

**Results of Proficiency Test
AZO dyes in leather
March 2018**

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

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1 INTRODUCTION

Since 1997, the Institute for Interlaboratory Studies (iis) organizes a proficiency test for banned AZO dyes in leather. During the annual proficiency testing program 2017/2018, it was decided to continue the proficiency test for the analysis of banned AZO dyes in leather. In this interlaboratory study, 120 laboratories in 31 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2018 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 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 #18525.

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, 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 dark brown colored leather sample was made positive on banned AZO-dyes by a third party laboratory. The leather material was cut into small pieces and mixed thoroughly. In total 168 bags with 3.5 gram material were prepared and labelled #18525. 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 #18525-1	72.5
sample #18525-2	71.3
sample #18525-3	79.0
sample #18525-4	74.5
sample #18525-5	79.9
sample #18525-6	70.6
sample #18525-7	72.0
sample #18525-8	76.1

Table 1: homogeneity test results of subsamples #18525

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

	<i>Benzidine in mg/kg</i>
r (observed)	9.9
reference test method	ISO17234-1:2015
0.3 * R (ref. test method)	13.0

Table 2: evaluation of the repeatability of the aromatic amine in subsamples #18525

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

To each of the participating laboratories one sample containing approximately 3.5 grams testing material (labelled #18525) was sent on March 7, 2018.

2.5 ANALYSES

The participants were asked to determine the concentrations of 23 forbidden aromatic amines and 2,5-Xylidene and Total Xylidines, applying the analysis procedure that is routinely used in the laboratory. It was also requested to report if the laboratory was accredited for the determined components.

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 evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reported 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.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/. 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 visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. 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. Five participants reported test results after the deadline and three other participants were not able to report in time. The 117 participants did report 116 numerical test results. (One lab did not report the positive component, but only "<30" for a number of other banned AZO dyes). Observed were 4 outlying test results, which is 3.4% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

The test data set for Benzidine appeared not to be normally distributed.

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 78 mg/kg was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the reproducibility requirement estimated from the reference test method ISO 17234-1:2015.

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	112	78	45	45

Table 3: reproducibility of the aromatic amine in leather sample #18525

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 OF THE PROFICIENCY TEST OF MARCH 2018 WITH PREVIOUS PTS

The observed variation in the test results for the aromatic amine in the 2018 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>March 2018</i>	<i>Feb 2017</i>	<i>Feb 2016</i>	<i>March 2015</i>	<i>March 2014</i>	<i>March 2012-2005</i>	<i>Target *)</i>
4-Aminodiphenyl	n.e.	n.e.	n.e.	n.e.	n.e.	25 - 45%	15 - 33%
Benzidine	20%	20%	34%	n.e.	20%	20 - 66%	15 - 25%
3,3'-Dimethylbenzidine	n.e.	n.e.	39%	24%	n.e.	45 - 55%	17 - 24%
<i>o</i> -Toluidine	n.e.	n.e.	37%	n.e.	n.e.	50 - 63%	30 - 37%
2,4-Xylidine	n.e.	n.e.	n.e.	n.e.	n.e.	16 - 36%	15 - 33%

Table 4: development of the uncertainties over the years

*) the target reproducibility is concentration related

4.4 EVALUATION ANALYTICAL DETAILS

For this PT, only one question was requested: Is your laboratory accredited in accordance with ISO/IEC17025? Eighty-eight of the registered participants mentioned that they are accredited for determination of banned AZO-dyes in leather. Nineteen participants mentioned that they are not accredited. No effect was observed on the average Benzidine or variation between reported test results.

5 DISCUSSION

In this PT, no analytical details were asked like in previous proficiency tests. In the 2017 proficiency test it became clear that the group of participants was able to meet the precision criteria of the test method. Therefore, it was decided not to request for analytical details in the 2018 PT. The group of participants in the 2018 PT proved that it was justified as the reproducibility of the group meet again the precision data of the test method.

