grams	preparation	Test method followed
2429	Yes	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2442	Yes	1.0g	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2449		•		
2453	Yes	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2456	Yes	1	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2459				
2462	Yes	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2472	Yes	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2475				
2476	Yes	1 gm	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2482	Yes	0,5	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2489				
2492	Yes	0.5	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2494	Yes	1 Gram	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2500	Yes	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2501	No	1.0010g	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2504	Yes	0.5	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2511	No	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
0544	V	#20545:0.44g	Hand or an extend	10044000 4 shorter 40 4 and so although the second control of the s
2514	Yes	#20546:0.56g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2515	Yes	About 0.5 g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2527	Yes	1 gram	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2528	Yes	0.5g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2532				<del></del>
2534	 Van	05 1 ~	 Further Cut	
2538 2549	Yes 	0,5 - 1 g		ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2561				
2565			 	
2567	Yes	0.5	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2572		0.5		
2573	Yes	0.5g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2582		0.09		
2590				
2591	Yes	1	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2602	Yes	1 g	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2605	Yes	0.500	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2609	Yes	1.0g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2618		9		
2638				
2643	Yes	0.5098	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2644				<del></del>
2668				
2674	Yes	1.0g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2678	No	1 g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2689	Yes	0.5g	Further Cut	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
		#20545:0.53;		
2713	Yes	#20546:0.71	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2730				
2743				
2768	Yes	1 grams	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2789	Yes	1	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2798	Yes	0.5g	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2804	Yes	1	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2805	Yes	0.5g	Further Cut	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2812	Yes	1	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2823	Yes	1.0 g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2827	 Van	4	Lload on roosiyad	10044369 4 shorter 40 4 and ward the distance of the self-
2830	Yes	1	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2864	Yes	0.5	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2867	Yes	0.5g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2870	 Voo	10	Llood on received	ISO14262 1 abouter 10 4 and wood the distance court court
2910 2912	Yes 	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3100	Yes	0.5054	Further Cut	
3110	Yes Yes	0.505g 0.5	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column ISO14362-1 Annex E and did NOT use the diatomaceous earth column
3116	Yes	1 gram	Used as received	ISO14362-1 Affilex E and did NOT use the diatomaceous earth column
3118	Yes	0.5150 g	Further Cut	ISO14362-1 Chapter 10.4 and used the diatomaceous earth column
3110	Yes	1 gr	Used as received	ISO14362-1 Affilex E and did NOT use the diatomaceous earth column
3153	Yes	.5g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3154	Yes	.5g 1	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3160	Yes	0.9 g	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3172	Yes	2.5	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3176	Yes	0,5 g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3182	Yes	1 gram	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3185	Yes	1.0g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
		J		,

lab	Laboratory accredited	Sample intake in grams	Sample preparation	Test method followed
3190	Yes	1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3191	Yes	0.5 grams	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3197	Yes	0,5 grams #20545:0.45g	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3199	No	#20546:0.50g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3210		1g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3214	Yes	1 g	Further Cut	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3216		•		
3218	Yes	0.5g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3222		· ·		- <del></del>
3228	Yes	1	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3232				- <del></del>
3237	Yes	0,5g	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column
3248	Yes	0.5	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
3250	Yes	1 g	Used as received	ISO14362-1 chapter 10.4 and used the diatomaceous earth column
8008	Yes	0.5	Used as received	ISO14362-1 Annex E and did NOT use the diatomaceous earth column

## **APPENDIX 4**

# Number of participants per country

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

#### **APPENDIX 5**

#### **Abbreviations**

C = final test result after checking of first reported suspect test result

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \\ DG(0.05) &= \text{straggler in Double Grubbs' outlier test} \\ \end{array}$ 

R(0.01) = outlier in Rosner's outlier test R(0.05) = straggler in Rosner's outlier test E = possibly an error in calculations

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

n.a. = not applicablen.d. = not determinedn.e. = not evaluated

f+? = possible false positive f-? = possible false negative

### Literature

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