

**Results of Proficiency Test  
AZO dyes in leather  
February 2017**

**Organised by:** Institute for Interlaboratory Studies (iis)  
Spijkenisse, the Netherlands

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## **1 INTRODUCTION**

The Institute for Interlaboratory Studies (iis) organizes every year a proficiency test for banned AZO dyes in leather since 1997, with an exception in 2009. During the annual proficiency testing program 2016/2017, it was decided to continue the proficiency test for the analysis of banned AZO dyes in leather. In this interlaboratory study, 142 laboratories in 34 different countries registered for participation (see appendix 4). In this report, the results of the 2017 proficiency test are presented and discussed. This report is also electronically available through the iis website [www.iisnl.com](http://www.iisnl.com).

## **2 SET UP**

The Institute for Interlaboratory Studies (iis) in Spijkenisse was the organizer of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. Due to lack of a sufficient amount of suitable materials it was decided to send in this proficiency test only one leather sample, positive on banned AZO dyes, labelled #17520.

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/IEC 17043:2010 (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 organisation 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 March 2017 (iis-protocol, version 3.4). This protocol can be downloaded from the iis website [www.iisnl.com](http://www.iisnl.com), from the FAQ page.

### **2.3 CONFIDENTIALITY STATEMENT**

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

## 2.4 SAMPLES

A suitable black/green leather sample, dyed with Direct Red 28, was obtained from a third party laboratory. The leather material was mixed thoroughly after cutting it into small pieces. In total 180 subsamples with 3.5 gram material were prepared and labelled #17520. Eight stratified randomly selected samples were tested using an in-house test method to check the homogeneity of the batch. See the following table for the test results.

	<i>Benzidine in mg/kg</i>
sample #17520-1	81.6
sample #17520-2	74.1
sample #17520-3	86.9
sample #17520-4	77.8
sample #17520-5	79.4
sample #17520-6	81.7
sample #17520-7	89.1
sample #17520-8	73.9

Table 1: homogeneity test results of subsamples #17520

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

	<i>Benzidine in mg/kg</i>
r (observed)	15.4
reference test method	ISO17234-1:2015
r (reference test method)	23.5
0.3 * R (ref. test method)	14.0

Table 2: evaluation of the repeatability of the aromatic amine in subsamples #17520

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

One sample containing approximately 3.5 grams testing material labelled #17520 was sent to each of the participating laboratories on February 8, 2017. A letter of instructions was added to the sample package.

## 2.5 ANALYSES

The participants were asked to determine the concentrations of 23 forbidden aromatic amines and *o*-anisidine, applying the analysis procedure that is routinely used in the laboratory. Also some analytical details were requested to be reported.

It was explicitly requested to treat the sample as if it was a routine sample, but not to age or to dry the sample.

It was also requested to report the test results using the indicated units on the report form and not to round the test results, but to 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 evaluation.

To get comparable test results a detailed report form and a letter of instructions are prepared. The detailed report form and the letter of instructions are both made available on the data entry portal [www.kpmd.co.uk/sgs-iis-cts/](http://www.kpmd.co.uk/sgs-iis-cts/). The participating laboratories were 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.iisn.com](http://www.iisn.com).

## 3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that did not report 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 reanalyses). Additional or corrected test results are used for the data analysis and the 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 organisation 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 March 2017 (iis-protocol, version 3.4).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<... ' or '>... ' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

In accordance to ISO 5725 the original test results per determination were submitted subsequently 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. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them 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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

### 3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In general when no literature reproducibility is available, another target may be used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used. This should be done in order to evaluate whether the reported test results are fit-for-purpose.

The z-scores were calculated in accordance with:

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

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

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

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

## 4 EVALUATION

During the execution of this proficiency test some reporting problems occurred. Three participants were not able to report in time for the evaluation of the test results. Five participants reported test results after the deadline but were on time for the evaluation of the test results. The 139 participants did report 143 numerical test results in total. Observed were 7 outlying test results, which is 4.9% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

The data set of the positive aromatic amine proved to have a normal Gaussian distribution.

### 4.1 EVALUATION PER COMPONENT

In this section, the test results are discussed per component. All statistical results reported for Benzidine are listed in appendix 1. The abbreviations used in this table are listed in appendix 5. The target reproducibility for Benzidine is estimated over the three reproducibilities mentioned for Benzidine in table 2 of test method ISO 17234-1:2015. The reported test results of all other aromatic amines are listed in appendix 2.

#### Benzidine (CASno. 92-87-5):

The determination of this aromatic amine at a concentration level of 51 mg/kg was not problematic. Seven statistical outliers were observed and one another test result was excluded.

However, the calculated reproducibility after rejection of the suspect data is in agreement with the reproducibility requirement estimated from the reference test method ISO 17234-1:2015.

#### General:

Four participants reported the presence of other aromatic amines at various concentration levels.

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

Component	unit	n	Average	2.8 * sd	R(target)
Benzidine	mg/kg	126	51	29	30

Table 3: reproducibility of the aromatic amine in leather sample #17520

Without further statistical calculations, it can be concluded that the group of participating laboratories has no problem with the analysis of Benzidine in leather at this level.



### 4.3 COMPARISON WITH PREVIOUS INTERLABORATORY STUDIES

The observed variation in the test results for the aromatic amines in the 2017 PT is in agreement in comparison with the variation of the aromatic amine as observed in the previous PTs, see below table.

