Results of Proficiency Test
AZO dyes in leather
March 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

The Institute for Interlaboratory Studies (iis) organizes every year a proficiency test for banned AZO dyes in leather since 1997, with an exception in 2009. During the annual proficiency testing program 2014/2015, it was decided to continue the round robin for the analysis of banned AZO dyes in leather. In this interlaboratory study, 158 laboratories in 31 different countries have participated (see appendix 4). In this report, the results of the 2015 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse 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. This leather sample was especially dyed with Acid Red 114 to find 3,3'-Dimethylbenzidine by an Italian company. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited third party laboratory. Participants were requested to report rounded and unrounded test results. These unrounded test results were preferably used for statistical evaluation. The participants were asked to report the analytical results using the indicated units on the report form.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accreditation scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 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 http://www.iisnl.com.

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 pink coloured leather sample (coloured with Acid Red 114), positive on AZO dyes, was made available by an Italian company. After cutting it into small pieces of <0.1g, the material was mixed thoroughly. In total 160 sub samples were prepared of 3 gram leather each and subsequently labelled #15022. Eight stratified randomly selected samples were tested using ISO17234-1 to check the homogeneity of the batch. See the following table for the test results.

	3,3'-Dimethylbenzidine in mg/kg
sample #15022-1	121.2
sample #15022-2	135.2
sample #15022-3	119.3
sample #15022-4	127.5
sample #15022-5	122.7
sample #15022-6	131.8
sample #15022-7	123.6
sample #15022-8	126.3

Table 1: homogeneity test results of subsamples #15022

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

	3,3'-Dimethylbenzidine in mg/kg
r (observed)	15.2
reference method	ISO17234-1:2010
0.3 x R (reference method)	21.3

Table 2: evaluation of the repeatability of subsamples #15022

The repeatability of the results of homogeneity test for 3,3'-Dimethylbenzidine was in agreement with 0.3 times the estimated reproducibility mentioned in the reference method ISO17234-1:2010.
Therefore, homogeneity of the subsamples was assumed.

One sample with approx. 3.0 grams (labelled #15022) testing material was sent to each of the participating laboratories on March 4, 2015.

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 results reported, a detailed report form, on which the requested amines and the units were pre-printed, was sent together with each sample. Also a letter of instructions was sent along.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated in the appendices of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not yet reported. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test, see lit.5) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected data are placed under 'Remarks' in the result tables in appendix 1. A list of abbreviations used in the tables can be found in appendix 5.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organization, 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 results. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 5, no.15). Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05). 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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 5; nos.13 and 14). Also a normal Gauss curve was projected over the Kernel Density Graph.

3.3 Z-SCORES

To evaluate the performance of the individual participating laboratories the z-scores were calculated. In order to be able to have an objective evaluation of the performance of the individual participants, it was decided to evaluate this performance against the literature requirements. Therefore the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8.
The $z_{(target)}$-scores were calculated according to:

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

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

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 the fit-for-useness of the reported test result.

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. Thirty-two participants reported test results after the deadline and six participants did not report any test results. Finally, 152 participants did report 147 numerical test results for the aromatic amine present (3,3'-Dimethylbenzidine), while another 15 test results were reported for other aromatic amines. Observed were 3 outlying test results, which is 2.0% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.
The data set of 3,3'-Dimethylbenzidine did proof to have a normal Gaussian distribution.

4.1 EVALUATION PER COMPONENT

In this section, the results are discussed per sample. All statistical results reported for 3,3'-Dimethylbenzidine are summarised in appendix 1 and the reported test results of all other aromatic amines are listed in appendix 2.

<u>3,3'-Dimethylbenzidine:</u> The determination of this aromatic amine at a concentration level of 82 mg/kg was somewhat problematic. Three statistical outliers were observed. Three other participants reported "< 5 mg/kg", which is a false negative test result and one participant did not report a test result for 3,3'-Dimethylbenzidine but for six other aromatic amines.
The test results reported by the participants vary from <5 – 131.5 mg/kg. The observed reproducibility after rejection of the statistical outliers is almost in agreement with the reproducibility requirement of 56.4% estimated from the standard test method ISO 17234-1:2010.

