

Institute for Interlaboratory Studies

# Results of Proficiency Test AZO Dyes in Textile May 2022

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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 from AZO Dyes in Textile. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the determination of banned aromatic amines derived from AZO Dyes in Textile.

In this interlaboratory study 149 laboratories in 32 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the AZO Dyes in Textile 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 #22595 and the second sample is a blue knitted Tricot labelled #22596. 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 pink polyester textile was selected which was dyed with an AZO dye specially to detect 4-Aminobenzene. The batch was cut in small pieces and mixed thoroughly. After homogenization 190 small plastic bags were filled with approximately 3 grams each and labelled #22595.

The batch was used in a previous proficiency test on AZO Dyes in Textile as sample #18521 in PT iis18A03. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of blue knitted textile positive on AZO Dyes was selected. The batch was cut in small pieces and mixed thoroughly. After homogenization 190 small plastic bags were filled with approximately 3 grams each and labelled #22596.

The homogeneity of the subsamples was checked by the determination of Benzidine and 3,3-Dimethylbenzidine accordance with EN14362-1 on 8 stratified randomly selected subsamples.

	Benzidine in mg/kg	3,3-Dimethylbenzidine in mg/kg
sample #22596-1	80.3	44.1
sample #22596-2	72.9	39.2
sample #22596-3	71.8	40.1
sample #22596-4	72.7	42.3
sample #22596-5	82.0	44.8
sample #22596-6	71.3	40.7
sample #22596-7	76.2	39.4
sample #22596-8	76.7	42.8

Table 1: homogeneity test results of subsamples #22596

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

	Benzidine in mg/kg	3,3-Dimethylbenzidine in mg/kg
r (observed)	11.2	6.0
reference method	iis memo 2022	iis memo 2022
0.3 x R (reference method)	13.3	7.4

Table 2: evaluation of the repeatabilities of subsamples #22596

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample labelled #22595 and one sample labelled #22596 was sent on April 6, 2022.

### 2.5 ANALYZES

The participants were reguested to determine on sample #22595: 4-Aminoazobenzene (CAS No. 60-09-3)

On sample #22596 it was requested to determine: 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

It was decided not to request 4-Aminoazobenzene, CAS no. 60-09-3 on sample #22596, because the sample #22596 was 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.

To ensure homogeneity it was requested not to use less than 0.5 grams of the sample 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 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 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 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.

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon

(up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1 was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying 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 (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 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:

 $z_{(target)}$  = (test result - average of PT) / 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

Some problems were encountered with the dispatch of samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. Twenty-six participants reported test results after the extended reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.

In total 146 participants reported 431 numerical test results. Observed were 9 outlying test results, which is 2.1%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

#### 4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the 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 test 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.

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. Unfortunately, only for a few aromatic amines precision data are mentioned in this test method and when 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 quite strict target value for the 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 quality of the test results. Furthermore, it was decided to use the same target reproducibly for all aromatic amines. The average relative standard deviations over all iis PTs and components for Textile is 21%. This investigation is summarized in iis memo 2202.

#### sample #22595

<u>4-Amino-azobenzene (CAS No. 60-09-3)</u>: The determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the reproducibility derived from the iis memo 2202.

#### sample #22596

- <u>Benzidine (CAS No. 92-87-5)</u>: The determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility derived from the iis memo 2202.
- <u>3,3'-Dimethylbenzidine (CAS No. 119-93-7):</u> The determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility derived from the iis memo 2202.

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

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

Component	unit	n	average	2.8 * sd	R(target)
4-Aminoazobenzene	mg/kg	142	101.4	74.9	59.6

Table 3: reproducibility of the tests on aromatic amine in textile sample #22595

Component	unit	n	average	2.8 * sd	R(target)
Benzidine	mg/kg	141	70.2	25.3	41.2
3,3'-Dimethylbenzidine	mg/kg	139	48.5	17.8	28.5

Table 4: reproducibilities of the test on aromatic amine in textile sample #22596

Without further statistical calculations it can be concluded that the group of laboratories has difficulties with the analysis of 4-Amino-azobenzene but has no problem with the analysis of Benzidine and 3,3'-Dimethylbenzidine. The tests have been discussed in paragraph 4.1.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2022 WITH PREVIOUS PTS

	May 2022	March 2021	March 2020	March 2019	March 2018
Number of reporting laboratories	146	150	126	165	171
Number of test results	431	291	380	299	496
Number of statistical outliers	9	30	2	11	3
Percentage of statistical outliers	2.1%	10.3%	0.5%	3.7%	0.6%

Table 5: 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, expressed as relative standard deviations (RSD) of the PTs, see below table.

Component	May 2022	March 2021	March 2020	March 2019	2004 - 2018	iis memo 2202
4-Aminodiphenyl	n.e.	n.e.	n.e.	n.e.	18-36%	21%
Benzidine	13%	n.e.	n.e.	n.e.	12-35%	21%
4-Chloro-o-toluidine	n.e.	n.e.	n.e.	n.e.	24%	21%
2-Naphtylamine	n.e.	n.e.	n.e.	n.e.	27-41%	21%
4-Chloroaniline	n.e.	n.e.	n.e.	n.e.	27%	21%
2,4-Diaminoanisol	n.e.	n.e.	n.e.	n.e.	24-52%	21%
4,4'-Diaminodiphenylmethane	n.e.	n.e.	n.e.	n.e.	21%	21%
3,3'-Dimethoxybenzidine	n.e.	n.e.	11%	11%	12-31%	21%
3,3'-Dimethylbenzidine	13%	n.e.	n.e.	n.e.	15-36%	21%
4,4'-Diamino-3,3'- Dichlorodiphenylmethane	n.e.	n.e.	n.e.	n.e.	20-35%	21%
4,4'-Diaminodiphenylether	n.e.	n.e.	n.e.	n.e.	15%	21%
4,4'-Diaminodiphenylsulfide	n.e.	n.e.	n.e.	n.e.	18-26%	21%
2,4-Diaminotoluene	n.e.	25%	n.e.	n.e.	n.e.	21%
o-Anisidine	n.e.	14%	n.e.	n.e.	n.e.	21%
2,4-Xylidine	n.e.	n.e.	n.e.	26%	19%	21%
o-Aminoazotoluene *)	n.e.	n.e.	n.e.	n.e.	n.e.	21%
o-Toluidine *)	n.e.	n.e.	n.e.	n.e.	19-38%	21%
Sum of o-aminoazotoluene and o-Toluidine *)	n.e.	n.e.	35%	n.e.	34%	21%
4-Aminoazobenzene	26%	n.e.	n.e.	n.e.	30%	21%

Table 6: development of the uncertainties over the years

Aromatic amines not mentioned in table 6 are not determined in an iis PT yet. The uncertainty (RSD) of the determined AZO dyes in Textile in this PT is in line with previous PTs.

### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported analytical details from the participants are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- About 85% mentioned that they are accredited for determination of banned AZO Dyes in Textile.
- Almost all participants used 0.5 grams or more for testing.
- The samples were used as received by 50% of the participants and further cut by 40%.
- About 5% of the participants used the ASE technique to release/extract the analyte(s), 10% Soxhlet, 45% Mechanical Shaking, 10% Thermal desorption, 10% Ultrasonic and about 20% used another technique.
- t-Butyl methyl ether and citrate buffer were the most reported solvents used to release the analyte(s).
- About 90% of the participants reported an extraction time between 30 minutes and 60 minutes.
- About 90% of the participants reported and extraction temperature of 70 °C.
- About 65% of the participants reported to have used the diatomaceous earth column for sample #22596.

No effect was observed on the averages or variation between reported test results. Therefore, no further investigations were done.

#### 5 DISCUSSION

All of reporting participants were able to detect 4-Aminoazobenzene in sample #22595 and almost all reporting participants were able to detect 3,3'-Dimethylbenzidine and Benzidine in sample #22596.

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

Ecolabel baby clothes in mg/kg		in direct skin contact in mg/kg	no direct skin contact in mg/kg	
Bluesign® RSL	<20	<20	<20	
OEKO-TEX® 100	<20	<20	<20	

Table 7: Bluesign® BSSL and OEKO-TEX® Ecolabelling Standards and Requirements for Textiles in EU

Almost all reporting laboratories, except three, would have rejected sample #22595 for all categories. Almost all reporting laboratories, except four, would have rejected sample #22596 for all categories.

### 6 CONCLUSION

Although it can be concluded that the majority of the participants have no problem with the detgermination of 4-Aminoazobenzene, Benzidine and 3,3'-Dimethylbenzidine in the samples of this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

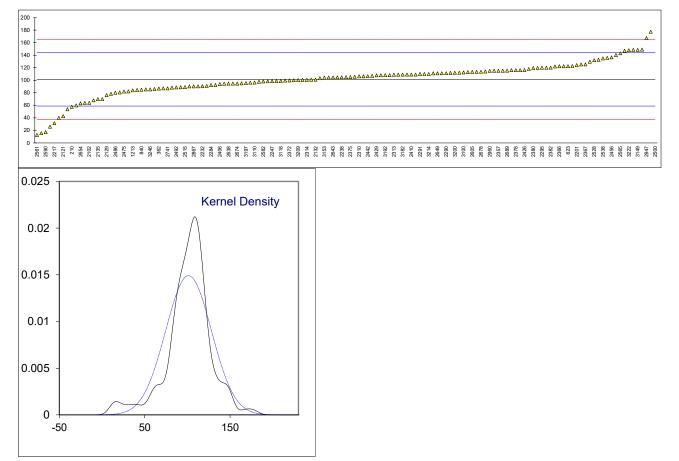
#### **APPENDIX 1**

Determination of 4-Aminoazobenzene (CAS No. 60-09-3) in sample #22595; results in mg/kg