<i>Component</i>	<i>Feb 2017</i>	<i>Feb 2016</i>	<i>March 2015</i>	<i>March 2014</i>	<i>March 2013</i>	<i>March 2012-2005</i>	<i>Target ISO17234-1</i>
4-Aminodiphenyl	n.e.	n.e.	n.e.	n.e.	n.e.	25 - 45%	15 - 33%
Benzidine	20%	34%	n.e.	20%	28%	20 - 66%	15 - 25%
3,3'-Dimethylbenzidine	n.e.	39%	24%	n.e.	n.e.	45 - 55%	17 - 24%
<i>o</i> -Toluidine	n.e.	37%	n.e.	n.e.	n.e.	50 - 63%	30 - 37%
2,4-Xylidine	n.e.	n.e.	n.e.	n.e.	36%	16 - 19%	15 - 33%

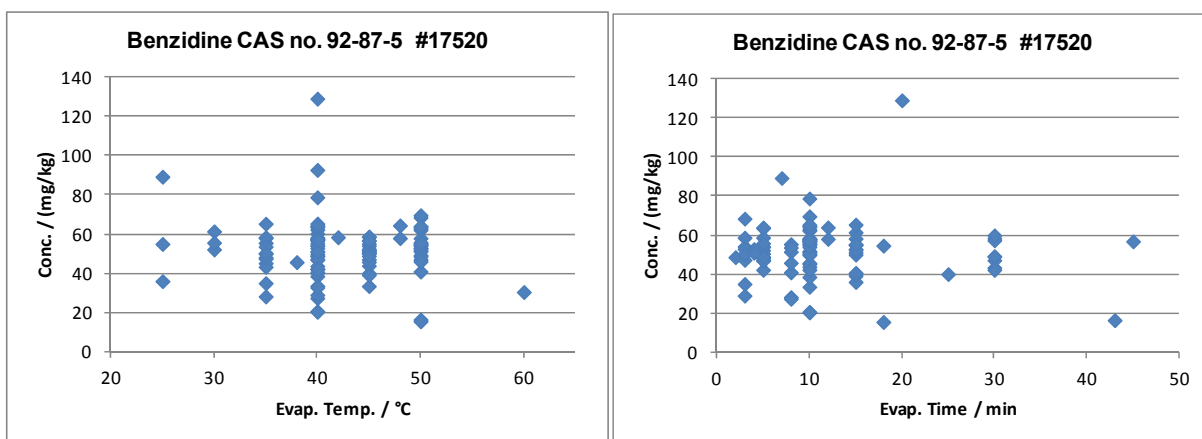
Table 4: development of the uncertainties over the years

## 5 DISCUSSION

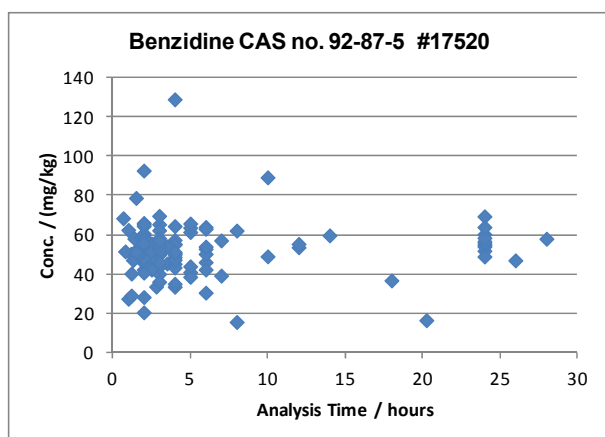
In this PT also some analytical details were asked (see appendix 3) to use for further statistical analyses. It appeared that 94% of the participants is accredited for the determination of aromatic amines. About 95% of the participants degreased the sample with n-Hexane. No separate statistical analysis has been performed over these two parameters.

Another question was about to what amount the extraction solution was evaporated. It appeared that 102 participants (83%) mentioned; "to a small residual quantity" (92 participants) or "to a certain quantity" (10 participants). No difference in quantity was mentioned in case a quantity was specified. Nine participants mentioned to evaporate "to complete dryness". Test method ISO 17234-1:2015 mentioned explicitly not to evaporate to complete dryness, however to blow to dryness with an inert Gas like Nitrogen. Seven of those nine participants reported a sufficient amount of Benzidine. The other two test results were either an outlier or reported as <15 mg/kg. These two participants had obviously lost the Benzidine during the analytical process. Another participant, whom test result was excluded in the statistical analysis, reported that presumably the aromatic amines were deteriorated due to high temperature during cleavage.

Test method ISO 17234-1:2015 mentioned that the temperature during the evaporation of MTBE should not exceed 50°C, otherwise the aromatic amines may deteriorate and may not be detected anymore. Also the time needed between reduction and actual analysis may be important. It appeared that 111 participants of the 114 who had answered this question did use a temperature <50° and 87 participants did the evaporation in ≤ 20 minutes. No clear relation was observed between these parameters and amount of Benzidine measured in mg/kg, see next two pictures.



The above mentioned mild conditions are necessary because aromatic amines are not very stable and should be determined after the reductive cleavage with-in 24 hours. However, seven participants reported a total duration of 48 or 72 hours. It appeared that two of these seven lost the Benzidine and reported “not detected”. Nevertheless, no correlation could be found between duration of the analytical process as reported by the laboratories and the test results (see picture below).



Therefore, it can be concluded that the observed variation in this interlaboratory study is not caused by just one critical point in the analysis but by a combination of experimental conditions. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