<u>General:</u> Seven participants reported the presence of other aromatic amines at various concentration levels, see appendix 2.
All laboratories, except five (5) laboratories, would have rejected this leather sample for containing too much of a banned aromatic amine (according to OEKO-TEX Std 100 ed. 04/2013 of 20 mg/kg).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard methods (references 1 - 4) 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 ISO17234-1:2010 (equal to the reproducibility from LMBG 82.02.3:97) are presented in the next table.

Parameter	unit	n	Average	2.8 * sd	R(target)
3,3'-Dimethylbenzidine	mg/kg	145	81.8	54.0	46.1

Table 3: reproducibility of the aromatic amine in leather sample #15022

Without further statistical calculations, it can be concluded that the group of participating laboratories has some problems with the analysis of 3,3'-Dimethylbenzidine in leather. See also the discussion in paragraphs 4.1 and 6.

5 COMPARISON WITH PREVIOUS INTERLABORATORY STUDIES

The observed spread in the test results for the aromatic amine in the 2014 PT is in good agreement in comparison with the spread of the aromatic amine as observed in the previous PTs, see below table.

Parameter	*March 2015*	*March 2014*	*March 2013*	*March 2012*	*March 2011*	*March 2010*	*March 2008*	*March 2007*	*ISO17234-1: 2010*
4-Aminodiphenyl	n.e.	n.e.	n.e	25%	n.e.	n.e.	n.e.	n.e.	Unknown
Benzidine	n.e.	20%	28%	20%	n.e.	n.e.	38%	45%	12 – 25%
3,3'-Dimethylbenzidine	24%	n.e.	n.e	n.e.	n.e.	n.e.	n.d.	45%	17 – 24%
o-Toluidine	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	50%	n.e.	30– 37%
2,4-Xylidine	n.e.	n.e.	36%	n.e.	19%	16%	n.e.	n.e.	n.e.

Table 4: development of the uncertainties over the years

6 DISCUSSION

From the reported test methods it appeared that almost all participants treated the leather samples according identical test methods: ISO17234-1 or LFBG 82.02.3.
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 spread in this interlaboratory study is not caused by just one critical point in the analysis.
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