Deterr		noazobenzer	ne (CAS	5 No. 60-0	09-3) in sample #22595; results in mg/kg
lab	method	value	mark	z(targ)	remarks
210	ISO14362-3	57.65		-2.05	
339					
362	ISO14362-3	86.799		-0.69	
551	ISO14362-3	15.53		-4.03	
623	ISO14362-3	122.39		0.99	
840	EN14362-3	85.0		-0.77	
841	ISO14362-3	87		-0.68	
1213	ISO14362-3	83.94		-0.82	
1910	ISO14362-3	115.045		0.64	
2102	In house	63.93		-1.76	
2108	ISO14362-3	109	0	0.36	first year acts of 20, C2
2115	ISO14362-3	59.88	С	-1.95	first reported 39.63
2121	ISO14362-3	42.80		-2.75	
2129 2132	ISO14362-3 EN14362-3	76.3 100.9749		-1.18 -0.02	
2132	ISO14362-3	69.8		-0.02 -1.48	
2135	ISO14362-3	107		-1.46	
2165	EN14362-3	92.4		-0.42	
2170	ISO14362-3	84.49		-0.79	
2184	EN14362-3	82.2		-0.90	
2201	ISO14362-3	124.2		1.07	
2213	ISO14362-3	106.29		0.23	
2217	EN14362-3	31.47		-3.28	
2232	EN14362-3	90.44790		-0.51	
2238	ISO14362-3	104.8		0.16	
2241	ISO14362-3	97.400		-0.19	
2247	EN14362-3	98.48		-0.14	
2256	EN14362-3	115.12		0.64	
2258	10011000				
2265	ISO14362-3	88.8		-0.59	
2284	ISO14362-3	92.22		-0.43	
2286	ISO14362-3	129.63		1.33	
2290	ISO14362-3	111.2 110		0.46	
2291 2293	ISO14362-3	110		0.40	
2293	ISO14362-3	120		0.87	
2295	ISO14362-3	106.23		0.87	
2311	ISO14362-3	98.723		-0.13	
2313	ISO14362-3	108.64		0.34	
2314	ISO14362-3	100.52		-0.04	
2320	ISO14362-3	100.96		-0.02	
2330	ISO14362-3	105.50		0.19	
2347	ISO14362-3	116		0.69	
2350	ISO14362-3	111.01		0.45	
2352	ISO14362-3	114		0.59	
2357	ISO14362-3	115.1		0.64	
2358	ISO14362-3	100.5		-0.04	
2364	ISO14362-3	111.12		0.46	
2365	ISO14362-3	122.7		1.00	
2366	EN14362-3	122.3		0.98	
2367	ISO14362-3	125.4		1.13	
2370 2372	ISO14362-3	94.67 99.6457		-0.32 -0.08	
2372	ISO14362-3 ISO14362-3	99.6457 125.23		-0.08 1.12	
2375	ISO14362-3	125.23		0.17	
2375	ISO14362-3	116		0.17	
2379	EN14362-3	135.5643		1.60	
2380	EN14362-3	119.5		0.85	
2381	ISO14362-3	118.00		0.78	
2382	ISO14362-3	120.1		0.88	
2386	EN14362-3	85.9		-0.73	
2389	ISO14362-3	119.67		0.86	
2390	ISO14362-3	111.70		0.48	
2406	ISO14362-3	93.93		-0.35	
2410	ISO14362-3	109		0.36	
2425	In house	112.0	-	0.50	
2426	ISO14362-3	116.331	С	0.70	first reported 167.795
2429	ISO14362-3	107.83		0.30	
2442	ISO14362-3	106.47		0.24	
2449	ISO14362-3	107.89		0.30	
2453	ISO14362-3	132.64		1.47	
2456 2475	ISO14362-3 EN14362-3	136.5 82.048		1.65 -0.91	
2475	ISO14362-3	96.22		-0.91	
2710	1001-002-0	00.22		-0.24	

lab	mothod	valuo	mark	z(tara)	romarks
2486	ISO14362-3	value 80.13	mark	z(targ) -1.00	remarks
2489	EN14362-3	98.42		-0.14	
2492	GB/T23344	88.48		-0.61	
2500	ISO14362-3	496	R(0.01)	18.53	
2511	ISO14362-3	122.0	()	0.97	
2515	EN14362-3	89.05		-0.58	
2520	ISO14362-3	80.5		-0.98	
2527	ISO14362-3	90.2		-0.53	
2528	EN14362-3	132.11		1.44	
2532	EN14362-3	94.58		-0.32	
2534	ISO14362-3	148.9		2.23	
2538	EN14362-3	134.77		1.57	
2549	EN14362-3	99.42		-0.09	
2560	ISO14362-3	109		0.36	
2561	ISO14362-3	12.71		-4.17	
2565	ISO14362-3	143.05		1.96	
2567	EN14362-3	108.9		0.35	
2572 2573	ISO14362-3	108.2 110		0.32 0.40	
2573	ISO14362-3				
2582	ISO14362-3 ISO14362-3	98.13 17.321		-0.15 -3.95	
2590	ISO14362-3	146.93		-3.95 2.14	
2605	ISO14362-3	140.93		0.56	
2618	EN14362-3	113.40		0.56	
2638	EN14362-3	94.653		-0.32	
2643	EN14362-3	104.49		0.14	
2644	ISO14362-3	177.3		3.56	
2649	EN14362-3	111.05	С	0.45	first reported 170.5
2654	ISO14362-3	63.12		-1.80	
2668	EN14362-3	103.22		0.09	
2674	EN14362-3	94.7		-0.31	
2678	ISO14362-3	113.502		0.57	
2689	ISO14362-3	115.3		0.65	
2703	EN14362-3	148.38		2.21	
2740	EN14362-3	70		-1.47	
2741	EN14362-3	87.079	0	-0.67	
2743	ISO14362-3	26.01	С	-3.54	first reported 9.24
2793	ISO14362-3	78.2162 120		-1.09	
2798 2823	ISO14362-3	120		0.87	
2827	EN14362-3	104.96		0.17	
2829	ISO14362-3	63.90		-1.76	
2852	ISO14362-3	67.9		-1.57	
2864					
2867	ISO14362-3	90.2		-0.53	
2947	In house	167.118		3.09	
2960	ISO14362-3	115		0.64	
2976	ISO14362-3	88.2295		-0.62	
2977	ISO14362-3	39.8426	С	-2.89	first reported 29.8295
2979	EN14362-3	85.32		-0.76	
2980	EN14362-3	95		-0.30	
3100	ISO14362-3	112.43		0.52	
3110	EN14362-3	96.37		-0.24	
3116	ISO14362-3	90.88		-0.49	
3118	ISO14362-3	98.78 148 4		-0.12	
3149 3153	ISO14362-3	148.4 104.1		2.21 0.13	
3153	ISO14362-3 ISO14362-3	104.1 90.3		-0.52	
3154 3160	ISO14362-3	90.3 140.16		-0.52 1.82	
3172	ISO14362-3	122.31		0.98	
3172	ISO14362-3	104.42		0.30	
3182	ISO14362-3	108.99		0.36	
3185	ISO14362-3	112.98		0.54	
3192	ISO14362-3	108.178		0.32	
3197	ISO14362-3	95.5		-0.28	
3200	ISO14362-3	111.8		0.49	
3209	I7SO14362-3	100.32		-0.05	
3210	In house	53.6		-2.24	
3214	ISO14362-3	110.05		0.41	
3218	ISO14362-3	116.0		0.69	
3222	EN14362-3	147.85		2.18	
3230 3237	EN14362-3	 104.5		0.15	
3237 3246	ISO14362-3	104.5 85.33		-0.75	
3240	EN14362-3	100		-0.07	
0210				0.01	

normality n outliers mean (n) st.dev. (n) R(calc.) st.dev.(iis memo 2202)	not OK 142 1 101.408 26.7483 74.895 21.2957	RSD = 26%
st.dev.(iis memo 2202) R(iis memo 2202)	21.2957 59.628	

