

**Results of Proficiency Test
AZO dyes in leather
February 2016**

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis16A02

May 2016

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1 INTRODUCTION

The Institute for Interlaboratory Studies (iis) organizes every year a proficiency test for banned AZO dyes in leather since 1997, with an exception in 2009. During the annual proficiency testing program 2015/2016, it was decided to continue the proficiency test for the analysis of banned AZO dyes in leather. In this interlaboratory study, 113 laboratories in 30 different countries have registered for participation (see appendix 4). In this report, the results of the 2016 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 in Spijkensisse was the organizer of this proficiency test. Due to lack of a sufficient amount of suitable materials it was decided to use in this proficiency test only one leather sample. It was decided to use a sample which was used in an earlier proficiency test of iis (iis00A01). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. It was decided to send one sample of approximately 3.5 grams, labelled #16520.

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 Spijkensisse, 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 April 2014 (iis-protocol, version 3.3). 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 brown coloured leather sample, positive on AZO dyes, was used in an earlier proficiency test of iis (iis00A01). After cutting it into small pieces, the material was mixed thoroughly. In total 180 sub samples were prepared of 3.5 gram leather each and subsequently labelled #16520. Five stratified randomly selected samples were tested using HPLC to check the homogeneity of the batch. See the following table for the test results.

	<i>Benzidine in mg/kg</i>	<i>3,3'-Dimethylbenzidine in mg/kg</i>
sample #16520-1	11.4	11.0
sample #16520-2	12.9	12.1
sample #16520-3	13.8	13.2
sample #16520-4	11.9	11.4
sample #16520-5	15.5	13.2

Table 1: homogeneity test results of subsamples #16520

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

	<i>Benzidine in mg/kg</i>	<i>3,3'-Dimethylbenzidine in mg/kg</i>
r (observed)	4.6	2.8
reference test method	ISO17234-1:2015	ISO17234-1:2015
r (reference test method)	3.8	4.0
0.3 * R (calc) of iis00A01	7.1	10.6

Table 2: evaluation of the repeatabilities of aromatic amines in subsamples #16520

The calculated repeatability of the test results of homogeneity tests for 3,3'-Dimethylbenzidine was in agreement with the estimated repeatability mentioned in the reference test method ISO17234-1:2015. Unfortunately the calculated repeatability of the test results of homogeneity tests for Benzidine was not in agreement with the estimated repeatability mentioned in the reference test method ISO17234-1:2015. However, the calculated repeatability of Benzidine is smaller than 0.3 times the calculated reproducibility of proficiency test from 2000 (iis00A01), in which PT this material was used before. Therefore, homogeneity of the subsamples was assumed.

One sample containing approximately 3.5 grams testing material labelled #16520 was sent to each of the participating laboratories on February 10, 2016.

2.5 ANALYSES

The participants were requested to determine the concentrations of 23 forbidden aromatic amines and *o*-anisidine, applying the analysis procedure that is routinely used in the laboratory. To get comparable test results a detailed report form, on which the requested amines and the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The laboratories were also requested to report some of the analytical details that the laboratory had used.

A form to confirm receipt of the sample and a letter of instructions were added to the sample package.

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 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and the original reported test results 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.

A list of abbreviations used in the tables can be found in appendix 5.

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 April 2014 (iis-protocol, version 3.3).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were in general 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. The statistical evaluation of the test results should be used with due care in case that a data set does not prove to have a normal distribution.

In accordance to ISO 5725 the original test results per determination were submitted subsequently to Dixon's and 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 the averages and the 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 consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

The z-scores were calculated according to:

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

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

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare. 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. Fortunately all participants were able to report in time for the evaluation of the test results. Nine participants reported test results after the deadline. The 113 participants did report 205 numerical test results in total for Benzidine, 3,3'-Dimethylbenzidine and o-Toluidine, while another 8 test results were reported for other aromatic amines (see appendix 2). Observed were 3 outlying test results, which is 1.4% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

The data sets of the three amines proved to have a normal Gaussian distribution.

4.1 EVALUATION PER COMPONENT

In this section, the test results are discussed per component. All statistical results reported for Benzidine, 3,3'-Dimethylbenzidine and o-Toluidine are summarised in appendix 1 and 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 18 mg/kg may be problematic. Two statistical outliers were observed. Four other participants reported "< 5 mg/kg", which is obviously falsely negative.

The test results reported by the participants vary from <5 – 42 mg/kg. The calculated reproducibility after rejection of the statistical outliers is not in agreement

with the reproducibility requirement estimated from the reference test method ISO 17234-1:2015. However, both the consensus value and the calculated reproducibility are in good agreement with the PT results of 2000 (iis00A01) in which the same material has been used.

3,3'-Dimethylbenzidine (CASno. 119-93-7):

The determination of this aromatic amine at a concentration level of 28 mg/kg was very problematic. However, no statistical outliers were observed. Two participants reported "< 5 mg/kg" or "< 10 mg/kg", which are both obviously falsely negative. The test results reported by the participants vary from <5 – 56 mg/kg. The calculated reproducibility is not at all in agreement with the reproducibility requirement estimated from the reference test method ISO 17234-1:2015. However, both the consensus value and the calculated reproducibility are in good agreement with the PT results of 2000 (iis00A01) in which the same material has been used.

o-Toluidine (CASno. 95-53-4):

The determination of this aromatic amine at a concentration level of 21 mg/kg may be problematic. One statistical outlier was observed.

The test results reported by 24 participants vary from <2 – 56 mg/kg and 54 (!) other participants reported n.d. (or "less than" test result). Therefore it was decided not to calculate the z-scores for o-Toluidine.

The same phenomenon was observed during the 2000 PT iis00A01. A possible explanation for the fact that only 30% of the participating laboratories reported the presence of o-Toluidine and 70% did not detect this component may be the degradation of the rather instable o-aminoazotoluene (CASno. 97-56-3), although none of the participants actually detected this component. This is not strange in view of the relatively low concentration o-Toluidine found in this material, see also the PT report iis15A02T in which both o-aminoazotoluene and o-Toluidine were detected in a textile sample.

General:

Eight participants reported the presence of other aromatic amines at various concentration levels, see appendix 2.

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 reproducibilities 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	83	18	17	10
3,3'-Dimethylbenzidine	mg/kg	96	28	31	16
o-Toluidine	mg/kg	23	(21)	(22)	(20)

Table 3: reproducibility of the aromatic amine in leather sample #16520

Without further statistical calculations, it can be concluded that the group of participating laboratories has problems with the analysis of the three aromatic amines in leather. See also the discussion in paragraphs 4.1 and 6.

