Institute for
Interlaboratory Studies

Results of Proficiency Test
AZO Dyes in Leather/Footwear
February 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis22A02

April 2022

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1 INTRODUCTION

Since 1997 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of banned aromatic amines derived from AZO dyes in Leather/Footwear every year. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the analysis of banned aromatic amines derived from AZO dyes in Leather/Footwear.

In this interlaboratory study 98 laboratories in 29 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the AZO dyes in Leather/Footwear proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different leather samples of 3 grams each, one sample labelled #22510 and the other sample #22511.
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

The first sample is a batch of brown leather, positive on AZO dyes, bought from the local market. This batch was grinded. After homogenization the batch was divided over 160 small bags of approximately 3 grams each and labelled #22510.
The homogeneity of the subsamples was checked by the determination of Benzidine and 2,4-Xylidine using test method ISO17234 on ten stratified randomly selected subsamples.

	Benzidine in mg/kg	2,4-Xylidine in mg/kg
sample #22510-1	106.0	7.3
sample #22510-2	117.1	8.6
sample #22510-3	111.3	7.4
sample #22510-4	136.6	9.5
sample #22510-5	118.0	8.2
sample #22510-6	114.7	8.3
sample #22510-7	122.5	8.9
sample #22510-8	110.7	8.1
sample #22510-9	99.0	8.0
sample #22510-10	118.0	8.9

Table 1: homogeneity test results of subsamples #22510

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

	Benzidine in mg/kg	2,4-Xylidine in mg/kg
r (observed)	28.1	1.9
reference method	iis memo 2202	iis memo 2202
0.3 x R (reference method)	26.2	1.9

Table 2: evaluation of the repeatabilities of subsamples #22510

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

For the preparation of the second sample a batch of red/black leather blocks, made positive on AZO Dyes, was obtained from a third party. After homogenization the batch was divided over 161 small bags of approximately 3 grams each and labelled #22511.
The homogeneity of the subsamples was checked by determination of 3,3'-Dimethylbenzidine using test method ISO17234 on eight stratified randomly selected subsamples.

	3,3'-Dimethylbenzidine in mg/kg
sample #22511-1	105.4
sample #22511-2	100.5
sample #22511-3	99.0
sample #22511-4	101.3
sample #22511-5	104.7
sample #22511-6	106.4
sample #22511-7	96.5
sample #22511-8	104.4

Table 3: homogeneity test results of subsamples #22511

From the above test results the repeatability was 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.

	3,3'-Dimethylbenzidine in mg/kg
r (observed)	9.8
reference method	iis memo 2202
0.3 x R (reference method)	23.2

Table 4: evaluation of the repeatability of subsamples #22511

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

To each of the participating laboratories one leather sample labelled #22510 and one leather sample labelled #22511 was sent on February 2, 2022.

2.5 ANALYZES

The participants were asked to determine the following aromatic amines on both samples #22510 and #22511:
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 also requested to report if the laboratory was accredited to determine the reported components and to report some analytical details. To ensure homogeneity it was requested not to use less than 0.5 grams per determination and not to age and/or dry the samples, nor to determine volatile matter.

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 appendix 1 and 2 of this report. The laboratories are presented by their code numbers.

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

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by 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 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 shown as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

$z_{(target)}$ = (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

In this proficiency test no problems were encountered with the dispatch of the samples. Fourteen participants reported test results after the final reporting date and two other participants did not report any test results at all. Not all participants were able to report all components requested.

In total 96 participants reported 258 numerical test results. Observed were 3 outlying test results, which is 1.2%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal. All data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These 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 in Leather/Footwear test method ISO17234 is considered to be the official test method. Unfortunately, only for a few aromatic amines precision data are mentioned in this test method and if mentioned the precision data is often not for a large concentration range and sometimes not conclusive. As alternative for the aromatic amines not mentioned in the test method iis had used an estimated target reproducibility calculated with the Horwitz equation. Unfortunately, this could give a 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 Leather/Footwear is 27%. This investigation is summarized in iis memo 2202.

sample #22510

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

2,4-Xylidine (CAS No. 95-68-1): This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the target reproducibility as derived from iis memo 2202.

sample #22511

3,3'-Dimethylbenzidine (CAS No. 119-93-7): This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the target reproducibility as derived from iis memo 2202.

The majority of the participants agreed on a concentration near or below the limit of detection for all other aromatic amines mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these aromatic amines. The reported test results of these components are given in appendix 2.

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 previous iis PTs are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
Benzidine	mg/kg	94	140.9	112.6	106.5
2,4-Xylidine	mg/kg	68	9.09	5.29	6.87

Table 5: reproducibilities on sample #22510

Component	unit	n	average	2.8 * sd	R(target)
3,3'-Dimethylbenzidine	mg/kg	93	104.1	47.4	78.7

Table 6: reproducibility on sample #22511

Without further statistical calculations, it can be concluded that the group of participants has no problems with the analyzes of the observed aromatic amines in leather in this PT.

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2022 WITH PREVIOUS PTS

	February 2022	March 2021	March 2020	March 2019	March 2018
Number of reporting laboratories	96	108	90	117	117
Number of test results	258	212	166	117	116
Number of statistical outliers	3	6	1	3	4
Percentage of statistical outliers	1.2%	2.8%	0.6%	2.6%	3.4%

Table 7: 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 tests was compared, expressed as relative standard deviation (RSD) of the PTs in the next table.