**APPENDIX 1**

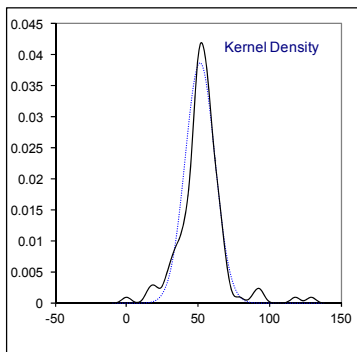
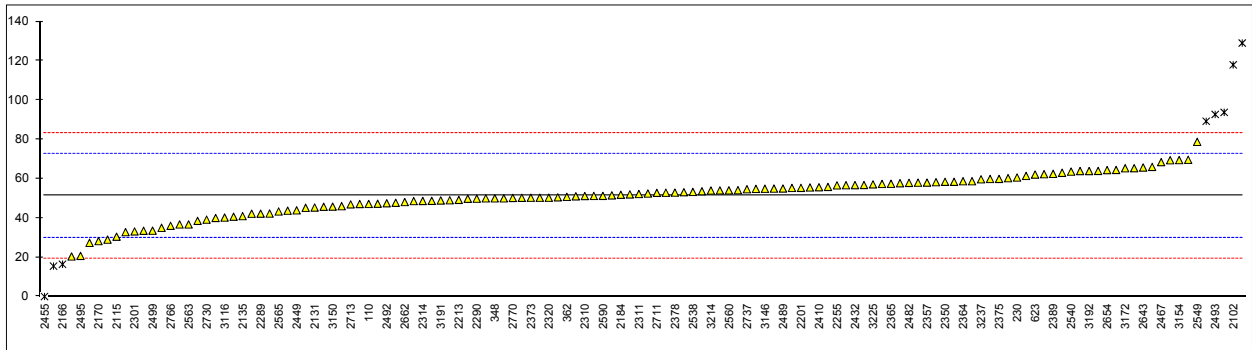
Determination of Benzidine (CASno.92-87-5) in sample #17520; test results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO17234-1	47.12		-0.40	
213	ISO17234-1	69.38		1.69	
230	ISO17234-1	60.6		0.87	
348	In house	50.00		-0.13	
362	ISO17234-1	50.72		-0.06	
551	ISO17234-1	93.78	C,R(0.05)	3.98	first reported: 102.83
623	ISO17234-1	62.094		1.01	
840	ISO17234-1	48.60		-0.26	
841		47.74		-0.34	
2102	EN14362-1	117.96	R(0.01)	6.25	
2115	ISO17234-1	30.47		-1.96	
2129		49.0		-0.22	
2131	EN14362-1	45.315		-0.57	
2132	ISO17234-1	50		-0.13	
2135	EN14362-1	40.92		-0.98	
2139	EN14362-1	66		1.37	
2165	ISO17234-1	54.8		0.32	
2166	EN14362-1	16.42	R(0.05)	-3.28	
2170	ISO17234-1	28.21		-2.17	
2172	ISO17234-1	36.70		-1.38	
2184	ISO17234-1	51.8		0.04	
2201	ISO17234-1	55.32		0.37	
2213	ISO17234-1	49.2		-0.20	
2228	ISO17234-1	27.34		-2.26	
2230	ISO17234-1	49.8		-0.15	
2241	ISO17234-1	33.49		-1.68	
2247		52.85		0.14	
2255	ISO17234-1	56.6		0.49	
2256	ISO17234-1	-----		-----	
2271	ISO17234-1	50.5		-0.08	
2289	ISO17234-1	42.2		-0.86	
2290	ISO17234-1	49.83		-0.14	
2295		40		-1.07	
2301	In house	33.16		-1.71	
2310	ISO17234-1	51.17		-0.02	
2311	ISO17234-1	52.19		0.08	
2314	ISO17234-1	48.66		-0.25	
2320	ISO17234-1	50.3058		-0.10	
2330	ISO17234-1&3	60.26		0.83	
2347	ISO17234-1	52		0.06	
2350	ISO17234-1	58.4		0.66	
2352	ISO17234-1	54.0		0.25	
2357	ISO17234-1	58.01		0.62	
2358	ISO17234-1	54.97		0.34	
2364	ISO17234-1	58.7		0.69	
2365	ISO17234-1	57.40		0.57	
2366	ISO17234-1	51.5		0.01	
2367	ISO17234-1	54.14		0.26	
2369	ISO17234-1	57.33		0.56	
2370	ISO17234-1&2	48.72		-0.25	
2373	ISO17234-1	50.3		-0.10	
2375	ISO17234-1	59.87		0.80	
2378	ISO17234-1	52.9		0.14	
2379	ISO17234-1	38.533		-1.20	
2380	ISO17234-1	63.96		1.18	
2381	ISO17234-1	63.00		1.09	
2386	ISO17234-1	61.4		0.94	
2389	ISO17234-1	62.47		1.04	
2390	ISO17234-1	59.86		0.80	
2403	ISO17234-1	47.1		-0.40	
2410	ISO17234-1	55.63		0.40	
2426		65.34		1.31	
2429	ISO17234-1	43.7		-0.72	
2432	ISO17234-1	56.72		0.50	
2442	In house	55.52		0.39	
2449	ISO17234-1	43.8703		-0.70	
2455	ISO17234-1	0.0	ex	-4.82	excluded: zero is not a real value
2459		-----		-----	
2467	ISO17234-1	68.43		1.60	
2472	ISO17234-1	45.69		-0.53	
2476	ISO17234-1	62.301		1.03	
2477	ISO17234-1	n.d.		-----	possibly a false negative test result?