5 COMPARISON WITH PREVIOUS INTERLABORATORY STUDIES

The observed variation in the test results for the aromatic amines in the 2016 PT is in agreement in comparison with the variation of the aromatic amine as observed in the previous PTs, see below table. Unfortunately, this PT showed a slight increased variation compared to recent PTs, except for o-Toluidine.

<i>Component</i>	<i>Feb 2016</i>	<i>March 2015</i>	<i>March 2014</i>	<i>March 2013</i>	<i>March 2012</i>	<i>March 2011-2005</i>	<i>Target</i>
4-Aminodiphenyl	n.e.	n.e.	n.e.	n.e.	25%	45%	15 - 30%
Benzidine	34%	n.e.	20%	28%	20%	38 - 66%	15 - 25%
3,3'-Dimethylbenzidine	39%	24%	n.e.	n.e.	n.e.	45 - 55%	17 - 24%
o-Toluidine	(37%)	n.e.	n.e.	n.e.	n.e.	50 - 63%	30 - 37%
2,4-Xylidine	n.e.	n.e.	n.e.	36%	n.e.	16 - 19%	15 - 30%

Table 4: development of the uncertainties over the years

6 DISCUSSION

Over the years iis tried to find the cause for the observed large variation in the reported test results based on the reported analytical details. From the reported test methods it appeared that almost all participants treated the leather samples according test method: ISO17234-1 (or LFBG 82.02.3, which is identical). Four participants reported to have used EN14362-1, which is a test method for determination of AZO-dyes in textile and this test method skips the degrease step. However, three of these laboratories reported to have done the degreasing.

It is remarkable that in spite of almost all participants used test method ISO17234-1, participants reported a large variety of experimental conditions in the analytical details. For example the experimental conditions in the evaporation step varies for the temperature used from room temperature (23°C) to 70°C (!) and for the duration from 3 to 90 minutes.

Method ISO17234-1 mentions in paragraph 9.3 that the methyl t-butyl ether (MTBE) extract should be concentrated under mild conditions; which means: $T < 50^\circ$ under slight vacuum in a rotary vacuum evaporator. The extract should be concentrated to approximately 1 ml and the remainder of the ether should be further evaporated by a slight flow of inert gas (to dryness).

The above mentioned mild conditions are necessary because aromatic amines are not very stable and should be determined after the reductive cleavage with-in 24 hours. However, four participants reported a total duration longer than 24 hours, one of them even a total duration of 7 days (!).

Nevertheless, no correlation could be found between the details of the sample preparation as reported by the laboratories and the test results. Therefore, it can be concluded that the observed variation in this interlaboratory study is not caused by just one critical point in the analysis but by a combination of experimental conditions.

If the test results from participants were evaluated which reported to use the analytical process exactly as mentioned in ISO17234-1 than the calculated reproducibility was slightly better but still not close to the reproducibility requirements estimated from the reference test method ISO 17234-1:2015.

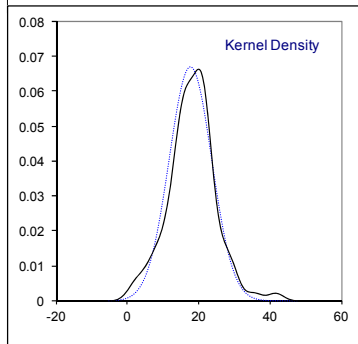
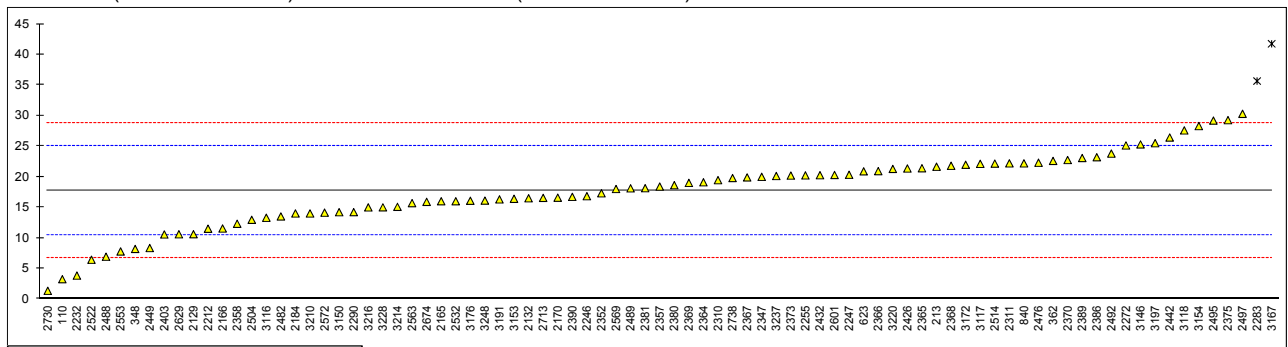
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

APPENDIX 1

Determination of Benzidine (CASno.92-87-5) in sample #16520; test results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO17234-1	3.24		-3.94	
213	EN17324-1	21.65		1.06	
230		----		----	
348	In house	8.19		-2.59	
362	ISO17234-1	22.6		1.32	
551		----		----	
623	ISO17234-1	20.90		0.86	
840	ISO17234-1	22.20		1.21	
1911		----		----	
2102	In house	n.d.		----	
2115		----		----	
2129	ISO17234-1	10.6		-1.94	
2132	ISO17234-1	16.48		-0.34	
2146		----		----	
2165	ISO17234-1	16		-0.47	
2166	EN14362-1	11.54		-1.68	
2170	ISO17234-1	16.58		-0.31	
2184	ISO17234-1	14		-1.02	
2212	ISO17234-1	11.5	C	-1.70	first reported 43.7
2232	EN14362-1	3.83		-3.78	
2246	ISO17234-1	16.82		-0.25	
2247	ISO17234-1	20.33		0.70	
2255	ISO17234-1	20.23		0.68	
2256		----		----	
2272	ISO17234-1	25.13		2.01	
2283	ISO17234-1	35.67	R(0.05)	4.87	
2290	ISO17234-1	14.22		-0.96	
2291	ISO17234-1	n.d.		----	
2296		----		----	
2300	64LFGB82.02-3	n.d.		----	
2310	ISO17234-1	19.45		0.47	
2311	ISO17234-1	22.2		1.21	
2330	ISO17234-1	<5		<-3.46	false negative test result?
2347	ISO17234-1	20		0.62	
2352	ISO17234-1	17.3		-0.12	
2357	ISO17234-1	18.4		0.18	
2358	ISO17234-1	12.31		-1.47	
2364	ISO17234-1	19.1		0.37	
2365	ISO17234-1	21.4		1.00	
2366	ISO17234-1	20.93		0.87	
2367	ISO17234-1	19.9		0.59	
2368	ISO17234-1	21.8		1.10	
2369	ISO17234-1	19		0.34	
2370	ISO17234-1	22.74		1.36	
2373	ISO17234-1	20.2		0.67	
2375	ISO17234-1	29.27		3.13	
2379	ISO17234-1	n.d.		----	
2380	ISO17234-1	18.63		0.24	
2381	ISO17234-1	18.13		0.11	
2386	ISO17234-1	23.2		1.48	
2389	ISO17234-1	23.07		1.45	
2390	ISO17234-1	16.715		-0.28	
2403	ISO17234-1	10.56		-1.95	
2426	ISO17234-1	21.36		0.98	
2432	ISO17234-1	20.26		0.69	
2442	In house	26.43		2.36	
2449	ISO17234-1	8.345		-2.55	
2459	ISO17234-1	<5		<-3.46	false negative test result?
2472	ISO17234-1	<5		<-3.46	false negative test result?
2476	ISO17234-1	22.3		1.24	
2481	In house	n.d.		----	
2482	ISO17234-1	13.51		-1.15	
2488	ISO17234-1	6.92		-2.94	
2489	ISO17234-1	18.11		0.10	
2492	In house	23.8		1.65	
2495	ISO17234-1	29.2		3.12	
2497	ISO17234-1	30.31		3.42	
2499		----		----	
2504	ISO17234-1	12.96		-1.30	
2508		----		----	
2514	ISO17234-1	22.16		1.20	
2516		----		----	
2522	ISO17234-1	6.42		-3.08	
2532	ISO17234-1	16		-0.47	
2553	ISO17234-1	7.78		-2.71	