6 CONCLUSION

Although, it can be concluded that the majority of the participants has no problem with the determination of Benzidine in the sample of this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

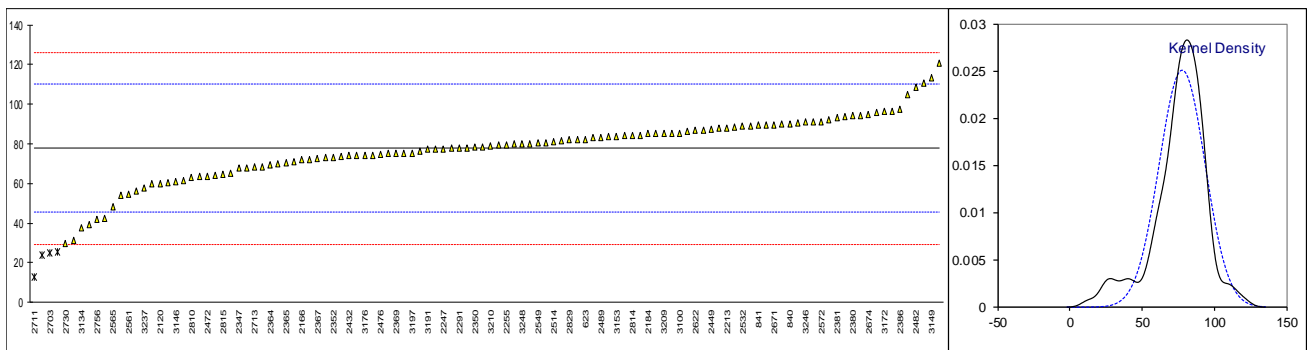
Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1**Determination of Benzidine (CASno.92-87-5) in sample #18525; test results in mg/kg**

lab	method	value	mark	z(targ)	Remarks
213	ISO17234-1	59.73		-1.12	
230	EN14362-1	39.39		-2.38	
348	In house	77.69		0.00	
362		-----		-----	
551	ISO17234-1	31.52	C	-2.87	First reported 13.586
623	ISO17234-1	82.195		0.28	
840	ISO17234-1	90.1		0.77	
841	ISO17234-1	89.27		0.71	
2102	ISO17234-1	56.42		-1.32	
2108	ISO17234-1	91.12		0.83	
2115	ISO17234-1	93.64		0.98	
2120	ISO17234-1	60.0	C	-1.10	First reported 29.0
2129	EN14362-1	64.2		-0.84	
2132	ISO17234-1	83.7271		0.37	
2165	ISO17234-1	83		0.33	
2166	EN14362-1	71.89		-0.36	
2170	EN14362-1	71.00		-0.42	
2184	ISO17234-1	85		0.45	
2201	ISO17234-1	95.62		1.11	
2213	ISO17234-1	88		0.64	
2215	ISO17234-1	78.47		0.04	
2247	ISO17234-1	77.56		-0.01	
2250	ISO17234-1	68		-0.60	
2255		79.7		0.12	
2256	ISO17234-1	80.5		0.17	
2266	ISO17234-1	25.40	R(0.05)	-3.25	
2290	ISO17234-1	89.3		0.72	
2291	ISO17234-1	78		0.02	
2301	ISO17234-1	94.04		1.01	
2310	ISO17234-1	87.09		0.58	
2311	ISO17234-1	89.02		0.70	
2330	ISO17234-1	90.68		0.80	
2347	ISO17234-1	68		-0.60	
2350	ISO17234-1	78.28		0.03	
2352	GB/T19942	73.1		-0.29	
2357	ISO17234-1	73.5		-0.26	
2358	ISO17234-1	92.26522		0.90	
2364	ISO17234-1	69.34		-0.52	
2365	ISO17234-1	70.69		-0.44	
2366	ISO17234-1	75.0		-0.17	
2367	ISO17234-1	72.74		-0.31	
2369	ISO17234-1	75		-0.17	
2370	ISO17234-1	85.47		0.48	
2374	ISO17234-1	69.8		-0.49	
2375	ISO17234-1	73.98		-0.23	
2378	ISO17234-1	72.22		-0.34	
2379	ISO17234-1	120.54		2.65	
2380	ISO17234-1	94.0		1.01	
2381	ISO17234-1	93.07		0.95	
2382	ISO17234-1	73.0		-0.29	
2386	ISO17234-1	97.5		1.22	
2389	ISO17234-1	81.66		0.24	
2390	ISO17234-1	96.557		1.17	
2410	ISO17234-1	84.34		0.41	
2425	ISO17234-1	78		0.02	
2426	ISO17234-1	80.10		0.15	
2432	ISO17234-1	73.98		-0.23	
2442	In house	61.72		-0.99	
2449	ISO17234-1	87.11		0.58	
2453	ISO17234-1	54.11		-1.47	
2455	ISO17234-1	23.78	C,R(0.05)	-3.35	First reported 17.309
2459	ISO17234-1	83.945		0.38	
2472	ISO17234-1	63.7		-0.87	
2476	ISO17234-1	74.71		-0.19	
2482	ISO17234-1	108.6		1.91	
2489	ISO17234-1	83.0		0.33	
2492	ISO17234-1	88.5		0.67	
2495	ISO17234-1	65.42		-0.76	
2497	ISO17234-1	110.63		2.04	
2511	ISO17234-1	75.162		-0.16	
2514	ISO17234-1	81.0		0.20	
2532	ISO17234-1	89		0.70	
2549	ISO17234-1	80.36		0.16	