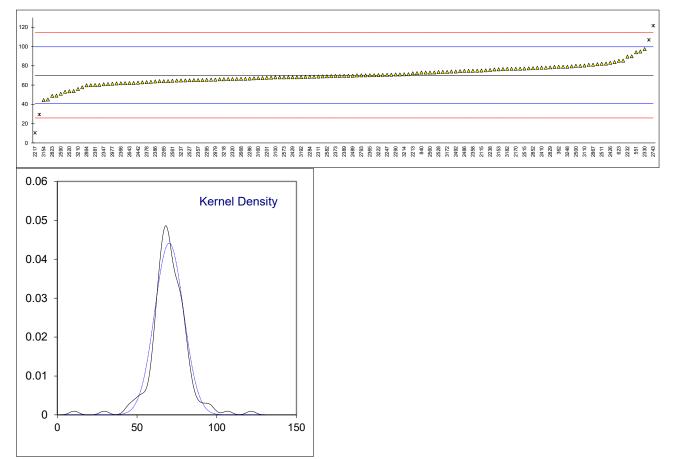


### Determination of Benzidine (CAS No. 92-87-5) in sample #22596; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO14362-1	73.58	manA	0.23	ionano
339	ISO14362-1	not detected			possibly a false negative test result?
362	ISO14362-1	79	С	0.60	first reported 112.6
551	ISO14362-1	93.98		1.62	·······
623	ISO14362-1	85.19		1.02	
840	EN14362-1	73.0		0.19	
841	ISO14362-1	73		0.19	
1213	ISO14362-1	74.07		0.27	
1910	ISO14362-1	69.46		-0.05	
2102	EN14362-1	67.29		-0.19	
2108	ISO14362-1	77		0.46	
2115	EN14362-1	75.04		0.33	
2121 2129	ISO14362-1 ISO14362-1	77.90 94.82		0.53 1.67	
2129	EN14362-1	94.82 82.292		0.82	
2132	EN14362-1	53.8		-1.11	
2139	ISO14362-1	81		0.74	
2165	EN14362-1	85.3		1.03	
2170	ISO14362-1	76.87		0.46	
2184	EN14362-1	78.9		0.59	
2201	ISO14362-1	67.5		-0.18	
2213	ISO14362-1	72.23		0.14	
2217	EN14362-1	10.465	R(0.01)	-4.05	
2232	ISO14362-1	89.4911		1.31	
2238	ISO14362-1	75.7		0.38	
2241	ISO14362-1	68.580		-0.11	
2247	EN14362-1	70.53		0.03	
2256 2258	EN14362-1 ISO14362-1	74.86 29.47	R(0.01)	0.32 -2.76	
2265	ISO14362-1	64.0	R(0.01)	-2.70	
2284	ISO14362-1	68.58		-0.11	
2286	ISO14362-1	66.77		-0.23	
2290	ISO14362-1	71.0		0.06	
2291	ISO14362-1	45		-1.71	
2293					
2295	ISO14362-1	65.48		-0.32	
2310	ISO14362-1	65.23		-0.33	
2311	ISO14362-1	68.815		-0.09	
2313	ISO14362-1	62.82		-0.50	
2314	ISO14362-1	65.11		-0.34	
2320	ISO14362-1	66.31		-0.26	
2330	ISO14362-1	97.35		1.85	
2347 2350	ISO14362-1 ISO14362-1	60.9 79.34		-0.63 0.62	
2350	ISO14362-1	65.6		-0.31	
2357	ISO14362-1	65.2		-0.34	
2358	ISO14362-1	74.8		0.32	
2364	ISO14362-1	68.40		-0.12	
2365	ISO14362-1	70.2		0.00	
2366	EN14362-1	61.8		-0.57	
2367	ISO14362-1	66.2		-0.27	
2370	ISO14362-1	75.27		0.35	
2372	ISO14362-1	73.04		0.20	
2373	ISO14362-1	69.43		-0.05	
2375	ISO14362-1	64		-0.42	
2378	ISO14362-1	63		-0.49	
2379 2380	ISO14362-1	64.0314 69.4		-0.42 -0.05	
2380	EN14362-1 ISO14362-1	69.4 60.00		-0.05 -0.69	
2381	ISO14362-1 ISO14362-1	62.2		-0.69 -0.54	
2382	EN14362-1	63.8		-0.34	
2389	EN14362-1	69.56		-0.04	
2390	ISO14362-1	64.78		-0.36	
2406	ISO14362-1	71.01		0.06	
2410	ISO14362-1	78		0.53	
2425	In house	66.0		-0.28	
2426	ISO14362-1	83.13		0.88	
2429	ISO14362-1	68.04		-0.14	
2442	ISO14362-1	62.54		-0.52	
2449	ISO14362-1	68.0		-0.15	
2453	ISO14362-1	77.42		0.49	
2456	ISO14362-1	61.98 67 76		-0.55	
2475 2476	EN14362-1 ISO14362-1	67.76 106.83	R(0.05)	-0.16 2.49	
2476	ISO14362-1	74.70	1 (0.03)	0.31	
2.00		• • • •		0.01	

lab	method	value	mark	z(targ)	remarks
2489	ISO14362-1	69.62	mark	-0.04	Temarks
2492	GB/T17592	74.10		0.27	
2500	ISO14362-1	80		0.67	
2511	ISO14362-1	82.1		0.81	
2515	EN14362-1	77.19		0.48	
2520	ISO14362-1	53.6		-1.12	
2527	ISO14362-1	65.0		-0.35	
2528	EN14362-1	73.54		0.23	
2532	ISO14362-1	72.4		0.15	
2534	ISO14362-1	70.1		0.00	
2538	EN14362-1	70.51		0.02	
2549 2560	EN14362-1 ISO14362-1	70.72		0.04	
2560 2561	ISO14362-1 ISO14362-1	73 64.41		0.19 -0.39	
2565	ISO14362-1	59.88		-0.70	
2567	EN14362-1	74.4		0.29	
2572	ISO14362-1	71.3		0.08	
2573	ISO14362-1	68		-0.15	
2582	ISO14362-1	69.05		-0.07	
2590	EN14362-1	51.04		-1.30	
2591	ISO14362-1	74.7		0.31	
2605	ISO14362-1	68.98		-0.08	
2618	EN14362-1	69.60	_	-0.04	
2638	EN14362-1	66.31	С	-0.26	first reported 37.77
2643	EN14362-1	62.03		-0.55	
2644	ISO14362-1	60.1		-0.68	
2649	EN14362-1	83.87		0.93	
2654	ISO14362-1	68.06 66.44		-0.14	
2668 2674	EN14362-1 EN14362-1	66.44 76.2		-0.25 0.41	
2674	ISO14362-1	61.701		-0.57	
2689	ISO14362-1	70		-0.01	
2703	EN14362-1	57.76		-0.84	
2740	EN14362-1	61		-0.62	
2741	EN14362-1	76.828		0.45	
2743	ISO14362-1	121.61	C,R(0.01)	3.49	first reported 177.77
2793	ISO14362-1	70.0675	C	-0.01	first reported 112.4494
2798	ISO14362-1	79		0.60	·
2823	EN14362-1	48.757	С	-1.45	first not reported
2827	EN14362-1	68.01		-0.15	
2829	ISO14362-1	78.31	С	0.55	first reported 97.89
2852	ISO14362-1	77.43		0.49	
2864	CNS16113-1	59.78		-0.70	
2867	ISO14362-1	81.1		0.74	
2947	In house	<loq< td=""><td></td><td></td><td></td></loq<>			
2960 2976	ISO14362-1 ISO14362-1	67 81.6438		-0.21 0.78	
2970	EN14362-1	61.3990		-0.59	
2977	ISO14362-1	65.6		-0.39	
2980	EN14362-1	53		-1.16	
3100	GB/T17592	67.96		-0.15	
3110	EN14362-1	80.33		0.69	
3116	ISO14362-1	76.52		0.43	
3118	ISO14362-1	70.22		0.00	
3149	ISO14362-1	49.1		-1.43	
3153	ISO14362-1	76.3		0.42	
3154	EN14362-1	44.503		-1.74	
3160	ISO14362-1	67.16		-0.20	
3172	ISO14362-1	73.686		0.24	
3176	ISO14362-1	64.63		-0.37	
3182	ISO14362-1	76.82		0.45	
3185	ISO14362-1	66.44		-0.25	
3192 3197	ISO14362-1	68.309 80.8		-0.12 1.33	
3197	ISO14362-1 ISO14362-1	89.8 63.3		-0.47	
3200 3209	ISO14362-1 ISO14362-1	63.3 80.02		-0.47 0.67	
3209 3210	In house	55.9		-0.97	
3210	ISO14362-1	71.15		0.07	
3218	ISO14362-1	66.0		-0.28	
3222	EN14362-1	70.35		0.01	
3230					
3237	EN14362-1	64.7		-0.37	
3246	ISO14362-1	78.0		0.53	
3248	EN14362-1	79		0.60	

normality	suspect	
n	141	
outliers	4	
mean (n)	70.150	
st.dev. (n)	9.0407	RSD = 13%
R(calc.)	25.314	
st.dev.(iis memo 2202)	14.7316	
R(iis memo 2202)	41.248	

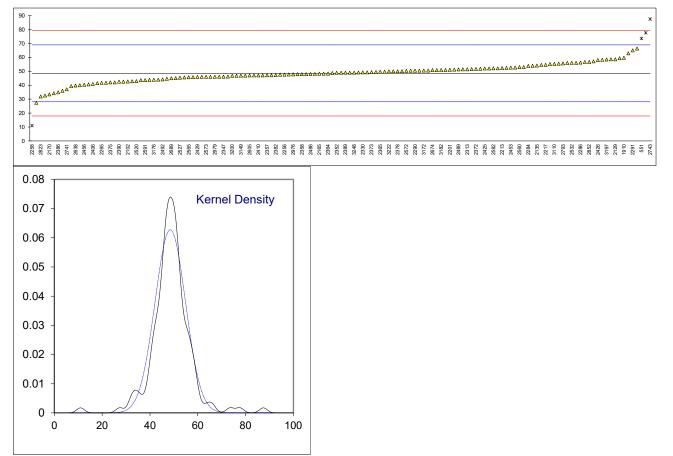


### Determination of 3,3'-Dimethylbenzidine (CAS No. 119-93-7) in sample #22596; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO14362-1	58.56	IIIdi K	2(targ) 0.98	Telliarks
339	ISO14362-1	not detected			possibly a false negative test result?
362					
551	ISO14362-1	73.57	R(0.05)	2.46	
623	ISO14362-1	55.95		0.73	
840	EN14362-1	50.0		0.14	
841 1213	ISO14362-1 ISO14362-1	44 51.79		-0.44 0.32	
1910	ISO14362-1	59.69		1.10	
2102	EN14362-1	42.64		-0.58	
2108	ISO14362-1	66.3		1.74	
2115	EN14362-1	53.9		0.53	
2121	100140004				
2129 2132	ISO14362-1 EN14362-1	58.6 47.595		0.99 -0.09	
2132	EN14362-1	54.1		0.55	
2139	ISO14362-1	46		-0.25	
2165	EN14362-1	48.3		-0.02	
2170	ISO14362-1	33.37		-1.49	
2184	EN14362-1	50.4		0.18	
2201 2213	ISO14362-1 ISO14362-1	51.0 52.30		0.24 0.37	
2213	EN14362-1	54.60		0.60	
2232	ISO14362-1	50.9387		0.24	
2238	ISO14362-1	47.0		-0.15	
2241	ISO14362-1	48.092		-0.04	
2247	EN14362-1	57.13		0.84	
2256 2258	EN14362-1	47.62 10.99	P(0.01)	-0.09 -3.68	
2265	ISO14362-1 ISO14362-1	41.7	R(0.01)	-0.67	
2284	ISO14362-1	53.86		0.52	
2286	ISO14362-1	56.25		0.76	
2290	ISO14362-1	50.4		0.18	
2291	ISO14362-1	65		1.62	
2293	100140004				
2295 2310	ISO14362-1 ISO14362-1	51.57 50.5		0.30 0.19	
2310	ISO14362-1	49.427		0.09	
2313	ISO14362-1	51.42		0.28	
2314	ISO14362-1	49.21		0.07	
2320	ISO14362-1	48.75		0.02	
2330	ISO14362-1 ISO14362-1	49.21		0.07	
2347 2350	ISO14362-1	46.2 47.89		-0.23 -0.06	
2352	ISO14362-1	48.9		0.04	
2357	ISO14362-1	47.1		-0.14	
2358	ISO14362-1	47.9		-0.06	
2364	ISO14362-1	48.34		-0.02	
2365	ISO14362-1	49.7		0.12	
2366 2367	EN14362-1 ISO14362-1	46.8 43.6		-0.17 -0.48	
2307	ISO14362-1	43.0 50.02		-0.48	
2372	ISO14362-1	51.65		0.31	
2373	ISO14362-1	49.31		0.08	
2375	ISO14362-1	42		-0.64	
2378	ISO14362-1	50		0.14	
2379 2380	ISO14362-1 EN14362-1	47.1219 47.9		-0.14 -0.06	
2380	ISO14362-1	46.10		-0.00	
2382	ISO14362-1	47.2		-0.13	
2386	EN14362-1	34.9		-1.34	
2389	EN14362-1	48.99		0.05	
2390	ISO14362-1	42.53		-0.59	
2406 2410	ISO14362-1 ISO14362-1	40.90 47		-0.75 -0.15	
2410 2425	In house	47 52.0		-0.15	
2426	ISO14362-1	58.052		0.93	
2429	ISO14362-1	45.94		-0.25	
2442	ISO14362-1	41.42		-0.70	
2449	ISO14362-1	36.0		-1.23	
2453	ISO14362-1	52.63 40.28		0.40 -0.81	
2456 2475	ISO14362-1 EN14362-1	40.28 49.11		0.06	
2476	ISO14362-1	58.24		0.95	
2486	ISO14362-1	48.04		-0.05	