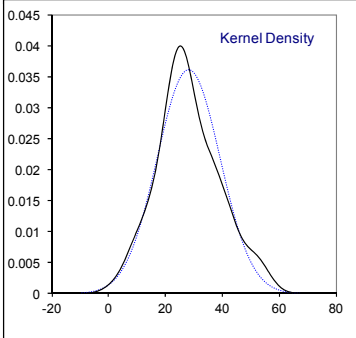
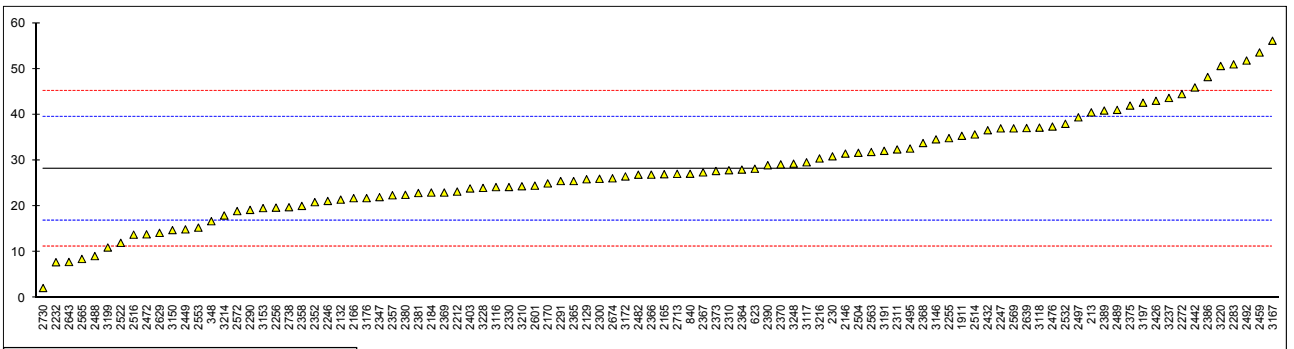
lab	method	value	mark	z(targ)	remarks
2563	ISO17234-1	15.69		-0.56	
2565		----		----	
2569	ISO17234-1	18		0.07	
2572	ISO17234-1	14.13		-0.98	
2590	ISO17234-1	<LOQ		----	
2592		----		----	
2601	ISO17234-1	20.3		0.70	
2612	ISO17234-1	<10		----	
2624	ISO17234-1	n.d.		----	
2629	ISO17234-1	10.59		-1.94	
2639		----		----	
2643	ISO17234-1	<5		<-3.46	false negative test result?
2656	ISO17234-1	<30		----	
2674	ISO17234-1	15.9		-0.50	
2695		----		----	
2713	ISO17234-1	16.55		-0.32	
2730	EN14362-1	1.34		-4.46	
2738	In house	19.8		0.56	
3116	ISO17234-1	13.295		-1.21	
3117	ISO17234-1	22.12		1.19	
3118	ISO17234-1	27.59		2.68	
3146	DIN/EN17234-1	25.3		2.06	
3150	ISO17234-1	14.2		-0.96	
3153	ISO17234-1	16.4		-0.36	
3154	ISO17234-1	28.3		2.87	
3167	ISO17234-1	41.76	R(0.01)	6.53	
3172	ISO17234-1	21.97		1.15	
3176	ISO17234-1	16.08		-0.45	
3191	ISO17234-1	16.3		-0.39	
3197	ISO17234-1	25.5		2.11	
3199	ISO17234-1	<10		----	
3210	In house	14.00		-1.02	
3214	ISO17234-1	15.08		-0.72	
3216	ISO17234-1	14.99		-0.75	
3220	ISO17234-1	21.29		0.97	
3228	ISO17234-1	15		-0.74	
3237	In house	20.13		0.65	
3248	ISO17234-1	16.10		-0.44	
					<u>Compare to iis00A01</u>
normality		OK		OK	
n		83		21	
outliers		2		3	
mean (n)		17.7368		16.33	
st.dev. (n)		5.97220		8.457	
R(calc.)		16.7222		23.68	
R(ISO17234-1:2015)		10.3018	R(LMBG82.02-3:97)	9.47	