lab	method	value	mark	z(targ)	Remarks
2561	ISO17234-1	54.46		-1.44	
2565	ISO17234-1	48.2		-1.83	
2566	ISO17234-1	89.83		0.75	
2572	ISO17234-1	91.2		0.83	
2590	ISO17234-1	82.182		0.27	
2592	ISO17234-1	42.5		-2.19	
2602	ISO17234-1	77.32		-0.03	
2622	ISO17234-1	86.6		0.55	
2643	ISO17234-1	68.55		-0.57	
2668	ISO17234-1	79.48		0.11	
2671	ISO17234-1	89.72		0.74	
2674	ISO17234-1	95		1.07	
2695	ISO17234-1	63.59		-0.88	
2703	ISO17234-1	25.16	C,R(0.05)	-3.26	First reported 12.6
2711	ISO17234-1	12.805	R(0.05)	-4.03	
2713	ISO17234-1	68.19		-0.59	
2727	ISO17234-1	86.25		0.53	
2730		29.957		-2.96	
2756	ISO17234-1	41.846	C	-2.23	First reported 17.15
2804	ISO17234-1	85.01		0.45	
2810	ISO17234-1	63.21		-0.90	
2812	ISO17234-1	76.44		-0.08	
2814	EN14362-1	83.947		0.38	
2815	ISO17234-1	64.43		-0.83	
2823		-----		-----	
2829	ISO17234-1	81.90		0.26	
3100	ISO17234-1	85.52		0.48	
3116	ISO17234-1	87.8		0.62	
3118	ISO17234-1	74.13		-0.22	
3134	ISO17234-1	37.50		-2.50	
3146	ISO17234-1	61.0		-1.04	
3149	ISO17234-1	113.5		2.22	
3150	ISO17234-1	60.5		-1.07	
3153	ISO17234-1	83.8		0.37	
3154	ISO17234-1	104.871		1.68	
3163		-----		-----	
3172	ISO17234-1	96.20		1.14	
3176	ISO17234-1	74.08		-0.23	
3191	ISO17234-1	77.23		-0.03	
3192		-----		-----	
3197	ISO17234-1	75.2		-0.16	
3209	ISO17234-1	85.01		0.45	
3210	In house	78.80		0.06	
3228	ISO17234-1	80		0.14	
3237	EN14362-1	57.7		-1.24	
3246	ISO17234-1	91.00		0.82	
3248	ISO17234-1	80		0.14	

normality suspect
n 112
outliers 4
mean (n) 77.7556
st.dev. (n) 15.90496
R(calc.) 44.5339
st.dev.(ISO17234-1:2015) 16.12917
R(ISO17234-1:2015) 45.1617