Component	February 2022	March 2021	March 2020	March 2019	March 2018	Target
4-Aminodiphenyl	n.e.	n.e.	n.e.	n.e.	n.e.	27%
Benzidine	29%	21%	29%	n.e.	20%	27%
3,3'-Dimethoxybenzidine	n.e.	n.e.	n.e.	23%	n.e.	27%
3,3'-Dimethylbenzidine	16%	n.e.	n.e.	n.e.	n.e.	27%
o-Toluidine	n.e.	n.e.	n.e.	n.e.	n.e.	27%
o-Ansidine	n.e.	n.e.	61%	n.e.	n.e.	27%
2,4-Xylidine	21%	n.e.	n.e.	n.e.	n.e.	27%
4-Aminoazobenzene	n.a.	19%	n.e.	n.e.	n.e.	27%

Table 8: development of uncertainties over the years

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

4.4 EVALUATION ANALYTICAL DETAILS

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

- 89% of the participants mentioned that they are accredited for the determination of aromatic amine components.
- 57% of the participants used the samples as received, 42% further cut/grinded the samples prior to analysis.
- 55% of the participants used around 1 grams of sample intake and 40% used around 0.5 grams.

5 DISCUSSION

All reporting participants were able to detect Benzidine in sample #22510 and 3,3'-Dimethylbenzidine in sample #22511. Also a low level of 2,4-Xylidine in sample #22510 was reported by most participants. The other aromatic amines that were requested in this PT were not detected.

When the results of this interlaboratory study were compared to the LEATHER STANDARD by OEKO-TEX and with the similar BlueSign® system substances list or BSSL (see Table 9), it was noticed that all participants would make an identical decision about the acceptability of the samples for the determined components.
All reporting laboratories would reject sample #22510 and sample #22511 for all categories.

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

Table 9: BlueSign® BSSL and LEATHER STANDARD by OEKO-TEX

6 CONCLUSION

Although it can be concluded that the majority participants have no problem with the determination of Benzidine, 2,4-Xylidine 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.