Determination of 3,3'-Dimethylbenzidine (CASno.119-93-7) in sample #16520; test results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
213	EN17234-1	40.5		2.18	
230	ISO17234-1	30.9		0.48	
348	In house	16.74		-2.01	
362		----		----	
551		----		----	
623	ISO17234-1	28.19		0.01	
840	ISO17234-1	27.10		-0.19	
1911	ISO17234-1	35.373		1.27	
2102	In house	n.d.		----	
2115		----		----	
2129	ISO17234-1	25.9		-0.40	
2132	ISO17234-1	21.45		-1.18	
2146	ISO17234-1	31.5		0.59	
2165	ISO17234-1	27		-0.20	
2166	EN14362-1	21.77		-1.13	
2170	ISO17234-1	24.99		-0.56	
2184	ISO17234-1	23		-0.91	
2212	ISO17234-1	23.2	C	-0.87	first reported 88.4
2232	EN14362-1	7.79		-3.59	
2246	ISO17234-1	21.16		-1.23	
2247	ISO17234-1	37.0		1.56	
2255	ISO17234-1	34.87		1.18	
2256	ISO17234-1	19.7		-1.49	
2272	ISO17234-1	44.47		2.88	
2283	ISO17234-1	50.97		4.02	
2290	ISO17234-1	19.21		-1.58	
2291	ISO17234-1	25.5		-0.47	
2296		----		----	
2300	64LFGB82.02-3	26		-0.38	
2310	ISO17234-1	27.86		-0.05	
2311	ISO17234-1	32.4		0.75	
2330	ISO17234-1	24.2		-0.70	
2347	ISO17234-1	22		-1.09	
2352	ISO17234-1	20.9		-1.28	
2357	ISO17234-1	22.4		-1.02	
2358	ISO17234-1	20.07		-1.43	
2364	ISO17234-1	28.0		-0.03	
2365	ISO17234-1	25.5		-0.47	
2366	ISO17234-1	26.90		-0.22	
2367	ISO17234-1	27.4		-0.13	
2368	ISO17234-1	33.8		1.00	
2369	ISO17234-1	23		-0.91	
2370	ISO17234-1	29.14		0.17	
2373	ISO17234-1	27.7		-0.08	
2375	ISO17234-1	41.97		2.44	
2379	ISO17234-1	n.d.		----	
2380	ISO17234-1	22.50		-1.00	
2381	ISO17234-1	22.897		-0.93	
2386	ISO17234-1	48.2		3.54	
2389	ISO17234-1	40.89		2.25	
2390	ISO17234-1	28.955		0.14	
2403	ISO17234-1	23.89		-0.75	
2426	ISO17234-1	43.02		2.62	
2432	ISO17234-1	36.60		1.49	
2442	In house	45.93		3.14	
2449	ISO17234-1	14.952		-2.33	
2459	ISO17234-1	53.56		4.48	
2472	ISO17234-1	13.87		-2.52	
2476	ISO17234-1	37.4		1.63	
2481	In house	<5	C	<-4.08	false negative test result? first reported n.d.
2482	ISO17234-1	26.89		-0.22	
2488	ISO17234-1	9.13		-3.36	
2489	ISO17234-1	41.03		2.27	
2492	In house	51.8		4.17	
2495	ISO17234-1	32.6		0.78	
2497	ISO17234-1	39.41		1.99	
2499		----		----	
2504	ISO17234-1	31.67		0.62	
2508		----		----	
2514	ISO17234-1	35.68		1.33	
2516	EN14362-1	13.8		-2.53	
2522	ISO17234-1	12.02		-2.85	
2532	ISO17234-1	38		1.74	
2553	ISO17234-1	15.34		-2.26	

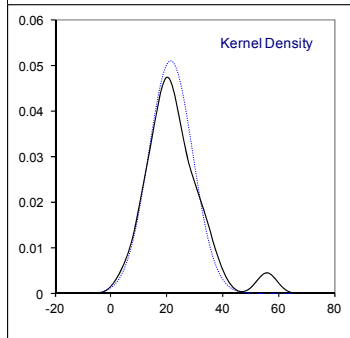
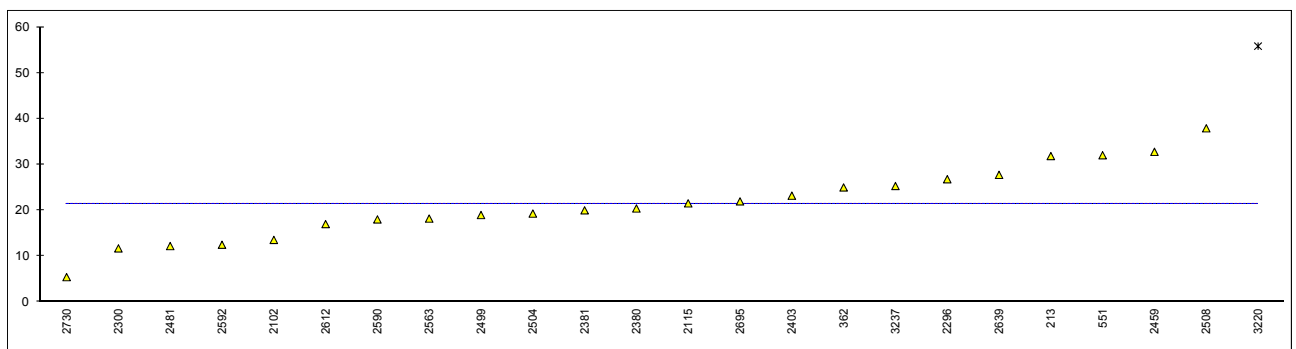
lab	method	value	mark	z(targ)	remarks
2563	ISO17234-1	31.84		0.65	
2565	ISO17234-1	8.5		-3.47	
2569	ISO17234-1	37		1.56	
2572	ISO17234-1	18.94		-1.63	
2590	ISO17234-1	<LOQ		----	
2592		----	W	----	first reported 2.4
2601	ISO17234-1	24.5		-0.64	
2612	ISO17234-1	<10		<-3.20	false negative test result?
2624	ISO17234-1	n.d.		----	
2629	ISO17234-1	14.198		-2.46	
2639	GB/T 19942	37.07		1.57	
2643	ISO17234-1	7.84		-3.58	
2656	ISO17234-1	<30		----	
2674	ISO17234-1	26.1		-0.36	
2695		----		----	
2713	ISO17234-1	27.08		-0.19	
2730	EN14362-1	2.16		-4.59	
2738	In house	19.8		-1.47	
3116	ISO17234-1	24.19		-0.70	
3117	ISO17234-1	29.59		0.25	
3118	ISO17234-1	37.16		1.59	
3146	DIN/EN17234-1	34.6		1.14	
3150	ISO17234-1	14.8		-2.36	
3153	ISO17234-1	19.6		-1.51	
3154		----		----	
3167	ISO17234-1	56.12	C	4.93	first reported 136.9
3172	ISO17234-1	26.51		-0.29	
3176	ISO17234-1	21.78		-1.12	
3191	ISO17234-1	32.1		0.70	
3197	ISO17234-1	42.6		2.55	
3199	ISO17234-1	11.0		-3.03	
3210	In house	24.37		-0.67	
3214	ISO17234-1	17.99		-1.79	
3216	ISO17234-1	30.43		0.40	
3220	ISO17234-1	50.61		3.96	
3228	ISO17234-1	24		-0.73	
3237	In house	43.64		2.73	
3248	ISO17234-1	29.30		0.20	
					<u>Compare to iis00A01</u>
normality		OK		OK	
n		96		27	
outliers		0		3	
mean (n)		28.1560		22.91	
st.dev. (n)		11.02826		12.621	
R(calc.)		30.8791		35.34	
R(ISO17234-1:2015)		15.8726	R(LMBG82.02-3:97)	12.83	