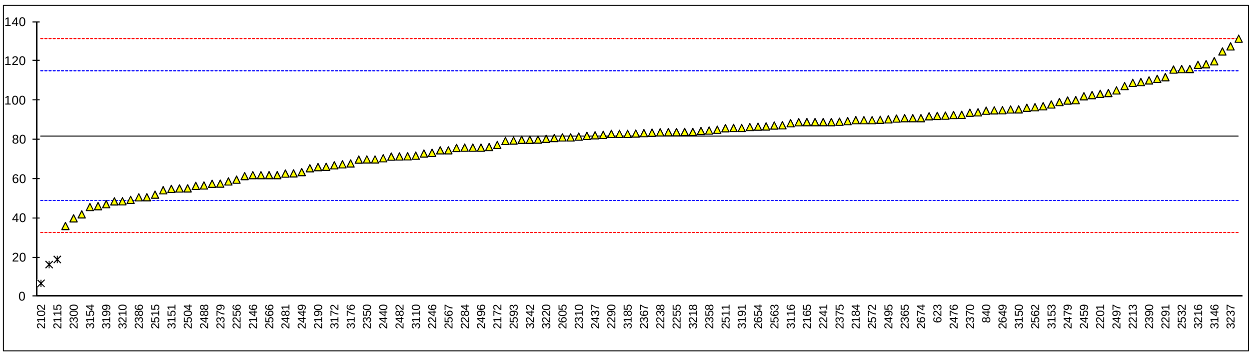
APPENDIX 1

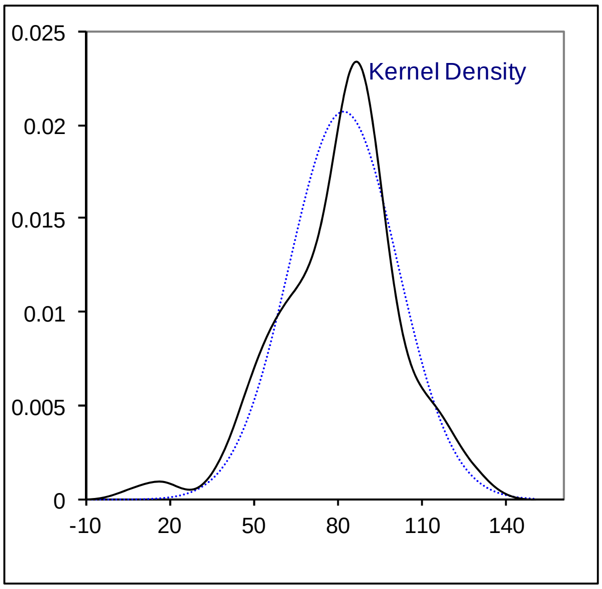
Determination of 3,3’-Dimethylbenzidine (CASno.119-93-7) in sample #15022; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO17234-1	57.6		-1.47	
213		-----		-----	
230	ISO17234-1	76.3		-0.34	
348	in house	107.37		1.55	
362	in house	<5.0		<-4.66	False negative test result?
551	ISO17234-1	111.01		1.77	
622	ISO17234-1	<5	C	<-4.66	First reported 13, False negative test result?
623	ISO17234-1	92.15		0.63	
840	ISO17234-1	94.8		0.79	
2102		6.87	R(0.05)	-4.55	
2115	ISO17234-1	19.03	R(0.05)	-3.81	
2121	ISO17234-1	67.51		-0.87	
2129	ISO17234-1	125		2.62	
2132	ISO17234-1	80.9		-0.06	
2137		-----		-----	
2139	ISO17234-1	56.5		-1.54	
2146	ISO17234-1	62.0		-1.20	
2165	ISO17234-1	89		0.44	
2166	EN14362	36.1		-2.77	
2169		-----		-----	
2170	ISO17234-1	86.8		0.30	
2172	ISO17234-1	77.4		-0.27	
2173	ISO17234-1	118.5		2.23	
2184	ISO17234-1	90		0.50	
2190	ISO17234-1	66.1		-0.95	
2201	ISO17234-1	103.4		1.31	
2213	ISO17234-1	109	C	1.65	First reported 149.2
2215	ISO17234-1	90.2		0.51	
2232	ISO17234-1	48.65		-2.01	
2238	ISO17234-1	83.8		0.12	
2241	ISO17234-1	89.0		0.44	
2246	ISO17234-1	73.39		-0.51	
2247	ISO17234-1	95.44	C	0.83	First reported 146.37
2255	ISO17234-1	83.9		0.13	
2256	ISO17234-1	59.7		-1.34	
2284	ISO17234-1	76		-0.35	
2289	ISO17234-1	109.4		1.67	
2290	ISO17234-1	83.0		0.07	
2291	ISO17234-1	111.9		1.82	
2295	ISO17234-1	70		-0.72	
2296	ISO17234-1	71.43		-0.63	
2297	ISO17234-1	92.7		0.66	
2300	64LFGB82.02.3	40.0		-2.54	
2301	ISO17234-1	58.8		-1.40	
2310	ISO17234-1	81.6		-0.01	
2311	ISO17234-1	86.5		0.28	
2314	ISO17234-1	79.4		-0.15	
2330	ISO17234-1	83.62		0.11	
2347	ISO17234-1	94		0.74	
2350	ISO17234-1	70		-0.72	
2352	ISO17234-1	90		0.50	
2357	ISO17234-1	87.4		0.34	
2358	ISO17234-1	84.7		0.17	
2364	ISO17234-1	86		0.25	
2365	ISO17234-1	91		0.56	
2366	ISO17234-1	91		0.56	
2367	ISO17234-1	83.4		0.10	
2368	ISO17234-1	80.0		-0.11	
2369	ISO17234-1	89		0.44	
2370	ISO17234-1	93.8		0.73	
2373	ISO17234-1	83.9		0.13	
2375	ISO17234-1	89.2		0.45	
2379	ISO17234-1	57.671		-1.47	
2380	ISO17234-1	90.8		0.54	
2386	EN14362	50.7		-1.89	
2389	ISO17234-1	103.80		1.33	
2390	ISO17234-1	110.265		1.73	
2403	ISO17234-1	84.5		0.16	
2410	ISO17234-1	76		-0.35	
2413		-----		-----	reported 6 other amines, see appendix 2
2415	ISO17234-1	83.2		0.08	
2426	ISO17234-1	97.1015		0.93	
2429	ISO17234-1	100.20		1.11	