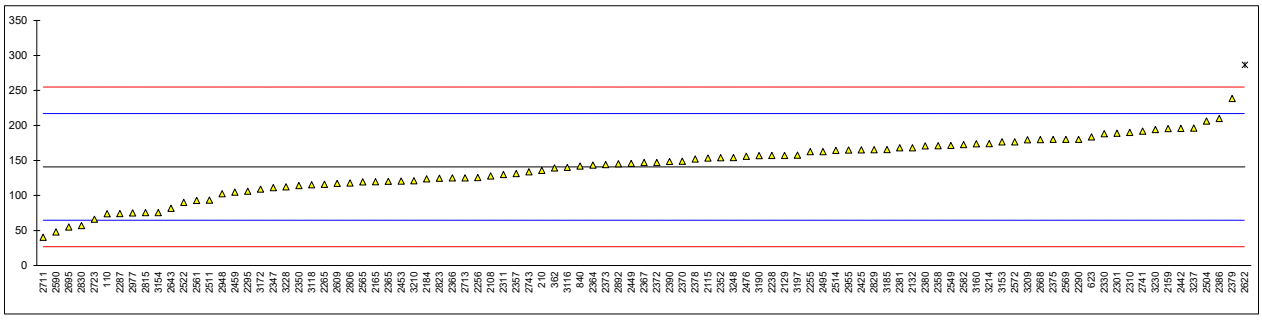
APPENDIX 1

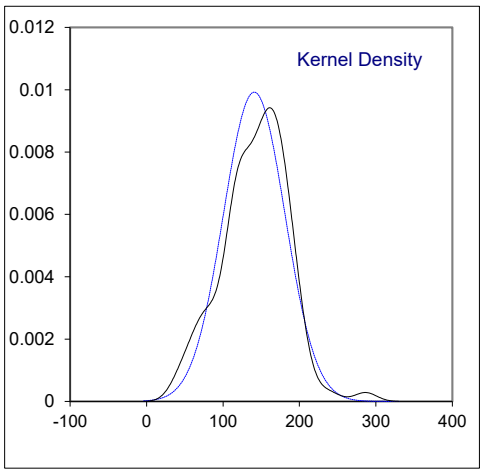
Determination of Benzidine (CAS No. 92-87-5) in sample #22510; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		73.78		-1.76	
210	ISO17234-1	135.73		-0.14	
362		139.2		-0.04	
551		-----		-----	
623	ISO17234-1	183.564		1.12	
840	ISO17234-1	142.0		0.03	
2108	ISO17234-1	127.8		-0.34	
2115	ISO17234-1	153.4		0.33	
2129	ISO17234-1	157		0.42	
2132	ISO17234-1	168		0.71	
2159	ISO17234-1	195.51		1.44	
2165	ISO17234-1	119.4		-0.56	
2184	ISO17234-1	123.7		-0.45	
2238	ISO17234-1	156.9		0.42	
2255	ISO17234-1	162.47		0.57	
2256	ISO17234-1	125.42		-0.41	
2265	ISO17234-1	115.9		-0.66	
2287	ISO17234-1	73.92	C	-1.76	first reported 35.37
2290	ISO17234-1	180.1		1.03	
2295	ISO17234-1	106		-0.92	
2301	ISO17234-1	189		1.27	
2310	ISO17234-1	190		1.29	
2311	ISO17234-1	129.9		-0.29	
2330	ISO17234-1	188.027		1.24	
2347	ISO17234-1	111		-0.79	
2350	ISO17234-1	114.08		-0.70	
2352	ISO17234-1	153.7		0.34	
2357	ISO17234-1	131.1		-0.26	
2358	ISO17234-1	170.87		0.79	
2364	ISO17234-1	143.5		0.07	
2365	ISO17234-1	120.2		-0.54	
2366	ISO17234-1	125		-0.42	
2367	ISO17234-1	146.94		0.16	
2370	ISO17234-1	148.83		0.21	
2372	ISO17234-1	147.03		0.16	
2373	ISO17234-1	144.01		0.08	
2375	ISO17234-1	180		1.03	
2378	ISO17234-1	152		0.29	
2379	ISO17234-1	238.3926		2.56	
2380	ISO17234-1	170.80		0.79	
2381	ISO17234-1	168.00		0.71	
2386	ISO17234-1	209.9		1.81	
2390	ISO17234-1	148.53		0.20	
2425	In house	165.0		0.63	
2442	ISO17234-1	195.63		1.44	
2449	ISO17234-1	145.64		0.13	
2453	ISO17234-1	120.61		-0.53	
2455		-----		-----	
2459	ISO17234-1	104.6		-0.95	
2476	ISO17234-1	155.92		0.40	
2495	ISO17234-1	162.53		0.57	
2504	ISO17234-1	206.140		1.72	
2511	ISO17234-1	93.31		-1.25	
2514	ISO17234-1	164.38		0.62	
2522	GB/T19942	90.052		-1.34	
2549		171.42		0.80	
2561	ISO17234-1	92.795		-1.26	
2565	ISO17234-1	119.260		-0.57	
2569		180		1.03	
2572		176.5		0.94	
2582	ISO17234-1	172.65		0.84	
2590	ISO17234-1	47.8		-2.45	
2609	ISO17234-1	117.082		-0.63	
2622	ISO17234-1	286.43	R(0.05)	3.83	
2643	ISO17234-1	81.53		-1.56	
2668	ISO17234-1	179.51		1.02	
2695	ISO17234-1	55.000		-2.26	
2711	ISO17234-1	40.1		-2.65	
2713	ISO17234-1	125.096		-0.41	
2723	ISO17234-1	66		-1.97	
2741	ISO17234-1	191.840		1.34	
2743	ISO17234-1	133.71		-0.19	
2756		-----		-----	
2806	ISO17234-1	117.8		-0.61	
2815	ISO17234-1	75.5		-1.72	

lab	method	value	mark	z(targ)	remarks
2823	ISO17234-1	124.503		-0.43	
2829	ISO17234-1	165.222		0.64	
2830		56.98		-2.21	
2892	ISO17234-1	145.000		0.11	
2948	ISO17234-1	102.35		-1.01	
2955	ISO17234-1	164.7		0.63	
2977	ISO17234-1	75.0		-1.73	
3116	ISO17234-1	140		-0.02	
3118	ISO17234-1	115.2969		-0.67	
3153	ISO17234-1	176.4		0.93	
3154	ISO17234-1	75.6		-1.72	
3160	ISO17234-1	173.74		0.86	
3172	ISO17234-1	109.02		-0.84	
3185	ISO17234-1	165.38		0.64	
3190	ISO17234-1	156.72		0.42	
3197	ISO17234-1	157.6		0.44	
3209	ISO17234-1	179.31		1.01	
3210	In house	121		-0.52	
3214	ISO17234-1	173.82		0.87	
3228	ISO17234-1	112.4		-0.75	
3230	In house	194.0366		1.40	
3237	ISO17234-1	196.00		1.45	
3248	ISO17234-1	154		0.35	

	normality	OK	
	n	94	
	outliers	1	
	mean (n)	140.8733	
	st.dev. (n)	40.20694	RSD=29%
	R(calc.)	112.5794	
	st.dev.(iis memo 2202)	38.03578	
	R(iis memo 2202)	106.5002	





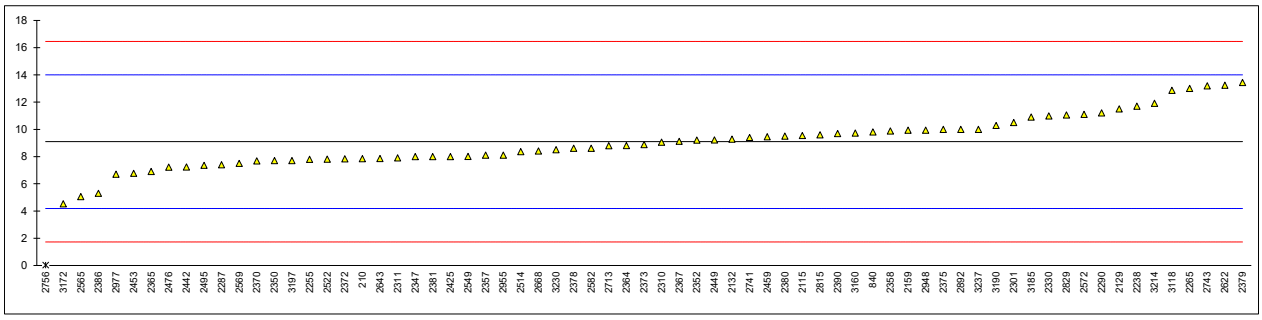
Determination of 2,4-Xylidine (CAS No. 95-68-1) in sample #22510; results in mg/kg