Determination of o-Toluidine (CASno.95-53-4) in sample #16520; test results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
213	EN17234-1	31.85		----	
230		----		----	
348	In house	n.d.		----	
362	ISO17234-1	25.0		----	
551	ISO17234-1	32.02581927		----	
623	ISO17234-1	n.d.		----	
840	ISO17234-1	n.d.		----	
1911		----		----	
2102	In house	13.55		----	
2115	ISO17234-1	21.52		----	
2129		----		----	
2132	ISO17234-1	<5		----	
2146		----		----	
2165	ISO17234-1	n.d.		----	
2166		----		----	
2170		----		----	
2184	ISO17234-1	n.d.		----	
2212	ISO17234-1	<5	C	----	first reported 35.7
2232		----		----	
2246		----		----	
2247	ISO17234-1	n.d.		----	
2255	ISO17234-1	n.d.		----	
2256		----		----	
2272		----		----	
2283		----		----	
2290	ISO17234-1	<5.0		----	
2291	ISO17234-1	n.d.		----	
2296	EN14362-1	26.790		----	
2300	64LFGB82.02-3	11.7		----	
2310	ISO17234-1	n.d.		----	
2311	ISO17234-1	n.d.		----	
2330	ISO17234-1	<5		----	
2347	ISO17234-1	<5		----	
2352		----		----	
2357	ISO17234-1	<5		----	
2358	ISO17234-1	n.d.		----	
2364		----		----	
2365	ISO17234-1	<5		----	
2366	ISO17234-1	<5		----	
2367	ISO17234-1	n.d.		----	
2368	ISO17234-1	n.d.		----	
2369	ISO17234-1	<5		----	
2370	ISO17234-1	n.d.		----	
2373		----		----	
2375		----		----	
2379	ISO17234-1	n.d.		----	
2380	ISO17234-1	20.41		----	
2381	ISO17234-1	20.00		----	
2386	ISO17234-1	<5		----	
2389	ISO17234-1	n.d.		----	
2390	ISO17234-1	n.d.		----	
2403	ISO17234-1	23.20		----	
2426	ISO17234-1	n.d.		----	
2432		----		----	
2442		----		----	
2449		----		----	
2459	ISO17234-1	32.78		----	
2472	ISO17234-1	<5		----	
2476	ISO17234-1	n.d.		----	
2481	In house	12.2		----	
2482		----		----	
2488	ISO17234-1	n.d.		----	
2489	ISO17234-1	n.d.		----	
2492		----		----	
2495	ISO17234-1	<5		----	
2497		----		----	
2499	GB/T 19942	18.986	C	----	first reported 18.92
2504	ISO17234-1	19.30		----	
2508	ISO17234-1	37.90		----	
2514		----		----	
2516		----		----	
2522	ISO17234-1	<5.0		----	
2532	ISO17234-1	n.d.		----	
2553	ISO17234-1	n.d.		----	

lab	method	value	mark	z(targ)	remarks
2563	ISO17234-1	18.2		----	
2565		----		----	
2569		<5		----	
2572	ISO17234-1	<5.0		----	
2590	ISO17234-1	18.02		----	
2592	ISO17234-1	12.5		----	
2601	ISO17234-1	<2		----	
2612	ISO17234-1	17		----	
2624	ISO17234-1	n.d.		----	
2629		----		----	
2639	GB/T 19942	27.74		----	
2643	ISO17234-1	<5		----	
2656	ISO17234-1	<30		----	
2674	ISO17234-1	n.d.		----	
2695	ISO17234-1	21.96		----	
2713		----		----	
2730	EN14362-1	5.43		----	
2738	In house	<15		----	
3116		----		----	
3117		----		----	
3118	ISO17234-1	n.d.		----	
3146	DIN/EN17234-1	<10		----	
3150		----		----	
3153	ISO17234-1	<5		----	
3154		----		----	
3167		----		----	
3172	ISO17234-1	<5.00		----	
3176		----		----	
3191	ISO17234-1	<10		----	
3197	ISO17234-1	n.d.		----	
3199	ISO17234-1	<10		----	
3210		----		----	
3214	ISO17234-1	n.d.		----	
3216	ISO17234-1	n.d.		----	
3220	ISO17234-1	55.83	R(0.01)	----	
3228	ISO17234-1	n.d.		----	
3237	In house	25.31		----	
3248		----		----	
					<u>Compare to iis00A01</u>
normality		OK			OK
n		23 and 54 labs reported n.d. or <..			11 and 25 labs reported n.d. or < ..
outliers		1			2
mean (n)		(21.45)			(14.25)
st.dev. (n)		(7.844)			(8.364)
R(calc.)		(21.96)			(23.42)
R(ISO17234-1:2015)		(20.02)	R(LMBG82.02-3:97)		(13.33)



APPENDIX 2

Summary of other reported aromatic amines in sample #16520

lab	Aromatic amines
362	6.96 mg/kg 2,6-Xylidine (CASno. 87-62-7)
2403	22.90 mg/kg 2,4-Diaminotoluene (CASno. 95-80-7)
2481	30.4 mg/kg 2,4-Diaminotoluene (CASno. 95-80-7)
2504	1.41 mg/kg 4-Aminodiphenyl (CASno. 92-67-1)
2695	32.25 mg/kg 2,4-Diaminotoluene (CASno. 95-80-7)
2730	0.05 mg/kg 4-Aminodiphenyl (CASno. 92-67-1)
2738	41.5 mg/kg o-Anisidine (CASno. 90-04-0)
3216	7.87 mg/kg 4-Aminodiphenyl (CASno. 92-67-1)

Summary of not detected aromatic amines in sample #16520

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)

DMB = 3,3'-Dimethoxybenzidine (CASno. 119-90-4)

DDDM = 3,3'-Dimethyl-4,4'-Diaminodiphenyl methane (CASno. 838-88-0)

Lab	4AD	4CoT	2NA	oAAT	ANT	4CA	DAA	DADM	DCB	DMB	DDDM
110	----	----	----	----	----	----	----	----	----	----	----
213	----	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----	----
348	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
362	----	----	----	----	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----	----	----	----	----
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.
1911	----	----	----	----	----	----	----	----	----	----	----
2102	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2115	----	----	----	----	----	----	----	----	----	----	----
2129	<4	----	----	----	----	----	----	----	----	----	----
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2146	----	----	----	----	----	----	----	----	----	----	----
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2166	----	----	----	----	----	----	----	----	----	----	----
2170	----	----	----	----	----	----	----	----	----	----	----
2184	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2212	<5	C <5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2232	----	----	----	----	----	----	----	----	----	----	----
2246	----	----	----	----	----	----	----	----	----	----	----
2247	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2255	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2256	----	----	----	----	----	----	----	----	----	----	----
2272	----	----	----	----	----	----	----	----	----	----	----
2283	----	----	----	----	----	----	----	----	----	----	----
2290	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2291	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2296	----	----	----	----	----	----	----	----	----	----	----
2300	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
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	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2352	----	----	----	----	----	----	----	----	----	----	----
2357	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2364	----	----	----	----	----	----	----	----	----	----	----
2365	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2366	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5