APPENDIX 2

Other reported aromatic amines in sample #18525

Abbreviations of amine names as used in appendix 2:

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)
24X = 2,4-Xylidine (CASno. 95-68-1)
25X = 2,5-Xylidine (CASno. 95-78-3)
26X = 2,6-Xylidine (CASno. 87-62-7)
TX = Total of Xylidines

Other reported aromatic amines in sample #18525, see abbreviations on page 12

Lab	4AD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM
213	----	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----	----
348	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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	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
2108	----	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----	----
2120	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2129	----	----	----	----	----	----	----	----	----	----	----	----
2132	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2165	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2166	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2170	----	----	----	----	----	----	----	----	----	----	----	----
2184	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
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
2215	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2247	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2250	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2255	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2256	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2266	0	0	0	0	0	0	0	0	0	0	0	0
2290	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2291	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2301	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
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.
2330	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2352	----	----	----	----	----	----	----	----	----	----	----	----
2357	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2364	----	----	----	----	----	----	----	----	----	----	----	----
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2367	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
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.
2374	----	----	----	----	----	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----	----	----	----	----	----
2378	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2379	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2380	----	----	----	----	----	----	----	----	----	----	----	----
2381	----	----	----	----	----	----	----	----	----	----	----	----
2382	----	----	----	----	----	----	----	----	----	----	----	----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2389	----	----	----	----	----	----	----	----	----	----	----	----
2390	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2410	----	----	----	----	----	----	----	----	----	----	----	----
2425	----	----	----	----	----	----	----	----	----	----	----	----
2426	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2432	----	----	----	----	----	----	----	----	----	----	----	----
2442	----	----	----	----	----	----	----	----	----	----	----	----
2449	----	----	----	----	----	----	----	----	----	----	----	----
2453	----	----	----	----	----	----	----	----	----	----	----	----
2455	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2459	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2476	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2482	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2489	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2492	----	----	----	----	----	----	----	----	----	----	----	----
2495	<5	<5	<5	----	----	<5	<5	<5	<5	<5	<5	<5
2497	----	----	----	----	----	----	----	----	----	----	----	----
2511	----	----	----	----	----	----	----	----	----	----	----	----
2514	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
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.
2549	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2561	----	----	----	----	----	----	----	----	----	----	----	----

Lab	4AD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM
2565	----	----	----	----	----	----	----	----	----	----	----	----
2566	<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	----	----	----	----	----	----	----	----	----	----	----	----
2602	----	----	----	----	----	----	----	----	----	----	----	----
2622	1.08	----	----	----	----	----	----	----	----	----	----	----
2643	----	----	----	----	----	----	----	----	----	----	----	----
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
2671	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
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	----	----	----	----	----	----	----	----	----	----	----	----
2703	----	----	----	----	----	----	----	----	----	----	----	----
2711	0	0	0	0	0	0	0	0	0	0	0	0
2713	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2727	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
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.
2756	----	----	----	----	----	----	----	----	----	----	----	----
2804	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2810	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
2812	----	----	----	----	----	----	----	----	----	----	----	----
2814	----	----	----	----	----	----	----	----	----	----	----	----
2815	0.19	0.01	0.06	----	----	0.08	4.58	0.26	0.08	0.02	0.06	0.04
2823	----	----	----	----	----	----	----	----	----	----	----	----
2829	----	----	----	----	----	----	----	----	----	----	----	----
3100	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3116	----	----	----	----	----	----	----	----	----	----	----	----
3118	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3134	6.30	----	----	----	----	----	----	----	----	----	----	----
3146	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3149	----	----	----	----	----	----	----	----	----	----	----	----
3150	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	----	----	----	----	----	----	----	----	----	----	----	----
3163	----	----	----	----	----	----	----	----	----	----	----	----
3172	----	----	----	----	----	----	----	----	----	----	----	----
3176	----	----	----	----	----	----	----	----	----	----	----	----
3191	----	----	----	----	----	----	----	----	----	----	----	----
3192	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
3197	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3209	----	----	----	----	----	----	----	----	----	----	----	----
3210	----	----	----	----	----	----	----	----	----	----	----	----
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	----	----	----	----	----	----	----	----	----	----	----	----
3246	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3248	----	----	----	----	----	----	----	----	----	----	----	----