lab	method	value	mark	z(targ)	remarks
2489	ISO14362-1	51.34		0.28	
2492	GB/T17592	44.18		-0.43	
2500	ISO14362-1	52		0.34	
2511	ISO14362-1	53.1		0.45	
2515	EN14362-1	44.62		-0.38	
2520	ISO14362-1	42.9		-0.55	
2527 2528	ISO14362-1 EN14362-1	45.4 59.34		-0.31 1.06	
2528	ISO14362-1	56.0		0.73	
2532	ISO14362-1	45.6		-0.29	
2538	EN14362-1	52.46		0.39	
2549	EN14362-1	55.49		0.68	
2560	ISO14362-1	53		0.44	
2561	ISO14362-1	51.39		0.28	
2565	ISO14362-1	45.67		-0.28	
2567	EN14362-1	49.0		0.05	
2572	ISO14362-1	50.3		0.17	
2573	ISO14362-1	46		-0.25	
2582	ISO14362-1	52.09		0.35	
2590	EN14362-1	32.57		-1.57	
2591	ISO14362-1	43.6		-0.48	
2605	ISO14362-1	46.97		-0.15	
2618	EN14362-1	52.65		0.40	
2638	EN14362-1	39.76		-0.86	
2643 2644	EN14362-1	43.77		-0.47 0.04	
2649	ISO14362-1 EN14362-1	48.95 55.14		0.04	
2654	ISO14362-1	56.66		0.80	
2668	EN14362-1	54.57		0.59	
2674	EN14362-1	50.8		0.22	
2678	ISO14362-1	40.600		-0.78	
2689	ISO14362-1	45		-0.35	
2703	EN14362-1	27.37		-2.08	
2740	EN14362-1	40		-0.84	
2741	EN14362-1	37.074		-1.12	
2743	ISO14362-1	87.46	C,R(0.01)	3.82	first reported 111.59
2793	ISO14362-1	55.6314	С	0.70	first reported 97.7207
2798	ISO14362-1	56	-	0.73	
2823	EN14362-1	31.920	С	-1.63	first not reported
2827	EN14362-1	52.13	0	0.35	first serviced 70.00
2829	ISO14362-1	62.89	С	1.41	first reported 78.62
2852 2864	ISO14362-1 CNS16113-1	56.7 34.23		0.80 -1.40	
2867	ISO14362-1	50.8		0.22	
2947	In house	not detected			possibly a false negative test result?
2960	ISO14362-1	46		-0.25	
2976	ISO14362-1	47.7709		-0.07	
2977	EN14362-1	42.5316		-0.59	
2979	ISO14362-1	46.0		-0.25	
2980	EN14362-1	42		-0.64	
3100	GB/T17592	46.23		-0.23	
3110	EN14362-1	55.32		0.67	
3116	ISO14362-1	50.55		0.20	
3118	ISO14362-1	49.72		0.12	
3149	ISO14362-1	46.9		-0.16	
3153	ISO14362-1	47.7		-0.08	
3154	EN14362-1	77.6	C,R(0.01)	2.85	first reported 31.166
3160	ISO14362-1	41.71		-0.67	
3172 3176	ISO14362-1 ISO14362-1	50.505 43.90		0.19 -0.45	
3182	ISO14362-1	43.90 50.92		-0.43	
3185	ISO14362-1	46.90		-0.16	
3192	ISO14362-1	45.016		-0.34	
3192	ISO14362-1	58.5		0.98	
3200	ISO14362-1	46.7		-0.18	
3209	ISO14362-1	48.32		-0.02	
3210	In house	39.4		-0.90	
3214	ISO14362-1	45.73		-0.27	
3218	ISO14362-1	47.0		-0.15	
3222	EN14362-1	49.90		0.14	
3230					
3237	EN14362-1	42.8		-0.56	
3246	ISO14362-1	51.25		0.27	
3248	EN14362-1	49		0.05	

normality	suspect	
n	139	
outliers	4	
mean (n)	48.524	
st.dev. (n)	6.3593	RSD = 13%
R(calc.)	17.806	
st.dev.(iis memo 2202)	10.1901	
R(iis memo 2202)	28.532	
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### **APPENDIX 2**

### Summary of other reported aromatic amines

#### Abbreviations of amine names

4AD	= 4-Aminodiphenyl (CAS No. 92-67-1)
BD	= Benzidine (CAS No. 92-87-5)
4CoT	= 4-Chloro-o-toluidine (CAS No. 95-69-2)
2NA	= 2-Naphtylamine (CAS No. 91-59-8)
ANT	= 2-Amino-4-nitrotoluene (CAS No. 99-55-8)
4CA	= 4-Chloraniline (CAS No. 106-47-8)
DAA	= 2,4-Diaminoanisol (CAS No. 615-05-4)
DADM	= 4,4'-Diaminodiphenylmethane (CAS No. 101-77-9)
DCB	= 3,3'-Dichlorobenzidine (CAS No. 91-94-1)
DMoxB	= 3,3'-Dimethoxybenzidine (CAS No. 119-90-4)
DMB	= 3,3'-Dimethylbenzidine (CAS No. 119-93-7)
DDDM	= 3,3'-Dimethyl-4,4'-Diaminodiphenylmethane (CAS No. 838-88-0)
рС	= p-Cresidine (CAS No. 120-71-8)
DDM	= 4,4'-Diamino-3,3'-dichlorodiphenylmethane (CAS No. 101-14-4)
DDE	= 4,4'-Diaminodiphenylether (CAS No. 101-80-4)
DDS	= 4,4'-Diaminodiphenylsulfide (CAS No. 139-65-1)
24DAT	= 2,4-Diaminotoluene (CAS No. 95-80-7)
ТМА	= 2,4,5-Trimethylaniline (CAS No. 137-17-7)
oA	= o-Anisidine (CAS No. 90-04-0)
24X	= 2,4-Xylidine (CAS No. 95-68-1)
25X	= 2,5-Xylidine (CAS No. 95-78-3)
26X	= 2,6-Xylidine (CAS No. 87-62-7)
ТХ	= Total of Xylidine
oAAT	= o-Aminoazotoluene (CAS No. 97-56-3)
oTol	= o-Toluidine (CAS No. 95-53-4)
oAAT&oTo	= Sum of o-Aminoazotoluene and o-Toluidine

### Summary of other reported aromatic amines in sample #22596, see abbreviations above

lab	4AD	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB	DDDM	рС	DDM
210	n.d.											
339	n.d.											
362 551												
623	n.d.											
840	n.d.											
841	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
1213	n.d.											
1910												
2102		n.d.										
2108 2115												
2113												
2129	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2135												
2139		 	 m al						 			
2165 2170	n.d. 											
2170	n.d.											
2201	n.d.											
2213												
2217	n.d.											
2232	 ~5	 ~5			 ~5		 ~5	 ~5		 <5	 ~5	 ~5
2238 2241	<5 <5											
2247	n.d.											
2256												
2258	n.d.											
2265	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2284 2286	n.d. <5											
2280	<5 <5											
2291	n.d.											
2293												
2295												
2310	n.d.											
2311 2313	n.d. n.d.											
2313												
2320	n.d.											
2330	n.d.											
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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		n.d.										
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366		<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2367		n.d.										
2370 2372		<3 n.d.										
2372		n.d.										
2375												
2378	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2379	n.d.											
2380	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2381 2382	 <5.0											
2386	<5.0 <5.0	<5.0	<5.0 <5.0	<5.0 <5.0								
2389		n.d.										
2390	n.d.											
2406		n.d.										
2410 2425	 n d											
2425 2426	n.a. n.d.	n.d. n.d.										
2420												
2442		n.d.										
2449												
2456		n.d.										
2475 2476												
2470		n.d.										

lab	4AD	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB	DDDM	рС	DDM
2489	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2492												
2500	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2511 2515												
2520	<5	<5	<5	 <5	<5	<5	<5	<5	<5	<5	<5	<5
2527												
2528												
2532	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2534	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2538	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2549 2560	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5
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	<5
2572	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2573	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2582 2590	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 
2590	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2605	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2618	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2638	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2643												
2644												
2649 2654												
2668	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2678	<5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2689	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2703												
2740 2741	 <5	 <5	 <5	 <5	 <5	 <5	 <5	 <5	 <5	 <5	 <5	 <5
2741	2.82	n.d.	-5 n.d.	<5 n.d.	n.d.	n.d.	∽5 n.d.	n.d.	∽5 n.d.	n.d.	∼5 n.d.	<5 n.d.
2793	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2798	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2823												
2827	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2829	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2852 2864	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 
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.
2947	<loq< td=""><td>n.d.</td><td>n.d.</td><td>n.d.</td><td>n.d.</td><td>n.d.</td><td>n.d.</td><td>n.d.</td><td>n.d.</td><td>n.d.</td><td>n.d.</td><td><loq< td=""></loq<></td></loq<>	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<loq< td=""></loq<>
2960	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2976		n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2977		n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2979 2980	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 
2980 3100	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
3110	< <u></u>	< <u>5.00</u>	< <u>5.00</u>	< <u>3.00</u>	< <u>5.00</u>	< <u>5</u> .00	< <u></u>	< <u></u>	< <u></u>	< <u>5</u> .00	< <u></u>	< <u>5</u> .00
3116												
3118	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3149												
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154 3160	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.
3172	11.u. < 1	11.u. < 1	11.u. < 1	11.u. < 1	11.u. < 1	11.u. < 1	11.u. < 1	11.u. < 1	11.u. < 1	11.u. < 1	11.u. < 1	11.u. < 1
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.
3185	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5
3192												
3197	<5 <5	<5 <5	<5	<5 <5	<5 <5	<5 <5	<5	<5 <5	<5	<5 <5	<5 <5	<5
3200 3209	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 	<5 
3209												
3210	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3218												
3222												
3230												
3237												
3246 3248	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 	n.d. 
5240												