lab	method	value	mark	z(targ)	remarks
2482	ISO17234-1	57.865		0.61	
2488	ISO17234-1	63.90	C	1.18	first reported: 423.72
2489	ISO17234-1	55		0.34	
2492	ISO17234-1	47.5		-0.36	
2493	ISO17234-1	92.71	R(0.05)	3.88	
2495	ISO17234-1	20.68		-2.88	
2497	ISO17234-1	129.06	C,R(0.01)	7.29	first reported: 259.14
2499	GB/T19942	33.54		-1.67	
2511	ISO17234-1	45.17		-0.58	
2514	ISO17234-1	57.97		0.62	
2516		-----		-----	
2532	EN14362-1	58.2		0.64	
2538	ISO17234-1	53.26		0.18	
2540	EN14362-1	63.6		1.15	
2549	ISO17234-1	78.76		2.57	
2553	ISO17234-1	28.97		-2.10	
2560	ISO17234-1	54.08		0.25	
2561	ISO17234-1	15.494	R(0.05)	-3.37	
2563	ISO17234-1	36.7		-1.38	
2565		43.3		-0.76	
2569	ISO17234-1	46		-0.50	
2572	ISO17234-1	53.12		0.16	
2590	ISO17234-1	51.23		-0.01	
2592	ISO17234-1	32.77		-1.75	
2597	ISO17234-1	50.3		-0.10	
2614	ISO17234-1	47.26		-0.39	
2624	EN14362-1	n.d.		-----	possibly a false negative test result?
2629	ISO17234-1	64.45	C	1.23	first reported: 138.86
2639	GB/T19942	52.45		0.10	
2643	In house	65.69		1.34	
2654	ISO17234-1	64.38		1.22	
2656		-----		-----	
2662	ISO17234-1	48.10		-0.31	
2666	ISO17234-1	56.66		0.50	
2674	ISO17234-1	58.4		0.66	
2695	ISO17234-1	20.39		-2.91	
2711	ISO17234-1	52.78	C	0.13	first reported: 95.12
2713	ISO17234-1	46.91383		-0.42	
2719	ISO17234-1	58.7		0.69	
2727	ISO17234-1	<15		<-3.41	possibly a false negative test result?
2730	ISO17234-1	39.138		-1.15	
2737		54.62		0.30	
2738		51.2		-0.02	
2741	ISO17234-1	56.79		0.51	
2749	ISO17234-1	89.25	R(0.05)	3.55	
2756	ISO17234-1	n.d.		-----	possibly a false negative test result?
2766	ISO17234-1	36.0		-1.44	
2770	GB/T19942	50.2		-0.11	
3116	ISO17234-1	40.2		-1.05	
3146	ISO17234-1	54.8775		0.33	
3150	ISO17234-1	45.79		-0.52	
3151	ISO17234-1	42.17		-0.86	
3153	ISO17234-1	53.6		0.21	
3154	ISO17234-1	69.45	C	1.70	first reported: 103.46
3160	ISO17234-1	69.62		1.71	
3172	EN14362-1	65.32		1.31	
3185	ISO17234-1	50.26		-0.10	
3191	ISO17234-1	48.9		-0.23	
3192	ISO17234-1	63.9		1.18	
3197	ISO17234-1	57.7		0.59	
3200	ISO17234-1	55.8		0.42	
3209	ISO17234-1	51.02		-0.03	
3210	In house	40.65		-1.01	
3214	ISO17234-1	53.97		0.24	
3216	ISO17234-1	42.24		-0.86	
3220	ISO17234-1	34.98		-1.54	
3225	ISO17234-1	57.1		0.54	
3228	ISO17234-1	55.3		0.37	
3237		59.69		0.78	
3248	ISO17234-1	50		-0.13	

Lab 2455 reported: Sample may have been lost due to overheating of the sand bath during cleavage

normality	OK
n	126
outliers	7+1ex
mean (n)	51.3705
st.dev. (n)	10.34218
R(calc.)	28.9581
R(ISO17234-1:2015)	29.8368



**APPENDIX 2****Summary of other reported aromatic amines in sample #17520**

lab	Components
2228	2.30 mg/kg 3,3'-Dimethoxybenzidine (CASno. 119-90-4), 2.48 mg/kg 4,4'-Diaminodiphenyl ether (CASno. 101-80-4)
2256	39.9 mg/kg 4-Aminodiphenyl (CASno. 92-67-1)
2592	0.466 mg/kg 4-Aminodiphenyl (CASno. 92-67-1)
2756	27.65 mg/kg 2-Amino-4-nitrotoluene (CASno. 99-55-8), 23.5 mg/kg 2,4-Diaminoanisol (CASno. 615-05-4), 33.85 mg/kg 4,4'-Diaminodiphenyl ether (CASno. 101-80-4); 6.725 mg/kg 2,4-Diaminotoluene (CASno. 95-80-7); 2.65 mg/kg 2,4,5-Trimethylaniline (CASno. 137-17-7)

**Abbreviations of amine names as used in appendix 3:**

4AD = 4-Aminodiphenyl (CASno. 92-67-1)  
 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)  
 oT = o-Toluidine (CASno. 95-53-4)  
 24DAT = 2,4-Diaminotoluene (CASno. 95-80-7)  
 TMA = 2,4,5-Trimethylaniline (CASno. 137-17-7)  
 oA = o-Anisidine (CASno. 90-04-0)  
 4AAT = 4-Amino-azobenzene (CASno. 60-09-3)  
 24X = 2,4-Xylidine (CASno. 95-68-1)  
 26X = 2,6-Xylidine (CASno. 87-62-7)

