# Results of Proficiency Test Colorants (Banned Dyes) in Textile March 2020

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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#### 1 Introduction

Colored fabrics, when in contact with human skin, may cause Allergic Contact Dermatitis. Several dyestuffs are therefore classified as allergenic. Textiles are not allowed to contain more than 20 mg/kg of the dyes listed in the latest Oeko-tex Standard 100 edition 03/2019 and bluesign® 07/2016. The Oeko-tex Standard 100 also lists many carcinogenic dyes and other banned dyestuffs. With every update of the standard new banned dyes are added. The ban on dyes has become a widely publicized issue in the textile industry. Dyestuff manufacturers, processors and exporters are careful in the selection of the dyes. However, several dyestuffs that are skin sensitizers may still be in use for dyeing polyester and nylon.

Since 2005 the Institute for Interlaboratory Studies (iis) organizes a proficiency test for <u>allergenic</u> dyes in textile every year. In 2016 the scope was extended with <u>carcinogenic</u> and <u>other banned</u> dyes. It was decided to continue the PT for the analyses of banned colorants in textile during the annual proficiency testing program 2019/2020.

In this interlaboratory study 96 laboratories in 26 different countries registered for participation. See appendix 4 for the number of participation. In this report the results of the 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 analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send 2 different textile samples of approximately 3 grams each. A beige polyester sample labelled #20555 and a pink/purple acrylic sample with fluffy fibers labelled #20556 which were both artificially fortified with different banned colorants.

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 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

For the first sample a batch of beige polyester was selected which was dyed with Disperse Blue 102 and Disperse Orange 76, both allergenic colorants, by a third party. This batch was cut into small pieces. After homogenization the batch was divided over 110 subsamples in small bags of approximately 3 grams each and labelled #20555.

The homogeneity of the subsamples was checked by determination of Disperse Blue 102 and Disperse Orange 76 according to DIN54231 on eight stratified randomly selected subsamples.

	Disperse Blue 102 in mg/kg	Disperse Orange 76 in mg/kg		
sample #20555-1	45.86	188.92		
sample #20555-2	44.90	186.75		
sample #20555-3	47.36	200.70		
sample #20555-4	45.09	194.60		
sample #20555-5	45.53	209.33		
sample #20555-6	46.34	197.54		
sample #20555-7	47.40	205.49		
sample #20555-8	47.73	208.34		

Table 1: homogeneity test results of subsamples #20555

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

	Disperse Blue 102 in mg/kg	Disperse Orange 76 in mg/kg
r (observed)	3.10	23.49
reference test method	DIN54231:05	DIN54231:05
0.3 * R (reference test method)	11.12	47.86

Table 2: repeatabilities of subsamples #20555

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

For the second sample a batch of purple acrylic was selected which was dyed with Basic Violet 14, a carcinogenic colorant, by a third party. This batch contained out of fluffy fibers. After homogenization the batch was divided over 110 subsamples in small bags of approximately 3 grams each and labelled #20556.

The homogeneity of the subsamples was checked by determination Basic Violet 14 according to DIN54231 on eight stratified randomly selected subsamples.

	Basic Violet 14 in mg/kg
sample #20556-1	49.4
sample #20556-2	50.2
sample #20556-3	48.6
sample #20556-4	49.6
sample #20556-5	52.2
sample #20556-6	52.1
sample #20556-7	52.3
sample #20556-8	50.4

Table 3: homogeneity test results of subsamples #20556

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

	Basic Violet 14 in mg/kg
r (observed)	4.0
reference test method	DIN54231:05
0.3 * R (reference test method)	12.2

Table 4: repeatability of subsamples #20556

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

To each participating laboratory one sample labelled #20555 and one sample labelled #20556 was sent on March 4, 2020.

### 2.5 ANALYZES

The participants were requested to determine the concentrations of 19 banned allergenic dyes, 10 banned carcinogenic dyes and 8 other banned dyes on sample #20555 and sample #20556, applying the analysis procedure that is routinely used in the laboratory. See list of colorants in appendix 2.

It was requested, to ensure homogeneity, to not use less than 0.5 grams per determination. It was also requested to report if the laboratory was accredited to determine the reported components and to report some analytical details.

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 results, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are above the detection limit, because such results cannot be used for meaningful statistical evaluations.

To get comparable 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 appropriate reference test method 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 appendices 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 reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

#### 3.1 STATISTICS

The protocol followed in the organization 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 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 dataset does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by D(0.05) for the Grubbs' test and by D(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 these 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. 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 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 - average of PT)} / \text{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

During the execution of this proficiency test some serious problems occurred. Due to COVID-19 outbreak thirty participants were not able to participate in this proficiency test because of restricted dispatch to some countries. It was decided that for those participants the deadline was extended. After closure of the round an extra round was created on the Data Entry Portal to enable participants to report the test results at a later moment. The test results of the extra round were not added to the evaluation data of this report but will be evaluated separately compared to this PT report. Not all laboratories were able to perform all analyzes requested. Finally, 66 participants reported 202 numerical test results. Observed were 16 outlying test results, which is 7.9% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

#### 4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the 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 test methods are also in the tables in appendix 1 together with the original data. The abbreviations used in these tables are explained in appendix 5.