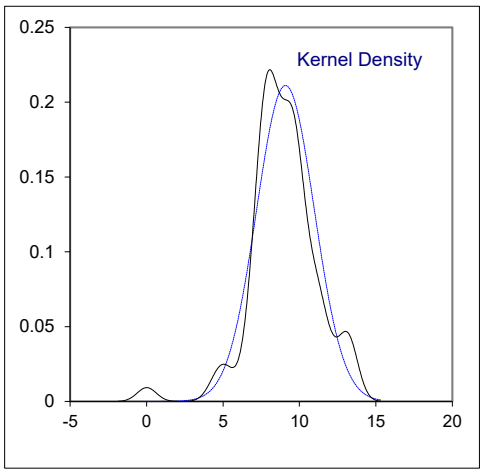
lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO17234-1	7.84		-0.51	
362		-----		-----	
551		-----		-----	
623	ISO17234-1	not detected		-----	
840	ISO17234-1	9.8		0.29	
2108	ISO17234-1	not detected		-----	
2115	ISO17234-1	9.54		0.18	
2129	ISO17234-1	11.5		0.98	
2132	ISO17234-1	9.28		0.08	
2159	ISO17234-1	9.93		0.34	
2165		-----	W	-----	test result withdrawn, reported not detected
2184		-----		-----	
2238	ISO17234-1	11.7		1.06	
2255	ISO17234-1	7.8		-0.53	
2256		-----		-----	
2265	ISO17234-1	13	C	1.59	first reported <5
2287	ISO17234-1	7.40		-0.69	
2290	ISO17234-1	11.2		0.86	
2295		-----		-----	
2301	ISO17234-1	10.5		0.57	
2310	ISO17234-1	9.05		-0.02	
2311	ISO17234-1	7.9		-0.49	
2330	ISO17234-1	10.98	C	0.77	first reported not detected
2347	ISO17234-1	8	C	-0.45	first reported <5
2350	ISO17234-1	7.70		-0.57	
2352	ISO17234-1	9.2		0.04	
2357	ISO17234-1	8.1		-0.40	
2358	ISO17234-1	9.88		0.32	
2364	ISO17234-1	8.8		-0.12	
2365	ISO17234-1	6.9		-0.89	
2366		-----		-----	
2367	ISO17234-1	9.12		0.01	
2370	ISO17234-1	7.68		-0.58	
2372	ISO17234-1	7.83		-0.51	
2373	ISO17234-1	8.88		-0.09	
2375	ISO17234-1	10		0.37	
2378	ISO17234-1	8.6		-0.20	
2379	ISO17234-1	13.4380		1.77	
2380	ISO17234-1	9.50		0.17	
2381	ISO17234-1	8.00		-0.45	
2386	ISO17234-1	5.3		-1.55	
2390	ISO17234-1	9.68		0.24	
2425	In house	8.00		-0.45	
2442	ISO17234-1	7.23		-0.76	
2449	ISO17234-1	9.21		0.05	
2453	ISO17234-1	6.77		-0.95	
2455		-----		-----	
2459	ISO17234-1	9.45		0.15	
2476	ISO17234-1	7.22		-0.76	
2495	ISO17234-1	7.353		-0.71	
2504	ISO17234-1	Not applicable	C	-----	first reported not detected
2511	ISO17234-1	<5		-----	
2514	ISO17234-1	8.35		-0.30	
2522	GB/T19942	7.812		-0.52	
2549		8.01		-0.44	
2561		-----		-----	
2565	ISO17234-1	5.062		-1.64	
2569		7.5		-0.65	
2572		11.1		0.82	
2582	ISO17234-1	8.61		-0.20	
2590		-----		-----	
2609		-----		-----	
2622	ISO17234-1	13.24		1.69	
2643	ISO17234-1	7.85		-0.51	
2668	ISO17234-1	8.41		-0.28	
2695	ISO17234-1	-----		-----	
2711	ISO17234-1	<5		-----	
2713	ISO17234-1	8.792	C	-0.12	first reported <5
2723	ISO17234-1	5-10	C	-----	first reported not detected
2741	ISO17234-1	9.401		0.13	
2743	ISO17234-1	13.19		1.67	
2756	ISO17234-1	0.02	R(0.01)	-3.70	
2806	ISO17234-1	<30	C	-----	first reported <LQ
2815	ISO17234-1	9.6	C	0.21	first reported not detected

lab	method	value	mark	z(targ)	remarks
2823		-----		-----	
2829	ISO17234-1	11.058		0.80	
2830		-----		-----	
2892	ISO17234-1	10.000		0.37	
2948	ISO17234-1	9.93		0.34	
2955	ISO17234-1	8.1		-0.40	
2977	ISO17234-1	6.7		-0.97	
3116		-----		-----	
3118	ISO17234-1	12.8540	C	1.53	first reported 15.269
3153	ISO17234-1	<5		-----	
3154		-----		-----	
3160	ISO17234-1	9.73		0.26	
3172	ISO17234-1	4.52	C	-1.86	first reported <1
3185	ISO17234-1	10.90		0.74	
3190	ISO17234-1	10.28		0.48	
3197	ISO17234-1	7.7		-0.57	
3209		-----		-----	
3210		-----		-----	
3214	ISO17234-1	11.91		1.15	
3228		-----	W	-----	test result withdrawn, reported not detected
3230	In house	8.49066	C	-0.25	first reported not detected
3237	ISO17234-1	10.00		0.37	
3248	ISO17234-1	<10		-----	

normality	OK
n	68
outliers	1
mean (n)	9.0935
st.dev. (n)	1.88873
R(calc.)	5.2884
st.dev.(iis memo 2202)	2.45526
R(iis memo 2202)	6.8747