2367	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2368	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2369	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2373	----	----	----	----	----	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----	----	----	----	----	----
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	----	----	----	----	----	----	----	----	----	----	----	----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2389	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2390	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2403	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2426	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2432	----	----	----	----	----	----	----	----	----	----	----	----
2442	----	----	----	----	----	----	----	----	----	----	----	----
2449	----	----	----	----	----	----	----	----	----	----	----	----
2459	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2476	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2481	<5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2482	----	----	----	----	----	----	----	----	----	----	----	----
2488	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2489	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2492	----	----	----	----	----	----	----	----	----	----	----	----
2495	----	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2497	----	----	----	----	----	----	----	----	----	----	----	----
2499	----	----	----	----	----	----	----	----	----	----	----	----
2504	1.41	----	----	----	----	----	----	----	----	----	----	----
2508	----	----	----	----	----	----	----	----	----	----	----	----
2514	----	----	----	----	----	----	----	----	----	----	----	----
2516	----	----	----	----	----	----	----	----	----	----	----	----
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
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.
2553	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2563	----	----	----	----	----	----	----	----	----	----	----	----
2565	----	----	----	----	----	----	----	----	----	----	----	----
2569	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2572	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2590	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
2592	----	----	----	----	----	----	----	----	----	----	----	----
2601	<0.5	<0.5	<0.5	----	----	<0.5	<2	<0.5	<0.5	<1	<0.5	<0.5
2612	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2624	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2629	----	----	----	----	----	----	----	----	----	----	----	----
2639	----	----	----	----	----	----	----	----	----	----	----	----
2643	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2656	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
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	----	----	----	----	----	----	----	----	----	----	----	----
2713	----	----	----	----	----	----	----	----	----	----	----	----
2730	0.05	0	0	0	0	0	0	0	0	0	0	0
2738	<15	<15	<15	----	<15	<15	----	<15	<15	<15	<15	<15
3116	----	----	----	----	----	----	----	----	----	----	----	----
3117	----	----	----	----	----	----	----	----	----	----	----	----
3118	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3146	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3150	----	----	----	----	----	----	----	----	----	----	----	----
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	----	----	----	----	----	----	----	----	----	----	----	----
3167	----	----	----	----	----	----	----	----	----	----	----	----
3172	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
3176	----	----	----	----	----	----	----	----	----	----	----	----
3191	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3197	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3199	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3210	----	----	----	----	----	----	----	----	----	----	----	----
3214	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3216	7.87	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3220	----	----	----	----	----	----	----	----	----	----	----	----
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3237	----	----	----	----	----	----	----	----	----	----	----	----
3248	----	----	----	----	----	----	----	----	----	----	----	----

Lab 2212 first reported 5.5 mg/kg for 4-Aminodiphenyl

Summary of not detected aromatic amines in sample #16520, continued

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

Lab	pC	DDM	DDE	DDS	24DAT	TMA	oA	4AAT	24X	26X
110	----	----	----	----	----	----	----	----	----	----
213	----	----	----	----	----	----	----	----	----	----
230	----	----	----	----	----	----	----	----	----	----
348	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
362	----	----	----	----	----	----	----	----	----	6.96
551	----	----	----	----	----	----	----	----	----	----
623	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.
1911	----	----	----	----	----	----	----	----	----	----
2102	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2115	----	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	<4	----
2132	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2146	----	----	----	----	----	----	----	----	----	----
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2166	----	----	----	----	----	----	----	----	----	----
2170	----	----	----	----	----	----	----	----	----	----
2184	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2212	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2232	----	----	----	----	----	----	----	----	----	----
2246	----	----	----	----	----	----	----	----	----	----
2247	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2255	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2256	----	----	----	----	----	----	----	----	----	----
2272	----	----	----	----	----	----	----	----	----	----
2283	----	----	----	----	----	----	----	----	----	----
2290	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2291	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2296	----	----	----	----	----	----	----	----	----	----
2300	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2310	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.
2330	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2347	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2352	----	----	----	----	----	----	----	----	----	----
2357	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2358	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
2366	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2367	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2368	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2369	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2373	----	----	----	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----	----	----	----
2379	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2380	----	----	----	----	----	----	----	----	----	----
2381	----	----	----	----	----	----	----	----	----	----
2386	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2389	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2390	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2403	n.d.	n.d.	n.d.	n.d.	22.90	n.d.	n.d.	n.d.	n.d.	n.d.
2426	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2432	----	----	----	----	----	----	----	----	----	----
2442	----	----	----	----	----	----	----	----	----	----
2449	----	----	----	----	----	----	----	----	----	----
2459	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2472	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2476	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2481	n.d.	n.d.	n.d.	n.d.	30.4	n.d.	n.d.	n.d.	----	----
2482	----	----	----	----	----	----	----	----	----	----

2488	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2489	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2492	----	----	----	----	----	----	----	----	----	----
2495	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2497	----	----	----	----	----	----	----	----	----	----
2499	----	----	----	----	----	----	----	----	----	----
2504	----	----	----	----	----	----	----	----	----	----
2508	----	----	----	----	----	----	----	----	----	----
2514	----	----	----	----	----	----	----	----	----	----
2516	----	----	----	----	----	----	----	----	----	----
2522	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2532	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2553	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2563	----	----	----	----	----	----	----	----	----	----
2565	----	----	----	----	----	----	----	----	----	----
2569	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2572	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
2590	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
2592	----	----	----	----	----	----	----	----	----	----
2601	<2	<0.5	<0.5	<1	<2	<1	<2	----	----	----
2612	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2624	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	----	n.d.	n.d.
2629	----	----	----	----	----	----	----	----	----	----
2639	----	----	----	----	----	----	----	----	----	----
2643	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2656	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2695	----	----	----	----	32.25	----	----	----	----	----
2713	----	----	----	----	----	----	----	----	----	----
2730	0	0	0	0	0	0	0	0	0	0
2738	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	41.5	n.d.	----	n.d.
3116	----	----	----	----	----	----	----	----	----	----
3117	----	----	----	----	----	----	----	----	----	----
3118	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3146	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3150	----	----	----	----	----	----	----	----	----	----
3153	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
3154	----	----	----	----	----	----	----	----	----	----
3167	----	----	----	----	----	----	----	----	----	----
3172	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
3176	----	----	----	----	----	----	----	----	----	----
3191	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3197	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3199	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
3210	----	----	----	----	----	----	----	----	----	----
3214	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3216	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.	n.d.	n.d.
3220	----	----	----	----	----	----	----	----	----	----
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3237	----	----	----	----	----	----	----	----	----	----
3248	----	----	----	----	----	----	----	----	----	----