### Summary of aromatic amines in sample #22596 continued

					_							oAAT+
lab	DDE	DDS	24DAT	TMA	oA	24X	25X	26X	TX	oAAT	oTol	οΤοΙ
210	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
339	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.		n.d.	n.d.	n.d.
362												
551												
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
841	<5	<5	<5	<5	<5	<5		<5	<5	<5	<5	<10
1213	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
1910					 					 		
2102	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2108												
2115 2121												
2121	 <5	 <5	 <5	 <5	 <5	 <5	 <5	 <5	 <5	 <5	 <5	 <5
2129	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	N/A	<5	N/A	<5 <5	<5 <5	N/A
2132			<ul> <li></li> </ul>		~5	<ul> <li></li> </ul>	IN/A	<ul><li></li></ul>	IN//A		<ul> <li></li> </ul>	IN/A
2139												
2165	n.d.	n.d.	n.d.	n.d.	n.d.					n.d.	n.d.	
2170												
2184	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2201	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2213												
2217	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2232												
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2241	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2247	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2256												
2258	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.	n.d.	n.d.	n.d.
2265	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2284	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5	n.d. <5
2286	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2290	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2291	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2293												
2295												
2310	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2311	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2313	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2314										 		
2320	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.	n.d.	n.d.	n.d.
2330	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.	n.d.	n.d.	n.d.
2347 2350	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	<5 <5	 <5	<5 <5	 <5	<5 <5	<5 <5	<5 <5
2350		<b>~</b> 5			<j </j 				<b>~</b> 5			-5
2352												
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	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2367	∼5 n.d.	_5 n.d.	n.d.	∼3 n.d.	∼5 n.d.	∽5 n.d.		n.d.		<5 n.d.	n.d.	
2370	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	N/A	n.d.	n.d.	n.d.	n.d.	n.d.
2373	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2375												
2378	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2379	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.	n.d.	n.d.	n.d.
2380	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2381												
2382	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2386	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0
2389	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2390	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2406	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.	
2410												
2425	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2426	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2429												
2442	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2449												
2453												
2456	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2475												
2476												

		-	•			•	-			-	-	oAAT+
lab	DDE	DDS	24DAT	TMA	οΑ	24X	25X	26X	ТΧ	oAAT	oTol	oTol
2486	n.d.											
2489	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.		n.d.	n.d.	
2492												
2500	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2511 2515												
2515	 <5											
2520												
2528												
2532			n.d.	n.d.	n.d.	n.d.		n.d.		n.d.	n.d.	n.d.
2534	n.d.											
2538	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.		n.d.	n.d.	n.d.
2549	n.d.											
2560	<5	<5	<5	<5	<5	<5		<5		<5	<5	<5
2561												
2565	<5	<5	<5	<5	<5	<5		<5		<5	<5	<5
2567	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2572 2573	<5 n d	<5	<5 n d	<5	<5	<5 n d	<5					
2582	n.d. n.d.											
2590												
2591	n.d.											
2605	<5	<5	<5	<5	<5					<5	<5	<5
2618	n.d.											
2638	n.d.											
2643												
2644												
2649												
2654												
2668	n.d.											
2674 2678	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.		n.d.	n.d.	
2689	n.d. n.d.											
2703												
2740												
2741	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2743	n.d.											
2793	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.	n.d.	n.d.	n.d.
2798	n.d.											
2823												
2827	n.d.											
2829	n.d.											
2852	n.d.											
2864												
2867 2947	n.d.											
2947	n.d. n.d.											
2976	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.			n.d.	
2977	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.		n.d.	n.d.	
2979	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2980												
3100	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00		<5.00		<5.00	<5.00	
3110												
3116												
3118	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3149												
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	n.d.											
3160	n.d.	n.d.	n.d.	n.d. ∠ 1	n.d.							
3172 3176	< 1 	< 1	< 1	< 1	< 1	< 1		< 1		< 1	< 1	
3176	 n.d.											
3185	n.d. [<5]		n.d. [<5]	n.u. 	n.d. [<5]	n.d. [<5]	n.d. [<5]					
3192												
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3200	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3209												
3210												
3214	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		n.d.	n.d.	n.d.	n.d.	n.d.
3218												
3222												
3230												
3237					 n d		 n d	 n d	 n d		 n d	 n d
3246 3248	n.d.											
3248												

### APPENDIX 3 Analytical details sample #22595

/ that y		Sample used as		
	Laboratory	received or further		Technique used to release/extract the
lab	accredited	grinded or cut	Sample intake in grams	analyte(s)
210	Yes		Cample Intake in grains	
			1~	
339			1g	extraction by reflux
362	No	Used as received	1g	Thermal Desorption
551	Yes	Further cut	1g	
623	Yes	Further cut	1 g	Soxhlet
840	Yes	Further cut	0.5	Mechanical Shaking
841	Yes	Used as received	1 gram	Other
1213	Yes	Further cut	0.5g	Other
1910			1 g	Other
2102	No	Used as received	1	Soxhlet
2108	Yes	Used as received	0,5g	Soxhlet
2115	Yes	Used as received	0.5 g	Other
2121	Yes	Used as received	m = 1.00445 g	Other
2129	Yes	Used as received	0,5g	n/a
2132	Yes	Used as received	1 gram	Water bath with temperature control
2135	No	Used as received	0,6	Other
2133	Yes	Used as received	0.508 g	Soxhlet
2165	Yes	Used as received	1.0g	Soxhlet
2170	Yes	Used as received	0.6grams	Mechanical Shaking
2184	Yes	Used as received	1g	Ultrasonic Markania Albakian
2201	Yes	Used as received	0.5g	Mechanical Shaking
2213	Yes	Used as received	Approximately 1.5 gram	
2217	Yes	Used as received	1: 0,5060 g, 2: 1,0003 g	Mechanical Shaking
2232	Yes	Used as received	1g	
2238	Yes	Used as received	0.5g	ASE
2241	Yes	Further cut	0.5g	waterbath
2247	Yes	Used as received	approx. 2gm	Mechanical Shaking
2256	Yes	Further cut	1.0130 g	Thermal Desorption
2258			1.0043	Mechanical Shaking
2265			0.5	Other
2284	Yes	Used as received	0.0	Thermosatic water bath
2286	Yes	Further cut	0.5g	Soxhlet
2290	Yes		0.59	
2290	Yes		0 500	ASE
		Used as received	0.50g	
2293		 Foundly on south	4	 Marahawia di Ohadiya y
2295	Yes	Further cut	1 gram	Mechanical Shaking
2310	Yes	Further cut	1.0	Solid phase extraction
2311	Yes	Further cut	0.5	Thermal Desorption
2313	Yes	Further cut	1.0g	Solid phase extraction
2314	Yes	Further cut	0.5	Mechanical Shaking
2320	Yes	Further cut	0.5g	Mechanical Shaking
2330	Yes	Further cut	0.5 g	Mechanical Shaking
2347			C C	
2350	Yes	Further cut	1.000 g	Soxhlet
2352	Yes	Further cut	0.5g	
2357			J	
2358	Yes	Further cut	1 g	Ultrasonic
2364	Yes	Further cut	0.5021g	
2365	Yes	Used as received		 Other
			1g 0.5	
2366	Yes	Further cut		Other Machanical Shaking
2367	Yes	Used as received	0.5202g	Mechanical Shaking
2370	Yes	Further cut	0.5 g	Mechanical Shaking
2372	Yes	Used as received	1g	used water bath to extract the analyte(s)
2373			0.5g	
2375	Yes	Further cut	1 gr	Water Bath
2378			0.5g	
2379	Yes	Further cut	1 g	Ultrasonic
2380	Yes	Further cut	1.0 g	Mechanical Shaking
2381	Yes	Further cut	0.5 gram per trial	Other
2382	Yes	Used as received	1.0g	water bath extraction
2386	Yes		0,5	
2389	Yes	Used as received	1.0 g	Mechanical Shaking
2389	Yes	Further cut	1g	Mechanical Shaking
2390				
	Yes	Used as received	1 g	Reflux Mechanical Sheking
2410	Yes	Used as received	0.5 g	Mechanical Shaking
2425	Yes	Further cut	0.5g	Mechanical Shaking
2426	Yes	Further cut		Mechanical Shaking
2429	Yes	Used as received	1.0g	Mechanical Shaking
2442	Yes	Further cut	0.5074	Mechanical Shaking