Other aromatic amines in sample #18525, continued, see abbreviations

Lab	pC	DDM	DDE	DDS	oT	24DAT	TMA	oA	24X	25X	26X	TX
213	----	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----	----
348	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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	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
2108	----	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----	----
2120	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2129	----	----	----	----	----	----	----	----	----	----	----	----
2132	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2165	ND	ND	ND	ND	ND	ND	ND	ND	ND	----	ND	----
2166	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2170	----	----	----	----	----	----	----	----	----	----	----	----
2184	ND	ND	ND	ND	ND	ND	ND	ND	ND	----	ND	----
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
2215	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2247	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2250	<5	<5	----	----	<5	<5	<5	<5	<5	----	<5	----
2255	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2256	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2266	0	0	0	0	0	0	0	0	0	0	0	0
2290	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2291	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2301	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
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.
2330	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2350	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2352	----	----	----	----	----	----	----	----	----	----	----	----
2357	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	N/A	n.d.	N/A
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	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
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.
2374	----	----	----	----	----	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----	----	----	----	----	----
2378	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2379	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2380	----	----	----	----	----	----	----	----	----	----	----	----
2381	----	----	----	----	----	----	----	----	----	----	----	----
2382	----	----	----	----	----	----	----	----	----	----	----	----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2389	----	----	----	----	----	----	----	----	----	----	----	----
2390	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2410	----	----	----	----	----	----	----	----	----	----	----	----
2425	----	----	----	----	----	----	----	----	----	----	----	----
2426	ND	ND	ND	ND	ND	ND	ND	ND	ND	----	ND	----
2432	----	----	----	----	----	----	----	----	----	----	----	----
2442	----	----	----	----	----	----	----	----	----	----	----	----
2449	----	----	----	----	----	----	----	----	----	----	----	----
2453	----	----	----	----	----	----	----	----	----	----	----	----
2455	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	----
2459	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2476	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2482	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	----	< 5	< 5
2489	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2492	----	----	----	----	----	----	----	----	----	----	----	----
2495	<5	<5	<5	<5	<5	<5	<5	<5	<5	----	<5	----
2497	----	----	----	----	----	----	----	----	----	----	----	----
2511	----	----	----	----	----	----	----	----	----	----	----	----
2514	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
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.
2549	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2561	----	----	----	----	----	----	----	----	----	----	----	----

Lab	pC	DDM	DDE	DDS	oT	24DAT	TMA	oA	24X	25X	26X	TX
2565	----	----	----	----	----	----	----	----	----	----	----	----
2566	<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	----
2592	----	----	----	----	----	----	----	----	----	----	----	----
2602	----	----	----	----	----	----	----	----	----	----	----	----
2622	----	----	----	----	----	----	----	----	----	----	----	----
2643	----	----	----	----	----	----	----	----	----	----	----	----
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
2671	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
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	----	----	----	----	----	----	----	----	----	----	----	----
2703	----	----	----	----	----	----	----	----	----	----	----	----
2711	0	0	0	0	0	0	0	0	0	0	0	0
2713	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2727	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
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.
2756	----	----	----	----	----	----	----	----	----	----	----	----
2804	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2810	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
2812	----	----	----	----	----	----	----	----	----	----	----	----
2814	----	----	----	----	----	----	----	----	----	----	----	----
2815	0.03	0.26	0.01	0.01	0.02	1.67	0.02	0.01	0.02	----	0.01	----
2823	----	----	----	----	----	----	----	----	----	----	----	----
2829	----	----	----	----	----	----	----	----	----	----	----	----
3100	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3116	----	----	----	----	----	----	----	----	----	----	----	----
3118	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3134	----	----	----	----	----	----	----	----	----	----	----	----
3146	<10	<10	<10	<10	<10	<10	<10	<10	<10	----	<10	<10
3149	----	----	----	----	----	----	----	----	----	----	----	----
3150	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	----	----	----	----	----	----	----	----	----	----	----	----
3163	----	----	----	----	----	----	----	----	----	----	----	----
3172	----	----	----	----	----	----	----	----	----	----	----	----
3176	----	----	----	----	----	----	----	----	----	----	----	----
3191	----	----	----	----	----	----	----	----	----	----	----	77.23
3192	<30	<30	<30	<30	<30	<30	<30	<30	<30	----	<30	----
3197	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3209	----	----	----	----	----	----	----	----	----	----	----	----
3210	----	----	----	----	----	----	----	----	----	----	----	----
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----
3237	----	----	----	----	----	----	----	----	----	----	----	----
3246	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3248	----	----	----	----	----	----	----	----	----	----	----	----