## APPENDIX 3

## Other reported aromatic amines in sample #17520, see abbreviations in appendix 2

Lab	4AD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB
110	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
213	---	---	---	---	---	---	---	---	---	---	---
230	---	---	---	---	---	---	---	---	---	---	---
348	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
362	---	---	---	---	---	---	---	---	---	---	---
551	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
623	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.
841	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2102	0	0	0	0	0	0	0	0	0	0	0
2115	---	---	---	---	---	---	---	---	---	---	---
2129	---	---	---	---	---	---	---	---	---	---	---
2131	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2135	---	---	---	---	---	---	---	---	---	---	---
2139	---	---	---	---	---	---	---	---	---	---	---
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2166	---	---	---	---	---	---	---	---	---	---	---
2170	---	---	---	---	---	---	---	---	---	---	---
2172	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2184	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2201	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2213	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2228	0	0	0	0	0	0	0	0	0	2.30	0
2230	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2241	<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.
2255	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2256	39.9	---	---	---	---	---	---	---	---	---	---
2271	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
2289	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2290	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2295	---	---	---	---	---	---	---	---	---	---	---
2301	0	0	0	0	0	0	0	0	0	0	0
2310	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.
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	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350	---	---	---	---	---	---	---	---	---	---	---
2352	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2357	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2358	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2369	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2373	---	---	---	---	---	---	---	---	---	---	---
2375	---	---	---	---	---	---	---	---	---	---	---
2378	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2379	---	---	---	---	---	---	---	---	---	---	---
2380	---	---	---	---	---	---	---	---	---	---	---
2381	---	---	---	---	---	---	---	---	---	---	---
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2389	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.
2403	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2410	---	---	---	---	---	---	---	---	---	---	---
2426	---	---	---	---	---	---	---	---	---	---	---
2429	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2432	---	---	---	---	---	---	---	---	---	---	---
2442	---	---	---	---	---	---	---	---	---	---	---
2449	---	---	---	---	---	---	---	---	---	---	---
2455	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
2459	---	---	---	---	---	---	---	---	---	---	---
2467	---	---	---	---	---	---	---	---	---	---	---
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2476	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Lab	4AD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB
2477	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2482	---	---	---	---	---	---	---	---	---	---	---
2488	---	---	---	---	---	---	---	---	---	---	---
2489	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2492	---	---	---	---	---	---	---	---	---	---	---
2493	0	0	0	0	0	0	0	0	0	0	0
2495	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2497	---	---	---	---	---	---	---	---	---	---	---
2499	---	---	---	---	---	---	---	---	---	---	---
2511	---	---	---	---	---	---	---	---	---	---	---
2514	---	---	---	---	---	---	---	---	---	---	---
2516	---	---	---	---	---	---	---	---	---	---	---
2532	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2538	---	---	---	---	---	---	---	---	---	---	---
2540	---	---	---	---	---	---	---	---	---	---	---
2549	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2553	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2560	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2561	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
2563	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2565	---	---	---	---	---	---	---	---	---	---	---
2569	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2572	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2590	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
2592	0.466	---	---	---	---	---	---	---	---	---	---
2597	---	---	---	---	---	---	---	---	---	---	---
2614	---	---	---	---	---	---	---	---	---	---	---
2624	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2629	---	---	---	---	---	---	---	---	---	---	---
2639	---	---	---	---	---	---	---	---	---	---	---
2643	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2654	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2656	---	---	---	---	---	---	---	---	---	---	---
2662	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00
2666	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2695	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2711	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
2713	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2719	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2727	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2730	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2737	---	---	---	---	---	---	---	---	---	---	---
2738	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2741	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2749	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
2756	---	---	---	---	27.65	---	23.5	---	---	---	---
2766	---	---	---	---	---	---	---	---	---	---	---
2770	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3116	---	---	---	---	---	---	---	---	---	---	---
3146	---	---	---	---	---	---	---	---	---	---	---
3150	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3151	---	---	---	---	---	---	---	---	---	---	---
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
3185	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3191	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3192	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
3197	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3200	---	---	---	---	---	---	---	---	---	---	---
3209	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3210	---	---	---	---	---	---	---	---	---	---	---
3214	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3216	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3220	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3225	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3237	---	---	---	---	---	---	---	---	---	---	---
3248	---	---	---	---	---	---	---	---	---	---	---



**Other aromatic amines in sample #17520, continued, see abbreviations in appendix 2**

Lab	DDDM	pC	DDM	DDE	DDS	oT	24DAT	TMA	oA	4AAT	24X	26X
110	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
213	---	---	---	---	---	---	---	---	---	---	---	---
230	---	---	---	---	---	---	---	---	---	---	---	---
348	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
362	---	---	---	---	---	---	---	---	---	---	---	---
551	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
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	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2102	0	0	0	0	0	0	0	0	0	0	0	0
2115	---	---	---	---	---	---	---	---	---	---	---	---
2129	---	---	---	---	---	---	---	---	---	---	---	---
2131	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2135	---	---	---	---	---	---	---	---	---	---	---	---
2139	---	---	---	---	---	---	---	---	---	---	---	---
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2166	---	---	---	---	---	---	---	---	---	---	---	---
2170	---	---	---	---	---	---	---	---	---	---	---	---
2172	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
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	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2213	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2228	0	0	0	2.48	0	0	0	0	0	0	0	0
2230	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
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.
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	---	---	---	---	---	---	---	---	---	---	---	---
2271	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
2289	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2290	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2295	---	---	---	---	---	---	---	---	---	---	---	---
2301	0	0	0	0	0	0	0	0	0	0	0	0
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.
2314	---	---	---	---	---	---	---	---	---	---	---	---
2320	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2330	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350	---	---	---	---	---	---	---	---	---	---	---	---
2352	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2357	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2358	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2364	---	---	---	---	---	---	---	---	---	---	---	---
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2367	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2369	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2373	---	---	---	---	---	---	---	---	---	---	---	---
2375	---	---	---	---	---	---	---	---	---	---	---	---
2378	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2379	---	---	---	---	---	---	---	---	---	---	---	---
2380	---	---	---	---	---	---	---	---	---	---	---	---
2381	---	---	---	---	---	---	---	---	---	---	---	---
2386	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
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.
2403	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2410	---	---	---	---	---	---	---	---	---	---	---	---
2426	---	---	---	---	---	---	---	---	---	---	---	---
2429	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2432	---	---	---	---	---	---	---	---	---	---	---	---
2442	---	---	---	---	---	---	---	---	---	---	---	---
2449	---	---	---	---	---	---	---	---	---	---	---	---
2455	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	---	---	---
2459	---	---	---	---	---	---	---	---	---	---	---	---
2467	---	---	---	---	---	---	---	---	---	---	---	---
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2476	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2477	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2482	---	---	---	---	---	---	---	---	---	---	---	---
2488	---	---	---	---	---	---	---	---	---	---	---	---