		Sample used as		
	Laboratory	received or further		Technique used to release/extract the
lab	accredited	grinded or cut	Sample intake in grams	analyte(s)
2449			14 -	 Mashaniaal Chalving
2453 2456	Yes Yes	Used as received Used as received	±1g All dispatched sample	Mechanical Shaking
2430	Yes	Used as received		 Other
2476	Yes	Used as received	1.0	
2486			1 gram	ASE
2489	Yes	Further cut	0.5001g	Other
2492	Yes	Used as received	0.5 g	Thermal Desorption
2500	Yes	Used as received	1g	Soxhlet
2511	Yes	Used as received		
2515	Yes	Used as received	0.5 grams	Other
2520 2527	Yes Yes	Used as received Used as received	1 gm	 Machanical Shaking
2527	Yes	Used as received	1g 0.5 grsms	Mechanical Shaking
2520	Yes	Further cut	0.5	Mechanical Shaking
2534	No	Used as received	1 gr for each test	Ultrasonic
2538	Yes	Further cut	1 gram	SPE
2549	Yes	Used as received	1 gm	Mechanical Shaking
2560	Yes	Used as received	0.5 gm	Mechanical Shaking
2561	Yes	Further cut	1	Waterbath
2565	Yes	Used as received	1g	ASE
2567 2572	Yes	Further cut	0.5 gm	Soxhlet
2572 2573	Yes Yes	 Used as received	0.5g	 water bath
2573	Yes	Used as received	0.6013 g	Mechanical Shaking
2590	Yes	Used as received	1g	thermostatic bath as tecniques for extraction
2591	Yes	Further cut	1.0 gram	Thermal Desorption
2605	Yes	Used as received	1.000 g	acc.ISO14362-1
2618	Yes	Used as received	1.0 gm	Mechanical Shaking
2638	No	Used as received	1 gm	water bath
2643	Yes	Used as received		
2644	Yes	Used as received	1 g	Ultrasonic
2649 2654	Yes No	Used as received Used as received	2 1	hot water bath, Rotary evaporator Soxhlet
2668	Yes	Used as received	0.5g	
2674	Yes	Used as received	2.0g	Mechanical Shaking
2678	Yes	Used as received	1 gram	Other
2689	Yes	Further cut	0.5g	Mechanical Shaking
2703	No	Further cut	Approximately 0.6g	Hotplate and water bath
2740	Yes	Used as received	approx. 0,8-1g	not necessary
2741	Yes	Further cut	0.5	Other
2743	Yes	Further cut	1	Soxhlet
2793	No	Used as received	0.5	Mechanical Shaking
2798 2823	Yes 	Used as received	0.5g 1.0142g	Mechanical Shaking Soxhlet
2823	Yes	 Further cut	0.5	Mechanical Shaking
2829	No	Used as received	1	Other
2852	Yes	Used as received	3g	water bath
2864			1 G	Other
2867	Yes	Used as received	0.5g	Thermal Desorption
2947	No	Further cut	1	microwave extraction
2960	Yes	Used as received	0.6grams	Ultrasonic, Mechanical Shaking
2976	Yes	Used as received	0.5g	Thermal Desorption
2977	No	Further cut	2,5 g	Thermal Desorption
2979 2980	No No	Used as received Used as received	0.5 g 1	Mechanical Shaking 
2980 3100	Yes	Used as received	0.5325g	 Other
3110			0.00209	
2.10				Buffer extraction and reductive cleavage in
3116	Yes	Used as received	1 gram	water bath
3118	Yes	Further cut	0.5	Reflux
3149	Yes	Used as received	1g	
3153	Yes	Used as received	0.5 gram	By heating water bath
3154	Yes	Used as received	0,3	Other
3160 3172	Yes	Further cut	0.7	See point 10.2 of standard without extraction
3172 3176	Yes Yes	 Used as received	1	 Water bath.
3170	No	Used as received	0.5 grams.	Ultrasonic
3185	Yes	Further cut	1g	Other
3192	Yes	Further cut	1 g	Look at 10.2 of ISO 14362-1
3197	Yes	Used as received	0,5	Thermal Desorption
3200	Yes	Used as received		Mechanical Shaking
3209	Yes	Used as received	1g	Follow ISO 14362-1:2017
3210	Yes	Used as received	1g	Thermal Desorption
3214	Yes	Further cut	1 g	Ultrasonic Mashaniaal Chaking
3218	Yes	Used as received	0.5g	Mechanical Shaking

	Lobovotowy	Sample used as		Technique used to release (extract the
	Laboratory	received or further		Technique used to release/extract the
lab	accredited	grinded or cut	Sample intake in grams	analyte(s)
3222	Yes	Used as received	1 g	Extraction in thermal bath
3230				
3237	Yes	Used as received	0,5	Ultrasonic
3246	Yes	Used as received	0.5g	Mechanical Shaking
3248	Yes	Used as received	1g	Soxhlet

### Analytical details #22595 continued

lab	Solvent used for release	Extraction time in minutes	Extraction temperature in °C
210 339			
339 362	Xylene	30 min	
551	Xylene	30 mm	
623	Xylene	40 mins	200
840	XYLENE	40MIN	<200
841	METHYL TERT-BUTYL ETHER	45 minutes	40°C
	Sample í first extracted fom the fiber in		
	headspace using xylene under reflux.		
	The extract is treared with sodium		
	dithionite in an alkaline solution at		10
1213	40oC	30 mins	40
1910 2102	Yulana	60	boiling
2102	Xylene Extraction dyestuff in xylene Extraction	00	boling
2108	from reaction solution in MTBE	30 min	40°C
2115		30 min	40° C
2121	O-Xylène	40 minutes	
2129	Xylene	60min	280°C
2132	Sodium hydroxide solution	30 minutes	40 °C
2135	t-Butylmethylether	30	roomtemperatur
2139	Xylene	40 min	Boiling Xylene
2165	Xylene	40 mim.	1000
2170	xylene Xylene	40 min 1 bour	100C Boiling point of vulopo
2184	Xylene	1 hour Reductive 30min and separation	Boiling point of xylene
2201	Colorant extraction with Xylene as per ISO14362-3	30min	70 degrees
2213	Xylene (CAS No 1330-20-7)	30 min to 40 min	60 °C
2217	Xylene (mixture of isomers)	Roughly 25 minutes	150 centigrade
2232	Sodium Hydroxide Solution	30 mins	40
2238	sodium hydroxide solution	30	40
2241	methyl tert-butyl ether	30 minutes	40°C
2247	Tert-Butyl methyl ether	30min	40°C
2256	Xylene	40 mins	
2258			
2265	Vulana	20min	40°C
2284 2286	Xylene xylene	30min Until colorless	40 C Over 150°C
2280	xyiene	Unul coloness	Over 150 C
2291	Xylen (CAS NO>1330-20-7)	30min	45°C
2293	· · · · · · · · · · · · · · · · · · ·		
2295	25 ml Xylene	40 minutes	40 C
2310	Tertiary-butyl Methyl ether	30	40
2311	TBME	30	40
2313	Xylene	30mts	40
2314	TBME	30 mts	40
2320	TBME	45min	40°C <200°C
2330 2347	Xylene	40 min 30min	40°C
2347	Xylene	45 min	about 300 °C
2352	xylene	40mins	/
2357	, -		
2358	Xylene	40 minutes	N/A
2364	Follow ISO14362-3	Follow ISO14362-3	Follow ISO14362-3
2365	methyl tert-butyl ether	45min	25°C
2366	xylene	45	200
2367	t-butyl methyl ether	40min	no control
2370	t-butyl methyl ether/Methanol/Xylene	30 min	40°C
2372 2373	Xylene TMBE	30 45min	70 180°C
2373	Xylene	45min 40 min	55 °C
2373	MTBE	30	40
2379	-	-	-
2380	Xylene	30 Minutes	180-200 °C
2381	Xylene	30 minutes	40 degree
2382	Tert butyl methyl ether (TBME)	30min	40°C
2386			
2389	Tertiary butyl methyl ether	45 min	N/A
2390	Ethyl acetate	15 mints	D affinite to man
2406	Xylene	40 minutes	Reflux temp.
2410 2425	Xylene Xylene	1 hr 30 min.	70 °C 40° C
2425 2426	Xylene Citrate pH 6, Sodium Thionite &TBME	30 min. 75min	Citrate Buffer 70C & SPE Extraction 24C
2420	MTBE	30min	40°C
-			

lab	Solvent used for release	Extraction time in minutes	Extraction temperature in °C
2442	Solvent used for release Xylene	Extraction time in minutes 40 min	200 °C
2442			0
2453			
2456	Xylene' citrate buffer pH 6 / MeOH	30 min then 30 min	Reflux temp then 70 °C
2475	Mada wa	40	450
2476 2486	Xylene	40	150
2480	TBME	30 Minutes	40 Degree
2492	Xylene	30 min	40 °C
2500	xylene	40min	Reflow temperature
2511			
2515	sodium hydroxide	30 minutes	40 °C
2520	Xylene	40 min	70°c
2527 2528	methanol/Dichloromethane Xylene	30min 30 minutes	65
2532	Xylene for colorant extraction	30 minutes in water bath	40°C in water bath
2534	Xylene	30 minuts	70°C
2538		30 min	40°C
2549	Tertiary butyl methyl ether	30 minutes	40 deg C
2560	Xylene	30 minutes	40 °C
2561	Sodium Hydroxide 2%	60	70 250°C
2565 2567	Xylene Xylene	30 30 min	40°C
2572	Xyiene	30 mm	40.0
2573	Xylene	1h	170
	2% Sodium Hydroxide aqueous		
2582	solution	30 minutes	40 °C
2590	xylene	40 min	N/A
2591 2605	xylene/ buffer solution acc.ISO14362-3	40 min acc.ISO14362-3	170 °C Reduction of arylamines at 40°C acc.ISO14362-3
2005	release by Chlorobenzene & extract	acc.13014302-3	acc.150 14502-5
2618	by Methyl tert-butyl ether	30 minutes	40 °C
2638	xylene	40 min	145 C
2643	Xylene	40 min	
2644	Xylene extraction, buffer solution	25 min xylene + 30 min in buffer	boiling point for xylene, 70°C for buffer
0040	Methanol, tert-Butyl methyl ether,	Total 110 minutes	10
2649 2654	Sodium dithionate Xylène	Total 140 minutes 40	40
2668	TBME	30 Minutes	40
2674	xylene	30	40
2678	Xylene	40 min	
2689	MTBE	45mins	room temperature
2703	Xylene	approximately 40 minutes	Xylene extraction set at 200°C on hotplate
2740 2741	Xylene	20min	Steam room /like EN 14362-3
2743	xylene	40	
2793	Sodium dithionite/MTBE	30	40
2798	MTBE	45	RT
2823			
2827 2829	Xylene Xylene	45 mins	60c
2852	NaoH + Sodium Dithionite	30min	40°C
2864		ooniin	10 0
2867	Xylene	60 minutes	132°C
2947	Acetone	75	70
2960	Sodium hydroxide 2%	40min	170°C
2976 2977	Xylene Xylene, TMBE	45 min 45 min + 45 min	40 139°C
2979	xylene	120 min	40 °C
2980	Agiono	120 1111	
3100	t-butyl-methyl ether	45minutes	40°C
3110			
3116	MTBE	30 mins	40°C
3118 3149	xylene Xylene	30 to 40 minutes	70°C
3149	2% NaOH solution	30 minutes	40°C
3154			
3160	Chlorobenzene	40°C	Not controlled
3172			
3176	Xylene / Ethyl-tert. butyl ether	60	70 40 degree celeius
3182 3185	Xylene /	105 minutes.	40 degree celsius /
3105	, Xylene	/ 40 minutes	/ Boiling point of xylene
3197	t-BME	30 min.	40
3200	t-butyl methyl ether	45 min	
3209	MTBE	30min	40°C
3210	pH 6 buffer	60 min	70°C

lab	Solvent used for release	Extraction time in minutes	Extraction temperature in °C
3214	NaOH solution	30 mins	40 degrees
3218	t-butyl-methyl ether	30min	40°C
	Xylene, methanol, NaOH 2%, Sodium		
3222	Hydrosulphite, tert-butyl-methylether	30	40
3230			
3237	xylene	40	200
3246	xylene	until colorless	uncontrol
3248	Xylene	30 minutes	45