2432	ISO17234-1	115.75	C	2.06	First reported 168.8
2437	ISO17234-1	82.3		0.03	
2440	ISO17234-1	70.6		-0.68	
2442	in house	69.91		-0.72	
2449	ISO17234-1	63.54		-1.11	
2452	ISO17234-1	62.89		-1.15	
2455	ISO17234-1	16.44	C,R(0.05)	-3.97	First reported 10
2459	in house	102.20		1.24	
2467	in house	116.05	C	2.08	First reported 175.60
2472	ISO17234-1	73		-0.54	
2476	ISO17234-1	92.6		0.65	
2479	ISO17234-1	100.0		1.10	
2481	ISO17234-1	62.8		-1.15	
2482	ISO17234-1	71.512		-0.63	
2488	ISO17234-1	56.73		-1.52	
2489	ISO17234-1	131.50		3.01	
2492	INHOUSE	49.4		-1.97	
2495	ISO17234-1	90.4		0.52	
2496	ISO17234-1	76.0		-0.35	
2497	ISO17234-1	105.17		1.42	
2499		-----		-----	
2504	ISO17234-1	55.26		-1.61	
2511	ISO17234-1	85.9		0.25	
2514	ISO17234-1	85.10		0.20	
2515	ISO17234-1	52.06		-1.81	
2516	ISO17234-1	65.5		-0.99	
2532	ISO17234-1	116		2.07	
2534	ISO17234-1	99.2		1.05	
2536	ISO17234	62		-1.20	
2538	64LFGB82.02.3	82.5		0.04	
2553	ISO17234-1	66.25		-0.94	
2560	ISO17234-1	<5		<-4.66	False negative test result?
2562	GB/T19942	96.6		0.90	
2563	ISO17234-1	87.28		0.33	
2565	ISO17234-1	61.5		-1.23	
2566	ISO17234-1	62		-1.20	
2567	ISO17234-1	74.64		-0.44	
2572	ISO17234-1	90		0.50	
2590	ISO17234-1	42.03		-2.41	
2592	ISO17234-1	50.76		-1.88	
2593	ISO17234-1	79.6		-0.13	
2605	ISO17234-1	81.2		-0.04	
2614	CPSD-AN-00017	62		-1.20	
2618	ISO17234-1	92.30		0.64	
2629	ISO17234-1	55.21		-1.61	
2639	GB/T19942	54.3		-1.67	
2643	ISO17234-1	84		0.13	
2649	ISO17234-1	95.07		0.80	
2654	ISO17234-1	86.70		0.30	
2656		-----		-----	
2658		-----		-----	
2668	ISO17234-1	81.2		-0.04	
2674	ISO17234-1	91		0.56	
2675	ISO17234-1	71.58		-0.62	
2677	ISO17234-1	46.21		-2.16	
3100	ISO17234-1	83		0.07	
3110	ISO17234-1	71.9		-0.60	
3116	ISO17234-1	88.44		0.40	
3117	ISO17234-1	82		0.01	
3118	ISO17234-1	75.82		-0.36	
3146	ISO17234-1	120		2.32	
3150	ISO17234-1	95.5		0.83	
3151	ISO17234-1	55.0		-1.63	
3153	ISO17234-1	98		0.98	
3154	DIN53316	45.75	C	-2.19	First reported 166.90
3167	ISO17234-1	95.0		0.80	
3172	ISO17234-1	67		-0.90	
3176	ISO17234-1	67.94		-0.84	
3185	ISO17234-1	83		0.07	
3190	ISO17234-1	88.96		0.43	
3191	ISO17234-1	86		0.25	
3197	ISO17234-1	96.3		0.88	
3199	CPSD-AN-00017	47.2		-2.10	
3210	ISO17234-1	48.7		-2.01	
3214	ISO17234-1	102.77		1.27	
3216	ISO17234-1	118.17		2.21	
3218	ISO17234-1	84		0.13	