RSD=21%



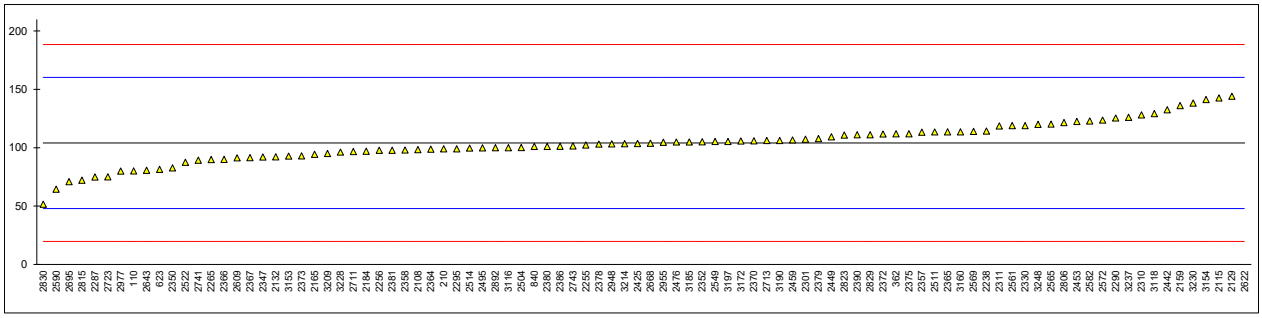


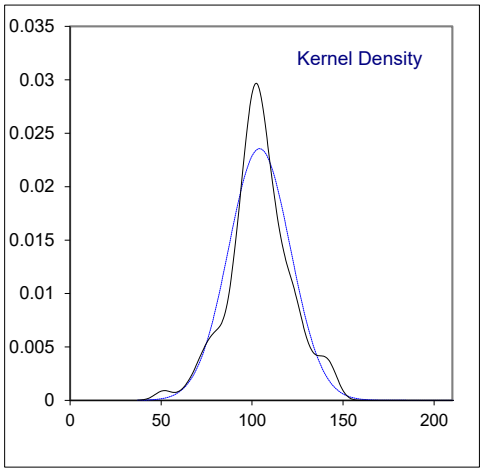
Determination of 3,3'-Dimethylbenzidine (CAS No. 119-93-7) in sample #22511; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO17234-1	80.05		-0.86	
210	ISO17234-1	98.99		-0.18	
362		112.0		0.28	
551		-----		-----	
623	ISO17234-1	81.487		-0.80	
840	ISO17234-1	101.0		-0.11	
2108	ISO17234-1	98.3		-0.21	
2115	ISO17234-1	142.72		1.37	
2129	ISO17234-1	144		1.42	
2132	ISO17234-1	92.1		-0.43	
2159	ISO17234-1	136.06		1.14	
2165	ISO17234-1	94.3		-0.35	
2184	ISO17234-1	97.0		-0.25	
2238	ISO17234-1	114.2		0.36	
2255	ISO17234-1	102.3		-0.06	
2256	ISO17234-1	97.69		-0.23	
2265	ISO17234-1	89.9		-0.51	
2287	ISO17234-1	74.84		-1.04	
2290	ISO17234-1	125.4		0.76	
2295	ISO17234-1	99		-0.18	
2301		107.3		0.11	
2310	ISO17234-1	128		0.85	
2311	ISO17234-1	118.7		0.52	
2330	ISO17234-1	118.984		0.53	
2347	ISO17234-1	92		-0.43	
2350		82.79		-0.76	
2352	ISO17234-1	105.1		0.04	
2357	ISO17234-1	113.2		0.32	
2358	ISO17234-1	97.96		-0.22	
2364	ISO17234-1	98.7		-0.19	
2365	ISO17234-1	113.6		0.34	
2366	ISO17234-1	90		-0.50	
2367	ISO17234-1	91.52		-0.45	
2370	ISO17234-1	105.79		0.06	
2372	ISO17234-1	111.7		0.27	
2373	ISO17234-1	93.08		-0.39	
2375	ISO17234-1	112		0.28	
2378	ISO17234-1	103		-0.04	
2379	ISO17234-1	107.7335		0.13	
2380	ISO17234-1	101.00		-0.11	
2381	ISO17234-1	97.70		-0.23	
2386	ISO17234-1	101.2		-0.10	
2390	ISO17234-1	111.08		0.25	
2425	In house	103.6		-0.02	
2442	ISO17234-1	132.46		1.01	
2449		109.25		0.18	
2453	ISO17234-1	122.47		0.65	
2455		-----		-----	
2459	ISO17234-1	106.67		0.09	
2476	ISO17234-1	104.86		0.03	
2495	ISO17234-1	99.87		-0.15	
2504	ISO17234-1	100.155	C	-0.14	first reported 207.533
2511	ISO17234-1	113.5169		0.34	
2514	ISO17234-1	99.62		-0.16	
2522	In house	87.472		-0.59	
2549		105.35		0.04	
2561	ISO17234-1	118.89		0.53	
2565	ISO17234-1	120.229		0.57	
2569		114		0.35	
2572		123.6		0.69	
2582	ISO17234-1	122.83		0.67	
2590	ISO17234-1	64.5		-1.41	
2609	ISO17234-1	91.214		-0.46	
2622	ISO17234-1	287.19	R(0.01)	6.51	
2643	ISO17234-1	80.54		-0.84	
2668	ISO17234-1	103.88		-0.01	
2695		70.996		-1.18	
2711	ISO17234-1	96.7		-0.26	
2713	ISO17234-1	106.043		0.07	
2723	ISO17234-1	75		-1.04	
2741	ISO17234-1	89.244		-0.53	
2743	ISO17234-1	101.53		-0.09	
2756		-----		-----	
2806	ISO17234-1	121.5		0.62	
2815	ISO17234-1	72.1		-1.14	