# Analytical details #22596

		Sample used as		
	Laboratory	received or further		Technique used to release/extract the
lab	accredited	grinded or cut	Sample intake in grams	analyte(s)
210	Yes		· · · ·	
339	No	Used as received	1g	extraction by reflux
362	Yes	Used as received	1g	Thermal Desorption
551	Yes	Further cut	1g	
623	Yes	Further cut	1 g	Soxhlet
840	Yes	Further cut	0.5	Mechanical Shaking
841	Yes	Used as received	1 grams	Other
1213 1910	Yes Yes	Further cut Used as received	0.5g 1 g	Other Other
2102	No	Used as received	1	Soxhlet
2102	Yes	Used as received	0,5g	Soxhlet
2115	Yes	Used as received	0.5 g	Other
2121	Yes	Used as received	m = 1.00445 g	Other
2129	Yes	Used as received	0,5g	n/a
2132	Yes	Used as received	1 gram	Water bath with temperature control
2135	Yes	Used as received	0,6	Other
2139	Yes	Used as received	0.508 g	Soxhlet
2165	Yes	Used as received	1.0g	Soxhlet
2170	Yes	Used as received	0.6grams	Mechanical Shaking
2184 2201	Yes Yes	Used as received Used as received	1g 0.5g	Ultrasonic Mechanical Shaking
2201	Yes	Further cut	Approximately 1.5 gram	
2213	Yes	Used as received	1: 0,5060 g, 2: 1,0003 g	 Mechanical Shaking
2232	Yes	Used as received	1g	
2238	Yes	Used as received	0.5g	ASE
2241	Yes	Used as received	0.5g	waterbath
2247	Yes	Further cut	approx. 2gm	Mechanical Shaking
2256	Yes	Further cut	1.0130 g	Thermal Desorption
2258	No	Further cut	1.0043	Mechanical Shaking
2265	Yes	Used as received	0.5	Other
2284	Yes	Used as received	0.5-	Thermosatic water bath
2286 2290	Yes Yes	Further cut	0.5g	Soxhlet 
2290	Yes	 Used as received	0.50g	ASE
2293			0.009	
2295	Yes	Further cut	1 gram	Mechanical Shaking
2310	Yes	Further cut	1.0	Solid phase extraction
2311	Yes	Further cut	0.5	Thermal Desorption
2313	Yes	Further cut	1.0g	Solid phase extraction
2314	Yes	Further cut	0.5	Mechanical Shaking
2320	Yes	Further cut	0.5g	Mechanical Shaking
2330	Yes	Further cut	0.5 g	Mechanical Shaking
2347 2350	 Yes	 Further cut	1 000 a	 Soxhlet
2350	Yes	Further cut	1.000 g	
2352			0.5g	
2358	Yes	Further cut	1 g	Ultrasonic
2364	Yes	Further cut	0.5021g	
2365	Yes	Used as received	1g	Other
2366	Yes	Further cut	0.5	Other
2367	Yes	Used as received	0.5202g	Mechanical Shaking
2370	Yes	Further cut	0.5 g	Mechanical Shaking
2372	Yes	Used as received	1g	used water bath to extract the analyte(s)
2373	 Voo	 Further out	0.5g	 Watar Bath
2375 2378	Yes 	Further cut	1 gr 0 5g	Water Bath 
2378	 Yes	 Further cut	0.5g 1 g	 Ultrasonic
2380	Yes	Further cut	1.0 g	Mechanical Shaking
2381	Yes	Further cut	0.5 gram per trial	Other
2382	Yes	Used as received	1.0g	water bath extraction
2386	Yes	Used as received	0,5ັ	
2389	Yes	Further cut	1.0 g	Mechanical Shaking
2390	Yes	Further cut	1g	Mechanical Shaking
2406	Yes	Used as received	1 g	Reflux
2410	Yes	Further cut	0.5 g	Mechanical Shaking
2425	Yes	Further cut	0.5g	Mechanical Shaking
2426 2429	Yes	Further cut	1.00	Mechanical Shaking
2429 2442	Yes Yes	Further cut Further cut	1.0g 0.5074	Mechanical Shaking Mechanical Shaking
2442 2449	res 		0.0014	
2445	Yes	Used as received	±1g	 Mechanical Shaking
2456	Yes	Used as received	All dispatched sample	
2475	Yes	Used as received	0.5011	Other

		Sample used as		
	Laboratory	received or further		Technique used to release/extract the
lab	accredited	grinded or cut	Sample intake in grams	analyte(s)
2476	Yes	Used as received	1.0	
2486	Yes	Used as received	1 gram	ASE
2489	Yes	Further cut	0.5001g	Other
2492	Yes	Used as received	0.5 g	Thermal Desorption
2500	Yes	Used as received	1g	Soxhlet
2511	Yes	Used as received	0.5	
2515	Yes	Used as received	0.5 grams	Other
2520	Yes	Used as received	1 gm	 Machaniaal Chaking
2527 2528	Yes Yes	Used as received Used as received	1g 0 E gromo	Mechanical Shaking
2520	Yes	Further cut	0.5 grsms 0.5	 Mechanical Shaking
2532	Yes	Used as received	1 gr for each test	Ultrasonic
2534	Yes	Further cut	1 gram	SPE
2538	Yes	Used as received	1 gm	Mechanical Shaking
2560	Yes	Used as received	0.5 gm	Mechanical Shaking
2561	Yes	Further cut	1	Waterbath
2565	Yes	Further cut	1g	ASE
2567	No	Further cut	0.5 gm	Soxhlet
2572	Yes		0.0 g	
2573	Yes	Used as received	0.5g	water bath
2582	Yes	Further cut	0.6013 q	Mechanical Shaking
2590	Yes	Used as received	1g	thermostatic bath as tecniques for extraction
2591	Yes	Further cut	1.0 gram	Thermal Desorption
2605	Yes	Used as received	1.000 g	acc.ISO14362-1
2618	Yes	Used as received	1.0 gm	Mechanical Shaking
2638	No	Further cut	1 gm	water bath
2643			-	
2644	Yes	Used as received	1 g	Ultrasonic
2649	Yes	Further cut	2	hot water bath, Rotary evaporator
2654	Yes	Used as received	1	Soxhlet
2668	Yes	Used as received	0.5g	
2674	Yes	Used as received	2.0g	Mechanical Shaking
2678	Yes	Used as received	1 grams	Other
2689	Yes	Further cut	0.5g	Mechanical Shaking
2703	Yes	Further cut	Approximately 0.6g	Hotplate and water bath
2740	Yes	Used as received	approx. 0,8-1g	not necessary
2741	Yes	Further cut	0.5	Other
2743	Yes	Further cut	1	Soxhlet
2793	No	Used as received	0.5	Mechanical Shaking
2798	Yes	Used as received	0.5g	Mechanical Shaking
2823	No	Used as received	1.0142g	Soxhlet
2827	Yes	Further cut	0.5	Mechanical Shaking
2829 2852	No	Further cut	1	Other
	Yes	Used as received	3g	water bath
2864	Yes	Used as received	1 G	Other Thermel Description
2867	Yes	Used as received Further cut	0.5g	Thermal Desorption
2947 2960	No	Used as received	1 0.6grams	microwave extraction
2960 2976	Yes Yes	Further cut	0.5g	Ultrasonic, Mechanical Shaking Thermal Desorption
2976 2977	res No	Further cut	0.5g 2,5 g	Thermal Desorption
2979	No	Used as received	2,3 g 0.5 g	Mechanical Shaking
2979	No	Used as received	0.5 g 1	
3100	Yes	Further cut	0.5325g	 Other
3110			0.00209	
3116	Yes	Used as received	1 gram	Buffer extract. reductive cleavage water bath
3118	Yes	Further cut	0.5	Reflux
3149	Yes	Further cut	1g	
3153	Yes	Used as received	0.5 gram	By heating water bath
3154	Yes	Used as received	0,3	Other
3160	Yes	Further cut	0.7	See point 10.2 of standard without extraction
3172				
3176	Yes	Further cut	1	Water bath.
3182	Yes	Used as received	0.5 grams.	Ultrasonic
3185	Yes	Further cut	1g	Other
3192	Yes	Further cut	1 g	Look at 10.2 of ISO 14362-1
3197	Yes	Further cut	0,5	Thermal Desorption
3200	Yes	Used as received		Mechanical Shaking
3209	Yes	Used as received	1g	Follow ISO 14362-1:2017
3210	Yes	Used as received	1g	Thermal Desorption
3214	Yes	Further cut	1 g	Ultrasonic
3218	Yes	Used as received	0.5g	Mechanical Shaking
3222	Yes	Used as received	1 g	Extraction in thermal bath
3230			o 5	
3237	Yes	Used as received	0,5	Ultrasonic
3246	Yes	Further cut	0.5g	Mechanical Shaking
3248	Yes	Used as received	1g	Soxhlet