3220	ISO17234-1	80.5		-0.08	
3222	ISO17234-1	74.63	C	-0.44	First reported 26.80
3225	ISO17234-1	80.1		-0.10	
3228	ISO17234-1	92		0.62	
3237	in house	127.5795		2.78	
3242	ISO17234-1	80		-0.11	
3246	ISO17234-1	89.5		0.47	
3248	ISO17234-1	89		0.44	
	normality	OK			
	n	145			
	outliers	2			
	mean (n)	81.825			
	st.dev. (n)	19.2791			
	R(calc.)	53.981			
	R(ISO17234-1:10)	46.149			





APPENDIX 2

Summary of other reported aromatic amines

lab	aromatic amines
2102	2.70 mg/kg Benzidine; 3.08 mg/kg 3.3'-Dimethoxybenzidine
2115	7.40 mg/kg 2,4-Diaminotoluene
2413	32.06 mg/kg 4-Chloro-o-toluidine; 6.10 mg/kg o-Aminoazotoluene; 32.08 mg/kg 2-Amino-4-nitrotoluene; 310.38 mg/kg 4,4'-Diaminodiphenylether; 9.73 mg/kg 2,4-Diaminotoluene; 5,050 mg/kg o-Anisidine
2497	1.73 mg/kg o-Toluidine
2534	9.5 mg/kg 4-Aminoazobenzene
2563	2.88 mg/kg o-Aminoazotoluene
2675	12.03 mg/kg p-Cresidine; 15.37 mg/kg o-Toluidine; 22.77 mg/kg 2,4-Xylidene

APPENDIX 3
Analytical details

Lab	Degreasing solvent	Main evaporation of solvent	evaporation temp.	Final evaporation of solvent
110	n-hexane	Vacuum Rotary Evaporator	45	weak flow of inert gas (N2)
213	--	--	--	--
230	n-hexane	Overnight in Fumehood	--	--
348	n-hexane	In oven for 1 hr	40	--
362	n-hexane	Decant and evaporate overnight	--	--
551	Ethylacetate	--	--	--
622	n-hexane	Vacuum Rotary Evaporator	50	--
623	n-hexane	weak flow of inert gas	--	--
840	n-hexane	weak flow of inert gas	--	--
2102	n-hexane	air drying 2 days	--	--
2115	n-hexane	Vacuum Rotary Evaporator	50	--
2121	n-hexane	speed extractor "buchi"	--	weak flow of inert gas (N2)
2129	n-hexane	weak flow of inert gas	--	--
2132	n-hexane	weak flow of inert gas	--	--
2137	--	--	--	--
2139	n-hexane	Vacuum Rotary Evaporator	50	weak flow of inert gas (N2)
2146	n-hexane	Vacuum Rotary Evaporator	35-36	weak flow of inert gas (N2)
2165	n-hexane	weak flow of inert gas	--	--
2166	--	Vacuum Rotary Evaporator	40	weak flow of inert gas (Ar)
2169	--	--	--	--
2170	n-hexane	Vacuum Rotary Evaporator	35	--
2172	n-hexane	Vacuum Rotary Evaporator	40	--
2173	n-hexane	weak flow of inert gas	46	--
2184	n-hexane	Vacuum Rotary Evaporator	45	--
2190	n-hexane	Vacuum Rotary Evaporator	50	--
2201	n-hexane	Vacuum Rotary Evaporator	40	weak flow of inert gas (N2)