APPENDIX 3**Analytical details**

Lab	Analysis method	Degrease with solvent	MTBE/MeOH extraction solution evaporated	Evaporation at T (°C) and took (min)	Time start reduction - start analysis (hrs)
110	ISO17234-1	Yes, n-Hexane	To complete dryness	40°C, < 10 min	< 2 h
213	EN17234-1	Yes, n-Hexane	No evaporation step was performed	N.A.	3.5 h
230	ISO17234-1:2010	Yes, n-Hexane	To complete dryness	rT, 15 min	3 h
348	in house	Yes, n-Hexane	No evaporation is required acc. to method	N.A.	3 h
362	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	50°C, 10 min	16 h
551	ISO17234-1	Yes, n-Hexane	To a quantity of 0.2 ml	---	---
623	ISO17234-1	Yes, n-Hexane	To almost dry	40°C, 15 min	24 h
840	ISO17234-1	Yes, n-Hexane	To complete dryness	50°C, 15 min	2 h
1911	PN-EN ISO17234-1:2015-07	Yes, n-Hexane	To a quantity of 2 ml	40°C, 15 min	2.5 h
2102	in house	Yes, n-Hexane	No evaporation step was performed	N.A.	1.5 h
2115	UNI EN ISO17234-1:2015	No, skipped this	To a small quantity (not further specified)	50°C, 3 min	5 h
2129	ISO17234-1	Yes, n-Hexane	To a certain quantity (not further specified)	40°C, 20 min	< 6 h
2132	ISO17234-1:2010	Yes, n-Hexane	To a quantity of 1 ml	49°C, 6 min	1 h
2146	EN ISO17234-1	Yes, n-Hexane	To a quantity of 1 ml	37°C, < 20 min	5 h
2165	ISO17234-1	Yes, n-Hexane	To a quantity of 0.5 ml	< 50°C, 15 min	< 12 h
2166	EN14362-1	No, skipped this	To complete dryness	60°C, 90 min	4 h
2170	ISO17234-1:2010	Yes, n-Hexane	To a small quantity (not further specified)	35°C, 10 min	2 h
2184	ISO17234-1	Yes, n-Hexane	To a quantity of 0.5 ml	< 50°C, 15 min	< 12 h
2212	ISO17234-1:2015	Yes, n-Hexane	To a quantity of 1 ml	50°C, 10 min	4 h
2232	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 2 min	3 h
2246	ISO17234-1:2010	Yes, n-Hexane	To a quantity less than 1 ml	49°C, 6 min	1 h
2247	ISO17234-1, 2	Yes, n-Hexane	To complete dryness	40°C, 10 min	2.5 h
2255	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	45°C, < 8 min	3.5 h
2256	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	40°C	8 h
2272	ISO17234-1:2015	Yes, n-Hexane	To a certain quantity (not further specified)	40°C, 10 min	< 5 h
2283	ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 6 min	4 h
2290	ISO17234-1:2011	Yes, n-Hexane	To complete dryness	60°C, 60 min	7 h
2291	ISO17234-1	Yes, n-Hexane	To a quantity less than 1 ml	42°C, 15 min	3 h
2296	in house	Yes, n-Hexane	To a small quantity (not further specified)	46°C, 60 min	24 h
2300	64LFB82.02-3(V):2004	Yes, n-Hexane	liquid-liquid extraction with MTBE	N.A.	0.5 h
2310	ISO17234-1	Yes, n-Hexane	To a quantity less than 1 ml	45°C, < 5 min	< 2 h
2311	ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	30°C, 5 min	2 h
2330	ISO17234-1:2010	No, skipped this	To complete dryness	20 min	1.5 h
2347	ISO17234	Yes, n-Hexane	To complete dryness	40°C, 20 min	72 h
2352	ISO17234-1:2010	Yes, n-Hexane	To a quantity less than 1 ml	< 50°C, < 5 min	< 1.5 h
2357	ISO17234-1:2010	Yes, n-Hexane	To a small quantity (not further specified)	35°C, 10 min	< 1.5 h
2358	ISO17234-1	Yes, n-Hexane	To a quantity of 0.5 ml	23°C, 15 min	0.75 h
2364	ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	45°C, 4 min	2.5 h
2365	ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 20 min	0.5 h
2366	ISO17234-1:2010	Yes, n-Hexane	To a quantity of 1 ml	45°C, 15 min	< 8 h
2367	ISO17234-1:2015	Yes, n-Hexane	To a certain quantity (not further specified)	40°C, 5 min	1.5 h
2368	ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	45°C, 5 min	1.5 h
2369	ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	40°C, < 20 min	2.5 h
2370	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 5 min	3 h
2373	ISO17234-1:2015	Yes, n-Hexane	To a quantity less than 0.5 ml	45°C, 5 min	< 2 h
2375	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	---	< 2.5 h
2379	ISO17234-1:2015	Yes, n-Hexane	To complete dryness	40°C, 5 min	72 h
2380	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	< 60°C, < 5 min	5 h
2381	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	< 60°C, < 5 min	5 h
2386	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	30°C, < 20 min	< 6 h
2389	ISO17234-1	Yes, n-Hexane	---	---	3 h
2390	ISO17234-1:2015	Yes, n-Hexane	To a quantity of 0.2 ml	45°C, 3 min	1.5 h
2403	ISO17234-1:2015	Yes, n-Hexane	To complete dryness	40°C, 10 min	1 h
2426	ISO17234-1:2015	Yes, n-Hexane	To a quantity of 0.5 ml	40°C, 12 min	3 h
2432	ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	50°C, 6 min	1.5 h
2442	in house	Yes, n-Hexane	To a small quantity (not further specified)	45°C, 5 min	2 h
2449	ISO17234-1	Yes, n-Hexane	To a quantity of 1 ml	40°C	1.5 h
2459	ISO17234-1:2015	Yes, n-Hexane	To a quantity of 1 ml	45°C, 5 min	5 h
2472	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	38°C, 10 min	2 h
2476	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 10 min	3 h
2481	in house	Yes, n-Heptane	To a quantity of 2-3 ml	34°C, 8 min	7 h
2482	DIN EN ISO17234-1:2015-07	Yes, n-Hexane	To a quantity of 1 ml	< 50°C, 20 min	< 6 h
2488	ISO17234-1	---	To a quantity of 2 ml	40°C, 5 min	1.5 h
2489	ISO/TS 17234-1:2012	Yes, n-Hexane	To a quantity of 1 ml	45°C, < 4 min	2 h
2492	in house	Yes, n-Hexane	To a small quantity (not further specified)	35°C, 30 min	< 1 h
2495	ISO17234-1	Yes, n-Hexane	To a quantity of 1 ml	40°C, 7 min	4 h
2497	ISO17234-1	No, skipped this	To a small quantity (not further specified)	50°C, 15 min	8 h
2499	GB/T 19942:2005	Yes, n-Hexane	To a quantity of 0.5 ml	40°C, 5 min	8 h
2504	ISO17234-1:2010	Yes, n-Hexane	To a certain quantity (not further specified)	40°C, < 3 min	1 h