lab	method	value	mark	z(targ)	remarks
2823	ISO17234-1	110.848		0.24	
2829	ISO17234-1	111.096		0.25	
2830		51.5		-1.87	
2892	ISO17234-1	100.000		-0.15	
2948	ISO17234-1	103.25		-0.03	
2955	ISO17234-1	104.6		0.02	
2977	ISO17234-1	79.9		-0.86	
3116	ISO17234-1	100		-0.15	
3118		129.1724		0.89	
3153	ISO17234-1	92.8		-0.40	
3154	ISO17234-1	141.3		1.32	
3160	ISO17234-1	113.63		0.34	
3172	ISO17234-1	105.7		0.06	
3185	ISO17234-1	105.00		0.03	
3190	ISO17234-1	106.10		0.07	
3197	ISO17234-1	105.4		0.05	
3209	ISO17234-1	95.02		-0.32	
3210		-----		-----	
3214	ISO17234-1	103.43		-0.02	
3228	ISO17234-1	96.2		-0.28	
3230	In house	138.2085		1.21	
3237	ISO17234-1	126.00		0.78	
3248	ISO17234-1	120		0.57	

normality	OK
n	93
outliers	1
mean (n)	104.0938
st.dev. (n)	16.93759 RSD=16%
R(calc.)	47.4253
st.dev.(iis memo 2202)	28.10532
R(iis memo 2202)	78.6949





APPENDIX 2

Other reported aromatic amines; results in mg/kg

4AD = 4-Aminodiphenyl (CASno. 92-67-1)
B = Benzidine (CASno. 92-87-5)
4CoT = 4-Chloro-o-toluidine (CASno. 95-69-2)
2NA = 2-Naphtylamine (CASno. 91-59-8)
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'-Diaminodiphenylmethane (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'-Diaminodiphenylmethane (CASno. 838-88-0)
pC = p-Cresidine (CASno. 120-71-8)
DDM = 4,4'-Diamino-3,3'-dichlorodiphenylmethane (CASno. 101-14-4)
DDE = 4,4'-Diaminodiphenylether (CASno. 101-80-4)
DDS = 4,4'-Diaminodiphenylsulfide (CASno. 139-65-1)
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 Xylidines
oAAT = o-Aminoazotoluene (CASno. 97-56-3)
oT = o-Toluidine (CASno. 95-53-4)
SUM = Sum of o-Aminoazotoluene and o-Toluidine

sample #22510

lab	4AD	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM
110	2.98	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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.
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2108	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2115	4.14	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2129	4.63	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2159	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2184	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2255	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2256	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2265	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	<5
2287	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2290	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2295	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2301	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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.
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	<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	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2367	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2370	3.49	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3
2372	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2373	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	ANT	4CA	DAA	DADM	DCB	DMoxB	DMB	DDDM
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2378	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2379	7.1334	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
2381	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2390	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2425	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2442	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2449	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2459	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2476	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2495	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2504	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2511	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2514	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2522	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2549	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2565	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2569	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ
2572	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2582	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2590	-----	-----	-----	-----	-----	-----	-----	-----	-----	12.7	-----
2609	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2622	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2643	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2668	<5.0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2695	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2711	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2713	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2723	5	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
2743	3.99	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2756	0.27	-----	2.26	-----	0.25	-----	-----	-----	-----	-----	-----
2806	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ
2815	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2823	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2829	5.098	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2830	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2892	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2948	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2955	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2977	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	< 5.0
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3118	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	-----	-----	-----	-----	-----	-----	-----	-----	-----	3873	-----
3160	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3172	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	< 1
3185	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3190	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3209	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3214	< 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.
3230	6.0933	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3248	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10

n.d. = not detected

sample #22510 -continued; abbreviations components explained at start of appendix 2