# Analytical details #22596 continued

lab	Solvent used for release	Extraction time in minutes	Extraction temperature in °C
210			
339	xylene	45 min	200
362	Methyl tert-butyl ether	60 min	70°C
551	Ethyl acetate	10	000
623	Xylene	40	200
840	buffer 6.0	1hour	70
841	citrate/ etylacetate	15 minutes	70°C
1010	sodium dithionite in an in a'citrate-		70
1213	bufferect aqueos solution (pH = 0)	1 hours	70
1910	Yulara a	40	h a llin a
2102	Xylene	40 20 min	boiling
2108	Methanol	30 min	70°C
2115 2121			
2121	Method without extraction used.	Method without extraction used.	Method without extraction used.
2129	Citric Buffer	30 minutes	70 °C
2132	t-Butylethylether , SPE	60	70
2133	Xylene	40 min	Boiling Xylene
2165	Xylene	40 min.	Dolling Aylene
2170	Xylene	40 min	100C
2184	pH 6 buffer	30 minutes	70 °C
2104	Extraction as per ISO14362-1:2017	Recuctive 30min and separation	10 6
2201	Section 10.2	30min	70 degrees
2201	00000110.2		
2210	Citrate puffer defined in EN 14362-1	30 minutes	70 centigrade
2232	Citrate Buffer solution	Total of 1hr	70
2238	citrate buffer solution	30min	70
2241	methyl tert-butyl ether	30 minutes	70°C
2247	Tert-butyl methyl ether	30min	70°C
2256	Citrate buffer solution	30 mins	70°C
2258	MTBE	1 Hrs	70
	Citrat-NaOH buffer Sodium dithionite	1) 30 min in ultrasonic bad 2) 30	
2265	NaOH(40%) TBME NaCl (7g)	min in ultrasonic bad	70°C
2284	Xylene	30min	70°C
	,	until the solvent dripping becomes	
2286	xylene	colorless.	Over 150°C
2290			
	Citrate/sodium hydroxide buffer		
2291	solution	30min	75°C
2293			
2295	Xyline	40 minutes	40 C
2310	Tertiary-butyl Methyl ether	60	70
2311	TBME	30	70
2313	Tertbutylmethylether	60mts	70
2314	TBME	30 mts	70
2320	Ethyl acetate	15min	70°C
2330	Ethyl acetate and Citric buffer	15 min and 60 min	70°C
2347			
2350	Xylene	45 min	about 300 °C
2352	citrate solution	60	70
2357			
0050	Citrate buffer (pH 6; preheated to	20 minutos	70°C
2358 2364	70°C) Follow ISO14362-1	30 minutes	Follow ISO14362-1
2364	ethyl acetate	Follow ISO14362-1	
2365	citrate buffer solution	15min 30	25℃ 70
2367	Ethyl acetate	no need because sample is cotton	no need because sample is cotton
2370	t-butyl methyl ether/ACN	60 min	70°C
2010	Sodium citrate dihydrate buffer		10.0
2372	solution	30	70
2373	TMBE	50	70°C
2375	IMBE		10.0
2378	МТВЕ	30	70
2379	-	•	•
2380	Ethyl acetate	60 Minutes	70 °C
	citrate Buffer, sodium dithionite, 40%		- <del>-</del>
	NaOH, 5ml+7g Nacl salt+ 5ml Ethyl		
2381	acetate	60 minutes in OVEN	70 degree temperature in Oven.
2382	Ethyl acetate	60min	70°C
2386	,		
2389	Ethyl Acetate	15 min	70
2390	Ethyl Acetate	15 mints	Not applicable
2406	Xylene	40 minutes	Reflux temp.
2410	citrate buffer	30 min	70 °C

lab	Solvent used for release	Extraction time in minutes	Extraction temperature in °C
2425	Methanol, Citrate Buffer	1 hour	70°C
	Citrate Buffer $pH = 6$ , Sodium Thoinite		Citrate Extraction at 70C & SPE Extraction
2426	& TBME	75min	at 24C
2429	MTBE	30min + 30min	70°C
2442	t-butyl methyl ether	1hr. 15 min	70
2449			
2453			
2456	Citrate buffer pH 6	30 min	70 °C
2475			
2476			
2486	Tert. Butyl methyl ether	60 minutes	70 °C
2489	Citrate buffer/TBME	60 minutes	70 degree
2492	pH 6.0 buffer	30 min	70 °C
2500	xylene	40min	Reflow temperature
2511	<b>.</b>		
2515	citrate buffer solution	60 minutes	70 °C
2520			
0507	Sodium sitrate anhydrous / NaOH		70
2527	Solution / pH 6	30min / 30min	70
2528	TOME	A have be seen to a hardle	70900
2532	TBME	1 hour in water bath	70°C?
2534	TMBE (Tert-butyl methyl ether)	30 minuts	70°C
2538 2549	Tertien, but dimethod other	2 x 30 min	70°C
2549 2560	Tertiary butyl methyl ether	60 mins	70 deg C
	TBME	60 minutes	70°C 70
2561		60	
2565	Buffer solution	30分	70°C
2567	Xylene	30 min	70°C
2572	situate (a solicius la columniale la c <b>ff</b> an		
0570	citrate/sodium hydroxide buffer	0.5h	70
2573	solution	0.5h	70
2582	Citric Buffer	30 minutes	70 °C
2590 2591	mtbe	60min	70°C 70°C
2605	acc.ISO14362-1	30 min acc.ISO14362-1	acc.ISO14362-1
2605		60 minutes	70 °C
2638	Methyl tert-butyl ether TBME	1 HR	70 C
2643	IDME		100
2644	buffer solution	30 min	70°C
2044	buffer solution, methanol, tert-Butyl	30 1111	10.0
2649	methyl ether, Sodium Dithionate	total 100 min	70
2654	Xylène	40	10
2668	Methanol	30 Minutes	70
2674	xylene	30	70
2678	sodium dithionite		70
2689	MTBE	15mins	Room temperature
2703	Xylene, citrate buffer	40 mins, Citrate buffer 1 hr total	Xylene extraction 200°C, Citrate buffer 70°C
2740	not necessary	not necessary	not necessary
2741	······	·····,	······································
2743	TBME	40 min + 30 min	70 °C
2793	Sodium dithionite MTBE	30	70
2798	MTBE	15min	RT
2823	Xylene.	30mins.	230°C.
2827	Tertiary butyl methyl ether	30 mins	70c
2829	Citrate buffer (pH6)	30	70
2852	citrate/ NaOH pH=6 ,Sodium Dithionite	30min	70°C
2864	Xylene	60 minutes	140
2867	MTBE	15minutes	Room temperature
2947	Acetone	75	70
	Citrate/sodium hydroxide buffer		
2960	solution	30min	70°C
2976	TBME	30 min	70
2977	TMBE	30 min	Room temperature
2979	as method	120 min	70 °C
2980		30	70
3100	t-butyl-methyl ether	30minutes	70°C
3110			
3116	Buffer followed by MBTE	30 mins	70 °C
3118	xylene	30 to 40 minutes, until colourless.	70°C
3149			
3153	Citrate/NaOH buffer solution	30 minutes + 30 minutes	70°C
3154	Citrate-Buffer pH 6, 0,06 mmol/l	30 min	70 °C
3160	See point 10.2 std without extraction	-	-
3172			
3176	Ethyl-tertbutyl ether	60	70
3182			70 degree celsius.
3185	/	/	/

lab	Solvent used for release	Extraction time in minutes	Extraction temperature in °C
3192	Buffer (7.5)	30 minutes	70°C
3197	t-BME	60 min.	70
3200	t-butyl methyl ether	30 min	70°C
3209	MTBE	60min	70°C
3210	pH6 buffer	60 min	70°C
3214	Citric Acid/NaOH buffer	30 mins	70 degrees
3218	t-butyl-methyl ether	30min	70°C
	Buffer pH 6, sodiun Hydrosulphite,		
3222	tert-butyl-methyl-ether and acetonytrile	60	70°C
3230			
3237	Buffer sitrat	60	70
3246	acid buffer solution (preheated 70°C)	30±1 min	70±2°C
3248			

### Analytical details #22596 continued

lab	ISO14362-1 chapter 10.4 or Annex E followed and used or not used the diatomaceous earth column
210	
339	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
362	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
551	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
623 840	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
840 841	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
1213	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
1910	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2102	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2108	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2115 2121	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2121	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2132	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2135	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2139	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2165	 I fellowed ISO14262 1 chapter 10.4 and wood the distances we parth column
2170 2184	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2201	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2213	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2217	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2232	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2238 2241	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2241	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2256	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2258	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2265	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2284	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2286 2290	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2291	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2293	
2295	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2310	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2311 2313	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2313	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2320	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2330	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2347	
2350	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2352 2357	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2358	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2364	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2365	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2366	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2367 2370	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2372	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2373	
2375	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2378	
2379 2380	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2380	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2382	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2386	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2389	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2390	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2406 2410	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2425	I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column
2426	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2429	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2442	
2449 2453	 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2455	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2475	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column
2476	I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column

ISO14362-1 chapter 10.4 or Annex E followed and used or not used the diatomaceous earth column lab 2486 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2489 2492 use the diatomaceous earth column followed GB/T 17592 Chapter 6.2.1 2500 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2511 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2515 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2520 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2527 2528 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2532 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2534 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2538 I followed a different test method 2549 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2560 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2561 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2565 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2567 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2572 2573 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2582 2590 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2591 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2605 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2618 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2638 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2643 2644 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2649 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2654 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2668 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2674 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2678 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2689 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2703 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2740 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2741 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2743 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2793 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2798 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2823 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2827 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2829 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2852 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2864 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2867 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 2947 I followed a different test method 2960 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2976 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2977 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2979 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 2980 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 3100 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3110 3116 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3118 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3149 3153 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3154 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3160 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3172 3176 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3182 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3185 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3192 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3197 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3200 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3209 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3210 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3214 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3218 3222 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column 3230 3237 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 3246 I followed ISO14362-1 Annex E and did NOT use the diatomaceous earth column 3248 I followed ISO14362-1 chapter 10.4 and used the diatomaceous earth column

#### **APPENDIX 4**

#### Number of participants per country

1 lab in AUSTRIA 8 labs in BANGLADESH 1 lab in BRAZIL 1 lab in BULGARIA 2 labs in CAMBODIA 4 labs in EGYPT 4 labs in FRANCE 10 labs in GERMANY 2 labs in GUATEMALA 9 labs in HONG KONG 1 lab in HUNGARY 11 labs in INDIA 2 labs in INDONESIA 10 labs in ITALY 3 labs in JAPAN 5 labs in KOREA, Republic of 1 lab in MAURITIUS 3 labs in MOROCCO 31 labs in P.R. of CHINA 5 labs in PAKISTAN 1 lab in POLAND 1 lab in PORTUGAL 2 labs in SINGAPORE 2 labs in SPAIN 2 labs in SRI LANKA 4 labs in TAIWAN 2 labs in THAILAND 1 lab in THE NETHERLANDS 3 labs in TUNISIA 5 labs in TURKEY 3 labs in UNITED KINGDOM 9 labs in VIETNAM

#### **APPENDIX 5**

#### Abbreviations

С	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
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

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
- 5 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 7 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 8 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
- 10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, <u>127</u>, 1359-1364, (2002)
- 11 W. Horwitz and R. Albert, J. AOAC Int, <u>79.3</u>, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, <u>25(2)</u>, 165-172, (1983)
- 13 iis memo 2202: reproducibility of AZO Dyes in Leather/Footwear andTextiles in iis PTs