APPENDIX 3

Accreditation / Remarks

labnrs	ISO/IEC 17025 accredited for these compounds	Remarks
213	Yes	
230	Yes	
348	Yes	
362	---	
551	Yes	
623	Yes	
840	Yes	
841	---	
2102	Yes	
2108	Yes	
2115	Yes	
2120	No	
2129	Yes	
2132	Yes	
2165	Yes	
2166	Yes	
2170	Yes	
2184	Yes	
2201	Yes	
2213	Yes	
2215	No	
2247	Yes	
2250	Yes	
2255	Yes	
2256	Yes	
2266	Yes	
2290	---	
2291	Yes	
2301	Yes	
2310	Yes	
2311	Yes	
2330	Yes	
2347	Yes	
2350	No	
2352	Yes	
2357	Yes	
2358	Yes	
2364	Yes	
2365	Yes	
2366	Yes	
2367	Yes	
2369	---	
2370	Yes	
2374	Yes	
2375	Yes	Aniline: 4,09 mg/kg & p-phenylendiamne 45,32 mg/kg, result for p-AAB is ND
2378	Yes	
2379	No	
2380	Yes	
2381	Yes	
2382	---	
2386	---	
2389	---	
2390	Yes	
2410	Yes	
2425	Yes	
2426	Yes	
2432	No	
2442	Yes	
2449	Yes	
2453	No	
2455	Yes	Used 100 ppm standard for analysis instead of the stated 30 ppm standard stated in the method. Sample heated in a ventilated oven for cleavage
2459	Yes	
2472	Yes	
2476	---	
2482	Yes	
2489	Yes	
2492	Yes	
2495	Yes	

labnrs	ISO/IEC 17025 accredited for these compounds	Remarks
2497	Yes	
2511	No	
2514	Yes	
2532	Yes	
2549	Yes	
2561	Yes	
2565	No	
2566	Yes	
2572	---	
2590	Yes	
2592	Yes	
2602	Yes	Phenylendiamin: 34,6 mg/kg, Anilin 3,0 mg/kg, recovery for Benzidine: 67%
2622	No	
2643	---	
2668	Yes	
2671	Yes	
2674	Yes	
2695	Yes	
2703	Yes	
2711	No	
2713	Yes	
2727	Yes	
2730	No	
2756	No	
2804	No	
2810	No	
2812	No	
2814	Yes	
2815	No	
2823	---	
2829	No	
3100	Yes	
3116	Yes	
3118	Yes	
3134	No	
3146	Yes	
3149	Yes	
3150	Yes	
3153	Yes	
3154	---	
3163	No	
3172	Yes	
3176	Yes	
3191	Yes	
3192	Yes	
3197	Yes	
3209	---	
3210	Yes	
3228	Yes	
3237	Yes	
3246	Yes	
3248	Yes	

APPENDIX 4

Number of participants per country

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

APPENDIX 5

Abbreviations:

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

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