Lab	DDDM	pC	DDM	DDE	DDS	oT	24DAT	TMA	oA	4AAT	24X	26X
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	---	---	---	---	---	---	---	---	---	---	---	---
2493	0	0	0	0	0	0	0	0	0	n	0	0
2495	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2497	---	---	---	---	---	---	---	---	---	---	---	---
2499	---	---	---	---	---	---	---	---	---	---	---	---
2511	---	---	---	---	---	---	---	---	---	---	---	---
2514	---	---	---	---	---	---	---	---	---	---	---	---
2516	---	---	---	---	---	---	---	---	---	---	---	---
2532	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2538	---	---	---	---	---	---	---	---	---	---	---	---
2540	---	---	---	---	---	---	---	---	---	---	---	---
2549	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2553	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2560	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2561	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
2563	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2565	---	---	---	---	---	---	---	---	---	---	---	---
2569	<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
2590	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
2592	---	---	---	---	---	---	---	---	---	---	---	---
2597	---	---	---	---	---	---	---	---	---	---	---	---
2614	---	---	---	---	---	---	---	---	---	---	---	---
2624	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2629	---	---	---	---	---	---	---	---	---	---	---	---
2639	---	---	---	---	---	---	---	---	---	---	---	---
2643	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2654	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2656	---	---	---	---	---	---	---	---	---	---	---	---
2662	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00	<3.00
2666	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2695	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2711	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
2713	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2719	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2727	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2730	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2737	---	---	---	---	---	---	---	---	---	---	---	---
2738	n.d.	n.d.	<6	n.d.	n.d.	<6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2741	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2749	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
2756	n.d.	---	---	33.85	---	n.d.	6.725	2.65	---	---	---	---
2766	---	---	---	---	---	---	---	---	---	---	---	---
2770	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3116	---	---	---	---	---	---	---	---	---	---	---	---
3146	---	---	---	---	---	---	---	---	---	---	---	---
3150	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3151	---	---	---	---	---	---	---	---	---	---	---	---
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
3185	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3191	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3192	<30	<30	<30	<30	<30	<30	<30	<30	<30	---	---	---
3197	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3200	---	---	---	---	---	---	---	---	---	---	---	---
3209	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3210	---	---	---	---	---	---	---	---	---	---	---	---
3214	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3216	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	---	n.d.	n.d.
3220	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3225	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3237	---	---	---	---	---	---	---	---	---	---	---	---
3248	---	---	---	---	---	---	---	---	---	---	---	---

## APPENDIX 4

## Analytical details

labnrs	ISO/IEC 17025 accredited for these compounds	Degreased sample with	MTBE/Methanol extraction solution evaporated	Temperature (°C), time (minutes) for the evaporation of MTBE/Methanol	Time of complete analysis (start of reduction till HPLC/GC)	Remarks
110	Yes	n-Hexane	To almost dryness (<0.2 ml)	50°C, 30 minutes	2.5 hours	
213	Yes	n-Hexane	---	---	24 hours	
230	Yes	n-Hexane	No evaporation	---	2 hours	
348	Yes	n-Hexane	---	Not applicable	4 hours	
362	Yes	n-Hexane	To a small residual quantity	---	---	
551	Yes	n-Hexane	To a small residual quantity	---	---	
623	Yes	n-Hexane	To almost dryness	50°C	8 hours	
840	---	---	---	---	---	
841	---	---	---	---	---	
2102	Yes	---	---	---	---	
2115	Yes	Not degreased	To a small residual quantity (2 ml)	60°C	6 hours	
2129	Yes	n-Hexane	To a small residual quantity (2-3 ml)	40°C, 30 minutes	<10 hours	
2131	Yes	n-Hexane	No evaporation	---	3.5 hours	
2132	Yes	n-Hexane	To almost dryness (<0.5 ml)	35°C, 15 minutes	<3 hours	
2135	Yes	n-Hexane	To a small residual quantity	50°C, 8 minutes	5 hours	
2139	Yes	n-Hexane	To a small residual quantity	---	2 hours	
2165	Yes	n-Hexane	To a small residual quantity	<50°C, <10 minutes	<24 hours	
2166	Yes	Not degreased	To complete dryness	50°C, 43 minutes	20.25 hours	simultaneous evaporation with cooling area (1ml), sample stored in vial in refrigerator
2170	Yes	n-Hexane	To a small residual quantity	35°C, <8 minutes	2 hours	
2172	Yes	---	---	---	---	
2184	Yes	n-Hexane	To a small residual quantity	<50°C, <10 minutes	<24 hours	
2201	Yes	n-Hexane	To a small residual quantity (2 ml)	40°C, <6-8 minutes	<4 hours	
2213	Yes	n-Hexane	To a small residual quantity	50°C, 30 minutes	48 hours	
2228	Yes	n-Hexane	To a certain quantity (1 ml)	40°C, <8 minutes	1 hour	
2230	Yes	n-Hexane	To a small residual quantity	40°C, <5-10 minutes	<3 hours	
2241	Yes	n-Hexane	To a small residual quantity (1 ml)	45°C, 10 minutes	<4 hours	
2247	Yes	n-Hexane	To a small residual quantity (2 ml)	40°C, 10-15 minutes	<3.34 hours	
2255	Yes	n-Hexane	To a small residual quantity	40°C, 8-10 minutes	<3 hours	
2256	Yes	n-Hexane	To a small residual quantity	40°C	6 hours	
2271	Yes	n-Hexane	To a small residual quantity	45°C, 5 minutes	4 hours	
2289	Yes	n-Hexane	To a small residual quantity (1 ml)	40°C, 5 minutes	2 hours	
2290	Yes	---	---	---	---	
2295	Yes	Another solvent (Not specified)	To a small residual quantity	45°C, 25 minutes	3 hours	
2301	---	---	---	---	---	
2310	Yes	n-Hexane	To a small residual quantity	50°C, 10-15 minutes	<3 hours	
2311	Yes	n-Hexane	To a small residual quantity	30°C, 5 minutes	2 hours	
2314	Yes	n-Hexane	To a small residual quantity	50°C, 5 minutes	4 hours	
2320	Yes	n-Hexane	liquid-liquid extraction with Ethyl Acetate		1.17 hours	
2330	Yes	n-Hexane	No evaporation	40°C	<24 hours	
2347	Yes	n-Hexane	To a small residual quantity	45°C	72 hours	
2350	Yes	n-Hexane	To a small residual quantity	42°C, 25-30 minutes	<2 hours	
2352	Yes	n-Hexane	To a small residual quantity	45°C, 3-5 minutes	<24 hours	
2357	Yes	n-Hexane	To a small residual quantity	35°C, 12 minutes	1.4 hours	
2358	Yes	n-Hexane	To a small residual quantity	25°C, 15 minutes	2.5 hours	
2364	Yes	n-Hexane	To a small residual quantity	45°C, 3 minutes	1.8 hours	
2365	Yes	n-Hexane	To a small residual quantity (0.2 ml)	40°C, 10 minutes	2 hours	
2366	Yes	n-Hexane	To a small residual quantity	45°C, 8 minutes	0.8 hours	