Lab	Analysis method	Degrease with solvent	MTBE/MeOH extraction solution evaporated	Evaporation at T (°C) and took (min)	Time start reduction - start analysis (hrs)
2508	ISO17234-1	Yes, n-Hexane	To a quantity of 1 ml	50°C, 30 min	25 h
2514	ISO17234-1	Yes, n-Hexane	To a quantity of 1 ml	50°C, < 6 min	0.5 h
2516	EN14362-1	Yes, n-Hexane	To a small quantity (not further specified)	35°C, 7 min	4 h
2522	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 10 min	17 h
2532	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 10 min	3 h
2553	ISO17234-1/ CPSD-AN-00017	Yes, n-Hexane	To complete dryness	40°C, < 20 min	1.5 h
2563	DIN ISO17234-1	Yes, n-Hexane	To a quantity of 3 ml	---	5 h
2565	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	35°C, 15 min	< 5 h
2569	ISO17234-1	Yes, n-Hexane	To a certain quantity (not further specified)	40°C, 10 min	3 h
2572	ISO17234-1:2011	Yes, n-Hexane	To complete dryness	60°C, 60 min	7 h
2590	ISO17234-1	Yes, n-Hexane	To a quantity of 1 ml	40°C, 10 min	< 2.5 h
2592	UNI EN ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	< 50°C, 15 min	5 h
2601	DIN EN ISO17234-1:2010	Yes, n-Hexane	To complete dryness	40°C, 10 min	8 h
2612	ISO17234-1	Yes, n-Hexane	To complete dryness	70°C, 30 min	4 h
2624	ISO17234-1	Yes, n-Hexane	To a quantity of 1 ml	38°C, 20 min	24 h
2629	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 10 min	2 h
2639	GB/T 19942:2005	Yes, n-Hexane	To a quantity of 1 ml	40°C, 10 min	< 3 h
2643	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	N.A.	< 3 h
2656	NF EN ISO17234-1, 2	Yes, n-Hexane	To complete dryness	40°C, 10 min	6 h
2674	ISO17234-1:2010	Yes, n-Hexane	To a quantity of 0.5 ml	45°C, 10 min	< 12 h
2695	UNI EN ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 5 min	2 h
2713	BS EN ISO17234-1	Yes, n-Hexane	To a quantity of 1 ml	40°C, < 7 min	5 h
2730	NF EN 14362-1:2012+AnnexE	Yes, n-Hexane	To complete dryness	60°C, 10 min	2 h
2738	in house	Yes, Citrate buffer	To a quantity of 1-2 drops	45°C, < 5 min	< 5 h
3116	ISO17234-1	Yes, n-Hexane	To a quantity of 1-2 ml	40°C, 15 min	4 h
3117	ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	34°C, < 5 min	< 1.5 h
3118	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	35°C	2 h
3146	DIN EN17234-1	Yes, n-Hexane	To a quantity of 3 ml	50°C, 45 min	---
3150	ISO17234-1	Yes, n-Hexane	To a certain quantity (not further specified)	45°C, 7 min	1 h
3153	ISO17234-1	Yes, n-Hexane	To a quantity of 2-3 ml	35°C, 7 min	7 h
3154	ISO17234	No, skipped this	To complete dryness	50°C, 5 min	2 h
3167	ISO17234-1:2015	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 3 min	4 h
3172	ISO17234-1	Yes, n-Hexane	To a certain quantity (not further specified)	45°C, 40 min	2 h
3176	ISO17234-1	Yes, n-Hexane	To a quantity of 1 ml	50°C, 30 min	2 h
3191	ISO17234-1:2010	Yes, n-Hexane	To complete dryness	45°C, 5 min	24 h
3197	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 10 min	3 h
3199	ISO17234-1	Yes, n-Hexane	To complete dryness	40°C, 7 min	< 0.75 h
3210	in house	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 10 min	2 h
3214	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	35°C, 10 min	8 h
3216	UNI EN ISO17234-1:2010	Yes, n-Hexane	To complete dryness	40°C, 10 min	7 days
3220	ISO17234-1	Yes, n-Hexane	To complete dryness	30°C, 10 min	4 h
3228	ISO17234-1	Yes, n-Hexane	To a quantity of 0.5 ml	< 50°C, 15 min	12 h
3237	EN14362-1	Yes, n-Hexane	To a small quantity (not further specified)	45°C, 8 min	2 h
3248	ISO17234-1	Yes, n-Hexane	To a small quantity (not further specified)	40°C, 10 min	3 h

lab	Remarks on additional questions
2166	Polyvap to 1 ml with cooled concentration zone, further reduced to complete dryness under Argon
2247	Evaporation has been done under gentle flow of nitrogen
2482	Evaporated to 1 ml, then filled up to 2 ml
2656	Evaporation at 450 mbar of MTBE using a rotavapor to 1-2mL then to dryness using a flow of inert gas (Nitrogen)
3153	Make up residue with MTBF in 10ml volumetric flask
3199	Evaporated to approximately 1mL. Remainder of solvent removed by very gentle flow of Nitrogen.

APPENDIX 4

Number of participants per country

5 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in CAMBODIA
1 lab in EGYPT
1 lab in FINLAND
4 labs in FRANCE
11 labs in GERMANY
9 labs in HONG KONG
8 labs in INDIA
2 labs in INDONESIA
9 labs in ITALY
2 labs in JAPAN
1 lab in KOREA
1 lab in MAURITIUS
1 lab in MOROCCO
25 labs in P.R. of CHINA
5 labs in PAKISTAN
1 lab in POLAND
2 labs in SINGAPORE
2 labs in SPAIN
1 lab in SRI LANKA
1 lab in SWITZERLAND
2 labs in TAIWAN R.O.C.
2 labs in THAILAND
1 lab in THE NETHERLANDS
6 labs in TURKEY
2 labs in U.S.A.
1 lab in UNITED KINGDOM
4 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' outlier test
R(0.05)	= straggler in Rosner' outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from calculations
n.e.	= not evaluated
n.a.	= not applicable
n.d.	= not detected

Literature:

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