lab	pC	DDM	DDE	DDS	24DAT	TMA	oA	25X	26X	TX	oAAT	oT	SUM
110	-----	-----	-----	-----	-----	-----	-----	-----	3.33	-----	-----	-----	-----
210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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.	n.d.
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	9.8	n.d.	n.d.	n.d.
2108	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	10	n.d.	n.d.	n.d.
2115	-----	-----	-----	-----	-----	-----	-----	-----	-----	9.54	-----	-----	-----
2129	<5	<5	<5	<5	<5	<5	<5	5.07	<5	16.6	<5	<5	<5
2132	<5	<5	<5	<5	<5	<5	<5	NA	<5	NA	<5	<5	NA
2159	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	13.63	<5.0	<5.0	<5.0
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	-----	-----	n.d.	n.d.	-----
2184	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	-----	-----	n.d.	n.d.	-----
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	11.7	<5	<5	<5
2255	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7.8	n.d.	n.d.	n.d.
2256	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2265	<5	<5	<5	<5	<5	<5	<5	<5	<5	< 5	<5	<5	<5
2287	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	<10
2290	<5	<5	<5	<5	<5	<5	<5	<5	<5	11.2	<5	<5	<5
2295	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2301	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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.	n.d.
2311	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7.9	n.d.	n.d.	n.d.
2330	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	5.608	16.588	n.d.	n.d.	n.d.
2347	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	<5
2350	<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.	n.d.	n.d.
2364	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	6.9	<5	<5	<5
2366	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2367	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	-----
2370	<3	<3	<3	<3	<3	<3	<3	<3	<3	7.68	<3	<3	<3
2372	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	154.86
2373	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	8.88	n.d.	n.d.	n.d.
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2378	<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	9.50	<5	<5	<5
2381	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	5.3	<5	<5	<5
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.	n.d.
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.	n.d.
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.	n.d.
2449	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2459	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2476	-----	-----	-----	-----	-----	-----	-----	-----	-----	7.22	-----	-----	-----
2495	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	-----	<5	-----
2504	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.
2511	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2514	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2522	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	N/A	<5.0	N/A	<5.0	<5.0	<5.0
2549	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	8.01	n.d.	n.d.	n.d.
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2565	<5	<5	<5	<5	<5	<5	<5	<5	5.259	10.321	<5	<5	<5
2569	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	-----	<LQ	-----	<LQ	<LQ	-----
2572	<5	<5	<5	<5	<5	<5	<5	<5	<5	11.1	<5	<5	<5
2582	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	n.d.
2590	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2609	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2622	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	13.24	n.d.	n.d.	n.d.
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.	n.d.
2695	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2711	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	-----
2713	<5	<5	<5	<5	<5	<5	<5	-----	<5	<5	<5	<5	<5
2723	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.
2741	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	-----
2743	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	1.37	14.56	n.d.	n.d.	n.d.
2756	1.43	-----	-----	-----	0.77	0.09	4.86	-----	-----	-----	-----	-----	-----
2806	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	< LQ	<LQ	<LQ	-----
2815	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	-----
2823	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

lab	pC	DDM	DDE	DDS	24DAT	TMA	oA	25X	26X	TX	oAAT	oT	SUM
2829	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5.426	16.484	n.d.	n.d.	n.d.
2830	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2892	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2948	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5.5	15.43	n.d.	n.d.	n.d.
2955	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.
2977	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	6.7	< 5.0	< 5.0	< 5.0
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3118	ND	ND	ND	ND	ND	ND	ND	ND	ND	-----	ND	ND	ND
3153	<5	<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.	-----
3172	< 1	< 1	< 1	< 1	< 1	< 1	< 1	-----	< 1	-----	< 1	< 1	-----
3185	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	<5
3190	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	<5
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	7.7	<5	<5	<5
3209	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3214	<5	<5	<5	<5	<5	<5	<5	<5	<5	11.91	<5	<5	<5
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	-----	-----	n.d.	n.d.	-----
3230	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	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10

n.d. = not detected
n.a. = not applicable

sample #22511; abbreviations components explained at start of appendix 2

lab	4AD	B	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB	DDDM	pC
110	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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.
2108	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2115	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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
2159	<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	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
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.
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2265	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2287	<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
2295	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2301	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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	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	<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.	n.d.
2364	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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.
2370	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3
2372	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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.	n.d.
2380	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2381	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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.
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.
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	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2459	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2476	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2495	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2504	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2511	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2514	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2522	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
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.
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2565	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2569	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ
2572	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2582	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	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2609	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2622	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	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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.
2695	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2711	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2713	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2723	n.d.	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	<5
2743	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	0.28	-----	-----	1.13	-----	0.71	-----	-----	-----	-----	-----	0.25
2806	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ
2815	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	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	94.076	-----

lab	4AD	B	4CoT	2NA	ANT	4CA	DAA	DADM	DCB	DMoxB	DDDM	pC
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.
2830	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2892	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2948	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2955	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2977	<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
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	-----	354	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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.
3172	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
3185	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3190	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3209	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----	-----	-----	-----	76.8	-----	-----
3214	<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.
3230	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	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10

n.d. = not detected

sample #22511 -continued; abbreviations components explained at start of appendix 2

lab	DDM	DDE	DDS	24DAT	TMA	oA	24X	25X	26X	TX	oAAT	oT	SUM
110	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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.	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.	n.d.
2108	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	n.d.	n.d.	n.d.	n.d.
2115	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2129	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2132	<5	<5	<5	<5	<5	<5	<5	NA	<5	NA	<5	<5	NA
2159	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	-----
2184	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	-----	-----	-----	n.d.	n.d.	-----
2238	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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.	n.d.
2256	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2265	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2287	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	<10
2290	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2295	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2301	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
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.	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.	n.d.
2330	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.
2347	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	<5
2350	<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.	n.d.	n.d.
2364	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2367	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	-----
2370	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3
2372	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	111.7
2373	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2378	<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.	n.d.
2380	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2381	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
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.	n.d.
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.	n.d.
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.	n.d.
2449	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2459	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2476	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2495	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	-----	<5	-----
2504	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.
2511	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2514	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2522	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	N/A	<5.0	N/A	<5.0	<5.0	<5.0
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.	n.d.
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2565	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2569	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	-----	<LQ	-----	<LQ	<LQ	-----
2572	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2582	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	n.d.
2590	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2609	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2622	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.
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.	n.d.
2695	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2711	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	-----
2713	<5	<5	<5	<5	<5	<5	<5	-----	<5	<5	<5	<5	<5
2723	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.
2741	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	-----
2743	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	-----	-----	-----	1.57	0.047	6.04	0.027	-----	-----	-----	-----	0.036	-----
2806	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	<LQ	-----
2815	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----	n.d.	n.d.	-----
2823	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