labnrs	ISO/IEC 17025 accredited for these compounds	Degreased sample with	MTBE/Methanol extraction solution evaporated	Temperature (°C), time (minutes) for the evaporation of MTBE/Methanol	Time of complete analysis (start of reduction till HPLC/GC)	Remarks
2367	Yes	n-Hexane	To a small residual quantity	40°C, 3 minutes	<b>1 min?</b>	
2369	Yes	n-Hexane	To a small residual quantity	40°C, 30 minutes	4 hours	
2370	Yes	n-Hexane	To a small residual quantity (0.1 ml)	40°C, 2 minutes	2 hours	
2373	Yes	n-Hexane	To a small residual quantity	45°C, 3 minutes	1.8 hours	
2375	Yes	n-Hexane	No evaporation	---	---	
2378	Yes	n-Hexane	To a small residual quantity	50°C, 3 minutes	1.6 hours	
2379	Yes	n-Hexane	To complete dryness	40°C, 10 minutes	5 hours	
2380	Yes	n-Hexane	To a small residual quantity	<50°C, 8-12 minutes	4-6 hours	
2381	Yes	n-Hexane	To a small residual quantity	<50°C, 8-10 minutes	4-6 hours	
2386	Yes	n-Hexane	To a small residual quantity	30°C, <15 minutes	<5 hours	
2389	Yes	n-Hexane	No evaporation	---	1 hour	
2390	Yes	n-Hexane	---	70°C, 30 minutes	2 hours	
2403	Yes	n-Hexane	To a small residual quantity	45°C, 5 minutes	1.3 hours	
2410	Yes	n-Hexane	To a small residual quantity (1 ml)	30°C, 5-10 minutes	24 hours	
2426	Yes	n-Hexane	To a small residual quantity	40°C, 15 minutes	3 hours	
2429	Yes	n-Hexane	To a small residual quantity (2 ml)	40°C, 10 minutes	3 hours	
2432	---	---	---	---	---	
2442	Yes	n-Hexane	To a small residual quantity	35°C, <10 minutes	2.5 hours	
2449	Yes	n-Hexane	To a small residual quantity	45°C	5 hours	
2455	Yes	n-Hexane	To a small residual quantity	45°C, <10 minutes	<2 hours	
2459	---	---	---	---	---	
2467	Yes	n-Hexane	To a small residual quantity	50°C, few minutes	0.67 hours	
2472	Yes	n-Hexane	To complete dryness	38°C, 10 minutes	2 hours	
2476	Yes	n-Hexane	To a small residual quantity	40°C, 10 minutes	3 hours	
2477	Yes	n-Hexane	To a small residual quantity	30°C, 30 minutes	48 hours	
2482	Yes	n-Hexane	To a small residual quantity	<50°C, <10 minutes	2 hours	
2488	Yes	n-Hexane	To a small residual quantity	40°C	---	
2489	Yes	n-Hexane	To a small residual quantity (1 ml)	45°C, 15 minutes	4 hours	
2492	Yes	n-Hexane	To a small residual quantity	35°C	2 hours	
2493	Yes	n-Hexane	To a certain quantity	40°C	2 hours	
2495	Yes	n-Hexane	To a certain quantity (5 ml)	40°C, <10 minutes	72 hours	
2497	Yes	Not degreased	To a small residual quantity	40°C, 20 minutes	4 hours	
2499	Yes	n-Hexane	To a small residual quantity (1 ml)	40°C, 90 minutes	2.8 hours	
2511	No	n-Hexane	To a certain quantity (1 ml)	35°C, <5-10 minutes	3-4 hours	
2514	Yes	n-Hexane	To a small residual quantity	48°C, 10 minutes	28 hours	
2516	---	---	---	---	---	
2532	Yes	n-Hexane	To a small residual quantity (1 ml)	40°C, 15 minutes	3 hours	
2538	Yes	n-Hexane	No evaporation	---	72 hours	
2540	Yes	n-Hexane	To a small residual quantity	40°C, 5 minutes	5 hours	
2549	Yes	n-Hexane	To a small residual quantity	40°C, 10 minutes	>1.5 hours	Q3. MTBE-90ml and MeOH-2ml
2553	Yes	n-Hexane	To complete dryness	40°C, <3 minutes	1.2 hours	
2560	Yes	n-Hexane	To a small residual quantity	40°C, 4-5 minutes	3.5 hours	
2561	Yes	n-Hexane	To a small residual quantity	50°C, <18 minutes	8 hours	
2563	Yes	n-Hexane	---	---	18 hours	
2565	Yes	n-Hexane	To a small residual quantity	35°C, <30 minutes	<4 hours	
2569	Yes	n-Hexane	No evaporation	50°C	6 hours	
2572	Yes	---	---	---	---	
2590	Yes	n-Hexane	To a small residual quantity (1 ml)	40°C, 10 minutes	2.4 hours	
2592	Yes	n-Hexane	To a small residual quantity	40°C	48 hours	
2597	---	---	---	---	---	
2614	Yes	n-Hexane	To a certain quantity	40°C, 2-3 minutes	4 hours	
2624	No	n-Hexane	To a small residual quantity (2 ml)	---	---	
2629	Yes	n-Hexane	To a small residual quantity	40°C, <10 minutes	2 hours	
2639	Yes	n-Hexane	To a small residual quantity (1 ml)	45°C, 15 minutes	3 hours	
2643	---	n-Hexane	No evaporation	Not applicable	5 hours	
2654	Yes	n-Hexane	To a small residual quantity	48°C, 10 minutes	4 hours	