2213	n-hexane	Vacuum Rotary Evaporator	50	weak flow of inert gas (N2)
2215	n-hexane	Vacuum Rotary Evaporator	40	--
2232	n-hexane	Vacuum Rotary Evaporator	40	--
2238	n-hexane	Vacuum Rotary Evaporator	40	--
2241	n-hexane	Vacuum Rotary Evaporator	40	--
2246	MTBE	Vacuum Rotary Evaporator	40	--
2247	n-hexane	Vacuum Rotary Evaporator	40	--
2255	n-hexane	Vacuum Rotary Evaporator	50	weak flow of inert gas (N2)
2256	n-hexane	Vacuum Rotary Evaporator	40	--
2284	MTBE	Vacuum Rotary Evaporator	45	--
2289	n-hexane	weak flow of inert gas (N2)	--	--
2290	n-hexane	Vacuum Rotary Evaporator	45	--
2291	n-hexane	Vacuum Rotary Evaporator	40	--
2295	n-hexane	Vacuum Rotary Evaporator	50	--
2296	n-hexane	Vacuum Rotary Evaporator	70	weak flow of inert gas (N2)
2297	n-hexane	Vacuum Rotary Evaporator	45	weak flow of inert gas (N2)
2300	n-hexane	weak flow of inert gas (N2)	--	--
2301	n-hexane	Vacuum Rotary Evaporator	40	--
2310	n-hexane	--	--	--
2311	n-hexane	--	--	--
2314	n-hexane	Vacuum Rotary Evaporator	50	--
2330	MTBE	Vacuum Rotary Evaporator	40	weak flow of inert gas (N2)
2347	n-hexane	Evaporated Overnight		--
2350	MTBE	Vacuum Rotary Evaporator	50	--
2352	n-hexane	Vacuum Rotary Evaporator	<50	--
2357	n-hexane	Vacuum Rotary Evaporator	70	--
2358	n-hexane	Vacuum Rotary Evaporator	30	--
2364	n-hexane	Vacuum Rotary Evaporator	45	--
2365	n-hexane	Vacuum Rotary Evaporator	40	--
2366	n-hexane	Vacuum Rotary Evaporator	45	--
2367	n-hexane	Vacuum Rotary Evaporator	40	--
2368	n-hexane	Vacuum Rotary Evaporator	45	weak flow of inert gas (N2)
2369	n-hexane	Vacuum Rotary Evaporator	40	--
2370	n-hexane	Vacuum Rotary Evaporator	40	--
2373	n-hexane	Vacuum Rotary Evaporator	45	weak flow of inert gas (N2)
2375	n-hexane	Fumehood	--	--
2379	n-hexane	Vacuum Rotary Evaporator	50	--
2380	n-hexane	Vacuum Rotary Evaporator	70	--
2386	n-hexane	Turbo Vaporisation	--	--
2389	n-hexane	screening method	--	--
2390	n-hexane	--	--	--
2403	MTBE	Vacuum Rotary Evaporator	40	--
2410	MTBE	Vacuum Rotary Evaporator	30	--
2413	n-hexane	Vacuum Rotary Evaporator	40	--
2415	n-hexane	Vacuum Rotary Evaporator	45	weak flow of inert gas (N2)
2426	n-hexane	weak flow of inert gas (N2)	--	--
2429	n-hexane	Vacuum Rotary Evaporator	40	--