lab	DDM	DDE	DDS	24DAT	TMA	oA	24X	25X	26X	TX	oAAT	oT	SUM
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.	n.d.
2830	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2892	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2948	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.
2955	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.
2977	<5	<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	ND
3153	<5	<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.	-----
3172	<1	<1	<1	<1	<1	<1	<1	-----	<1	-----	<1	<1	-----
3185	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	<5
3190	<5	<5	<5	<5	<5	<5	<5	-----	<5	-----	<5	<5	<5
3197	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3209	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3214	<5	<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.	-----
3230	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	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10

n.d. = not detected
n.a. = not applicable

APPENDIX 3 Analytical details

lab	ISO/IEC17025 accredited	sample intake (g)	sample pre-treatment
110	Yes	1.0	Further cut
210	Yes	1	Further cut
362	Yes	0.5	Used as received
551	---	---	---
623	Yes	1	Used as received
840	Yes	0.5	Further cut
2108	Yes	0.5	Used as received
2115	Yes	0.5	Used as received
2129	Yes	0.50	Used as received
2132	Yes	1	Used as received
2159	Yes	1.0	Used as received
2165	Yes	1.0	Used as received
2184	Yes	2	Used as received
2238	Yes	1	Used as received
2255	Yes	0.5	Further cut
2256	Yes	1.0049	Used as received
2265	No	0.5	Used as received
2287	Yes	1.0	Further cut
2290	---	---	---
2295	Yes	1	Further cut
2301	Yes	1	Used as received
2310	Yes	0.5	Further cut
2311	Yes	0.5	Further cut
2330	Yes	1	Further cut
2347	Yes	0.5	Used as received
2350	Yes	#22510: 0.9169 / #22511: 0.831	Further cut
2352	Yes	0.5	Further cut
2357	---	---	---
2358	Yes	1.0	Used as received
2364	Yes	#22510: 0.5013 / #22511: 0.5075	Used as received
2365	Yes	0.5	Used as received
2366	Yes	---	Further cut
2367	Yes	#22510: 0.5043 / #22511: 0.5022	Used as received
2370	Yes	0.5	Further cut
2372	Yes	1	Used as received
2373	Yes	0.5	Used as received
2375	Yes	1	Further cut
2378	No	1	Further cut
2379	Yes	1	Further cut
2380	No	0.50	Used as received
2381	Yes	0.5	Further cut
2386	Yes	0.5	Used as received
2390	Yes	1	#22510: Used as received / #22511: Further cut
2425	Yes	0.5	Further cut
2442	Yes	#22510: 0.5087 / #22510: 0.5043	Used as received
2449	Yes	1	Further cut
2453	No	±1	Used as received
2455	---	---	---
2459	---	1.0	---
2476	---	1	---
2495	Yes	0.5	Used as received
2504	Yes	0.5	Further cut
2511	Yes	3	Used as received
2514	Yes	#22510= 0.6345 #22511=0.5009	Further cut
2522	No	0.5	Further cut
2549	Yes	1	NA
2561	Yes	1	Used as received
2565	Yes	1	Used as received
2569	Yes	1	Used as received
2572	Yes	---	---
2582	Yes	#22510: 1.0055 / #22511: 1.0079	Used as received
2590	Yes	1	Further cut
2609	Yes	1.00	Used as received
2622	Yes	Approx. 1	Used as received
2643	Yes	1	Used as received
2668	Yes	0.5	Further cut
2695	Yes	1	Used as received
2711	No	1.0	Used as received
2713	Yes	0.5	#22510: Used as received / #22511: Further cut
2723	Yes	1	Used as received
2741	Yes	0.5	Further cut
2743	No	1	Used as received
2756	Yes	---	Used as received
2806	Yes	---	---
2815	Yes	1.00 - 1.05	Used as received

lab	ISO/IEC17025 accredited	sample intake (g)	sample pre-treatment
2823	No	#22510: 1.0110 / #22511: 1.0056	Used as received
2829	No	1	Further cut
2830	Yes	1.0	Used as received
2892	Yes	1.0	Further cut
2948	Yes	1	Further grinded
2955	Yes	0.6	Further cut
2977	No	3.5	Further cut
3116	Yes	1	Used as received
3118	Yes	0.5	Further cut
3153	Yes	0.5	Used as received
3154	Yes	0.5	Used as received
3160	Yes	0.75	Further cut
3172	---	---	---
3185	Yes	1	Used as received
3190	Yes	1	Used as received
3197	Yes	0.5	Further cut
3209	Yes	0.5	Further cut
3210	Yes	1	Used as received
3214	Yes	1	Further cut
3228	Yes	0.5	#22510: Used as received / #22511: Further cut
3230	Yes	1.0	Further cut
3237	Yes	0.5	Further cut
3248	Yes	1	Used as received

APPENDIX 4

Number of participants per country

7 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in CAMBODIA
1 lab in EGYPT
1 lab in ETHIOPIA
1 lab in FRANCE
5 labs in GERMANY
6 labs in HONG KONG
5 labs in INDIA
3 labs in INDONESIA
11 labs in ITALY
2 labs in JAPAN
2 labs in KOREA, Republic of
1 lab in MAURITIUS
2 labs in MOROCCO
18 labs in P.R. of CHINA
4 labs in PAKISTAN
1 lab in PORTUGAL
1 lab in SPAIN
1 lab in SRI LANKA
2 labs in SWITZERLAND
3 labs in TAIWAN
2 labs in THAILAND
1 lab in TUNISIA
6 labs in TURKEY
2 labs in U.S.A.
2 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
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?
SDS	= Safety Data Sheet

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