labnrs	ISO/IEC 17025 accredited for these compounds	Degreased sample with	MTBE/Methanol extraction solution evaporated	Temperature (°C), time (minutes) for the evaporation of MTBE/Methanol	Time of complete analysis (start of reduction till HPLC/GC)	Remarks
2656	---	---	---	---	---	
2662	Yes	n-Hexane	To a small residual quantity	35°C, 5 minutes	<2 hours	
2666	Yes	Acetone	To a small residual quantity (5 ml)	45°C, 10 minutes	24 hours	evaporated to 1 ml with Nitrogen
2674	Yes	n-Hexane	To a small residual quantity	35°C, 10 minutes	<24 hours	
2695	Yes	n-Hexane	To a small residual quantity	40°C, 10 minutes	2 hours	
2711	No	n-Hexane	To a small residual quantity	50°C, 4 minutes	6 hours	
2713	Yes	n-Hexane	To a small residual quantity (0.2 ml)	40°C, 5 minutes	26 hours	
2719	Yes	n-Hexane	To a small residual quantity	45°C, 5 minutes	2 hours	
2727	Yes	n-Hexane	To complete dryness	50°C, 1 minute	3 hours	
2730	No	n-Hexane	To a small residual quantity (1 ml)	45°C, 15 minutes	7 hours	under vacuum
2737	Yes	n-Hexane	To a small residual quantity	50°C, 18 minutes	3 hours	
2738	Yes	n-Hexane	To complete dryness	45°C, 10 minutes	4 hours	with nitrogen
2741	Yes	n-Hexane	To a small residual quantity	45°C, 45 minutes	<3 hours	
2749	No	n-Hexane	To a small residual quantity	room T, 7 minutes	10 hours	
2756	No	n-Hexane	To a certain quantity	45°C, 60 minutes	72 hours	
2766	Yes	n-Hexane	To complete dryness	25°C, 15 minutes	3 hours	
2770	Yes	n-Hexane	To a small residual quantity	45°C, 2-3 minutes	6 hours	
3116	Yes	n-Hexane	To a small residual quantity	40°C, 15 minutes	1.2 hours	
3146	Yes	n-Hexane	---	---	---	
3150	Yes	n-Hexane	To a certain quantity	45°C, <8 minutes	3 hours	
3151	Yes	n-Hexane	To a certain quantity	40°C, 30 minutes	2.5 hours	
3153	Yes	n-Hexane	To a certain quantity	35°C, <8 minutes	12 hours	
3154	Yes	Not degreased	---	---	---	
3160	No	n-Hexane	To a small residual quantity	50°C, 10 minutes	3 hours	
3172	Yes	n-Hexane	To a small residual quantity	35°C, 10 minutes	2 hours	
3185	Yes	n-Hexane	To a small residual quantity	35°C, 5-10 minutes	<1.7 hours	
3191	Yes	n-Hexane	To a small residual quantity	45°C, 5 minutes	24 hours	
3192	Yes	n-Hexane	To a small residual quantity (1 ml)	50°C, <5 minutes	24 hours	dryness by blowing with nitrogen; at 540-550 mbar
3197	Yes	n-Hexane	To a small residual quantity	40°C, 10 minutes	<2 hours	
3200	Yes	n-Hexane	To a certain quantity	50°C, 5 minutes	2 hours	
3209	Yes	n-Hexane	To a small residual quantity	40°C, 4 minutes	1.5 hours	
3210	Yes	n-Hexane	To a small residual quantity (0.5 ml)	40°C, 15 minutes	2 hours	
3214	Yes	n-Hexane	To a small residual quantity	<50°C, 10 minutes	6 hours	
3216	No	n-Hexane	To a small residual quantity	40°C, 5-10 minutes	6 hours	
3220	Yes	n-Hexane	To complete dryness	35°C, 3 minutes	4 hours	
3225	Yes	n-Hexane	To complete dryness	40°C, 10 minutes	7 hours	
3228	Yes	n-Hexane	To a small residual quantity (0.5 ml)	<50°C, <15 minutes	<12 hours	
3237	Yes	n-Hexane	No evaporation	---	14 hours	
3248	Yes	n-Hexane	To a small residual quantity	---	---	

## APPENDIX 5

### Number of participants per country

6 labs in BANGLADESH  
1 lab in BRAZIL  
1 lab in BULGARIA  
1 lab in CAMBODIA, Kingdom of  
1 lab in EGYPT  
1 lab in ETHIOPIA  
3 labs in FRANCE  
12 labs in GERMANY  
8 labs in HONG KONG  
1 lab in HUNGARY  
12 labs in INDIA  
2 labs in INDONESIA  
11 labs in ITALY  
2 labs in JAPAN  
5 labs in KOREA  
1 lab in MAURITIUS  
2 labs in MEXICO  
2 labs in MOROCCO  
32 labs in P.R. of CHINA  
5 labs in PAKISTAN  
1 lab in PORTUGAL  
1 lab in ROMANIA  
1 lab in SINGAPORE  
3 labs in SPAIN  
2 labs in SRI LANKA  
3 labs in SWITZERLAND  
3 labs in TAIWAN R.O.C.  
1 lab in THAILAND  
1 lab in THE NETHERLANDS  
1 lab in TUNISIA  
6 labs in TURKEY  
2 labs in U.S.A.  
1 lab in UNITED KINGDOM  
7 labs in VIETNAM

## APPENDIX 6

### Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected

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