2432	n-hexane	--	--	--
2437	n-hexane	Vacuum Rotary Evaporator	45	weak flow of inert gas (N2)
2440	n-hexane	Vacuum Rotary Evaporator	35	weak flow of inert gas (N2)
2442	n-hexane	--	--	--
2449	n-hexane	Vacuum Rotary Evaporator	50	weak flow of inert gas (N2)
2452	n-hexane	weak flow of inert gas (N2)	--	--
2455	n-hexane	Vacuum Rotary Evaporator	45	weak flow of inert gas (N2)
2459	n-hexane	Vacuum Rotary Evaporator	40	--
2467	n-hexane	Vacuum Rotary Evaporator	40	--
2472	n-hexane	Vacuum Rotary Evaporator	38	weak flow of inert gas (N2)
2476	n-hexane	Vacuum Rotary Evaporator	50	--
2479	n-hexane	Vacuum Rotary Evaporator	46	weak flow of inert gas (N2)
2481	n-hexane	weak flow of inert gas (N2)	--	--
2482	n-hexane	weak flow of inert gas (N2)	--	--
2488	n-hexane	Vacuum Rotary Evaporator	40	weak flow of inert gas (N2)
2489	n-hexane	Vacuum Rotary Evaporator	40	--
2492	n-hexane	--	--	--
2495	n-hexane	Overnight evaporation	--	--
2496	n-hexane	Vacuum Rotary Evaporator	40	--
2497	n-hexane	Vacuum Rotary Evaporator	40	--
2499	--	--	--	--
2504	--	--	--	--
2511	n-hexane	Vacuum Rotary Evaporator	35	--
2514	n-hexane	Vacuum Rotary Evaporator	45	--
2515	--	--	--	--
2516	--	Vacuum Rotary Evaporator	35	--
2532	n-hexane	Vacuum Rotary Evaporator	35	--
2534	MTBE	Vacuum Rotary Evaporator	30	--
2536	n-hexane	Vacuum Rotary Evaporator	70	weak flow of inert gas (N2)
2538	n-hexane	--	--	--
2553	n-hexane	weak flow of inert gas (N2)	--	--
2560	n-hexane	Vacuum Rotary Evaporator	40	--
2562	n-hexane	--	--	--
2563	n-hexane	Atmospheric pressure	40	--
2565	n-hexane	Vacuum Rotary Evaporator	35	--
2566	n-hexane	Vacuum Rotary Evaporator	40	--
2567	n-hexane	Vacuum Rotary Evaporator	70	--
2572	n-hexane	Vacuum Rotary Evaporator	45	--
2590	n-hexane	Vacuum Rotary Evaporator	50	weak flow of inert gas (N2)
2592	n-hexane	Vacuum Rotary Evaporator	42	--
2593	n-hexane	Vacuum Rotary Evaporator	40	weak flow of inert gas (N2)
2605	n-hexane	Vacuum Rotary Evaporator	35	weak flow of inert gas (N2)
2614	n-hexane	Vacuum Rotary Evaporator	45	--
2618	--	--	--	--
2629	n-hexane	Vacuum Rotary Evaporator	45	weak flow of inert gas (Ar)
2639	n-hexane	Vacuum Rotary Evaporator	40	--
2643	n-hexane	--	--	--
2649	n-hexane	Vacuum Rotary Evaporator	40	weak flow of inert gas (N2)
2654	n-hexane	Vacuum Rotary Evaporator	50	weak flow of inert gas (N2)
2656	--	--	--	--
2658	--	--	--	--
2668	n-hexane	weak flow of inert gas (N2)	--	--
2674	n-hexane	weak flow of inert gas (N2)	--	--
2675	n-hexane	solid phase extraction	--	--
2677	n-hexane	Overnight evaporation	--	weak flow of inert gas (N2)
3100	n-hexane	Vacuum Rotary Evaporator	35	--
3110	n-hexane	--	--	--
3116	n-hexane	Vacuum Rotary Evaporator	40	weak flow of inert gas (N2)
3117	MTBE	Vacuum Rotary Evaporator	40	--
3118	n-hexane	Vacuum Rotary Evaporator	40	--
3146	n-hexane	Vacuum Rotary Evaporator	45	--
3150	MTBE	Vacuum Rotary Evaporator	40	--
3151	MTBE	Turbo Vaporisation	40	--
3153	n-hexane	Vacuum Rotary Evaporator	35	--
3154	n-hexane	Vacuum Rotary Evaporator	50	weak flow of inert gas (N2)
3167	n-hexane	Vacuum Rotary Evaporator	35	--
3172	--	--	--	--
3176	n-hexane	weak flow of inert gas (N2)	--	--
3185	n-hexane	Vacuum Rotary Evaporator	35	weak flow of inert gas (N2)
3190	n-hexane	Vacuum Rotary Evaporator	40	--
3191	n-hexane	Vacuum Rotary Evaporator	50	--
3197	MTBE	Vacuum Rotary Evaporator	40	--
3199	n-hexane	Vacuum Rotary Evaporator	40	weak flow of inert gas (N2)
3210	n-hexane	weak flow of inert gas (N2)	--	--
3214	n-hexane	Vacuum Rotary Evaporator	70	--
3216	n-hexane	Vacuum Rotary Evaporator	40	weak flow of inert gas (N2)
3218	n-hexane	Vacuum Rotary Evaporator	40	--

3220	n-hexane	Vacuum Rotary Evaporator	40	--
3222	n-hexane	Vacuum Rotary Evaporator	46	weak flow of inert gas (N2)
3225	n-hexane	Vacuum Rotary Evaporator	48	weak flow of inert gas (N2)
3228	n-hexane	weak flow of inert gas (N2)	--	--
3237	n-hexane	filtration and oven	--	--
3242	n-hexane	Turbo Vaporisation	70	--
3246	MTBE	--	--	--
3248	n-hexane	Vacuum Rotary Evaporator	60	weak flow of inert gas (N2)

APPENDIX 4

Number of participants per country

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

APPENDIX 5

Abbreviations:

C	= final result after checking of first reported suspect 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
n.e.	= not evaluated
n.d.	= not detected

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