In DIN54231:2005 no reproducibility is mentioned. Only the standard deviation for the repeatability is mentioned. Therefore, the target reproducibility was estimated as follows: the repeatability standard deviation was multiplied with 2.8 to get the target repeatability. And this was multiplied with 3 to get an estimate of the target reproducibility.

### Sample #20555

<u>Disperse Blue 102 (CASno. 12222-97-8)</u>: The determination of this colorant at a concentration level of 13 mg/kg was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility estimated from the test method DIN54231:05.

<u>Disperse Orange 76 = 37 (CASno. 13301-61-6)</u>: The determination of this colorant at a concentration level of 255 mg/kg may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility estimated from the test method DIN54231:05.

### Sample #20556

Basic Red 9 (CASno. 569-61-9): The determination of this colorant may be problematic at a concentration level of 14 mg/kg. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility estimated from the test method DIN54231:05.

<u>Basic Violet 14 (CASno. 632-99-5)</u>: The determination of this colorant at a concentration level of 68 mg/kg was problematic for a number of participants. Seven statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility estimated from the test method DIN54231:05.

The majority of the participants agreed on a concentration near or below the limit of detection for all other colorants mentioned in appendix 2. Therefore, no z-scores were calculated for these components. The 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 reproducibilities as declared by the relevant reference test methods and the reproducibilities as found for the group of participating laboratories. The number of test results, the average, the calculated reproducibilities (2.8\*standard deviation) and the target reproducibilities, estimated from the test method DIN54231:05 are compared in the next two tables.

Component	unit	n	average	2.8 * sd	R(lit)
Disperse Blue 102	mg/kg	39	13.29	8.85	10.65
Disperse Orange 76 = 37	mg/kg	63	255.2	234.0	204.5

Table 5: reproducibilities of the colorants in textile sample #20555

Component	unit	n	average	2.8 * sd	R(lit)
Basic Red 9	mg/kg	35	14.49	13.18	11.61
Basic Violet 14	mg/kg	49	68.42	56.23	54.83

Table 6: reproducibilities of the colorants in textile sample #20556

Without further statistical calculations, it can be concluded that dependent on the sample the group of participating laboratories shows a good compliance to the majority of the analyses at the investigated concentration levels. See also the discussion in paragraphs 4.1 and 5.

#### 4.3 EVALUATION OF THE PROFICIENCY TEST OF MARCH 2020 WITH PREVIOUS PTS

	March 2020	March 2019	March 2018	February 2017	February 2016
Number of reporting laboratories	66	78	88	86	80
Number of test results	202	297	657	244	233
Number of statistical outliers	16	10	21	8	7
Percentage of statistical outliers	7.9%	3.4%	3.2%	3.3%	3.0%

Table 7: Comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The uncertainties in the test results of the evaluated colorants in the iis20A06 PT are listed in below table and are compared with previous proficiency tests.

	March 2020	March 2019	March 2018	February 2017	2006 – 2016	target DIN54321
Disperse Blue 1	n.e.	n.e.	n.e.	n.e.	43%	27%
Disperse Blue 3	n.e.	n.e.	n.e.	n.e.	36 - 56%	27%
Disperse Blue 26	n.e.	n.e.	n.e.	n.e.	47 - 68%	27%
Disperse Blue 35	n.e.	n.e.	n.e.	n.e.	31 - 84%	27%
Disperse Blue 102	24%	n.e.	n.e.	n.e.	n.e.	27%
Disperse Blue 106	n.e.	n.e.	n.e.	50%	28%	27%
Disperse Brown 1	n.e.	n.e	n.e	39%	33%	27%
Disperse Orange 1	n.e.	n.e.	35%	n.e.	42 - 47%	27%
Disperse Orange 3	n.e.	n.e.	n.e.	n.e.	24 - 54%	27%
Disperse Orange 76/37	33%	n.e.	33%	n.e.	n.e	27%
Disperse Orange 149	n.e.	21%	27%	n.e.	n.e	27%
Disperse Red 1	n.e.	33%	n.e.	n.e.	36 - 63%	27%
Disperse Red 11	n.e.	n.e.	n.e.	n.e.	41 - 65%	27%
Disperse Red 17	n.e.	n.e.	28%	n.e.	28 - 33%	27%
Disperse Yellow 1	n.e.	n.e.	n.e.	n.e.	24%	27%
Disperse Yellow 3	n.e.	34%	21%	n.e.	28 - 30%	27%
Disperse Yellow 9	n.e.	n.e.	21%	n.e.	31%	27%
Disperse Yellow 23	n.e.	13%	17%	n.e.	n.e	27%
Disperse Yellow 49	n.e.	n.e.	n.e.	n.e.	54%	27%
Basic Red 9	33%	n.e.	n.e.	n.e.	n.e.	27%
Basic Violet 14	29%	n.e.	n.e.	n.e.	n.e.	27%
Direct Black 38	n.e.	n.e.	n.e.	32%	n.e.	27%

Table 8: development of uncertainties over the last years

It is observed that the group performed the same at the determination of Disperse Orange 76 in this PT in comparison with the PT in 2018. Uncertainties of Disperse Blue 102, Basic Red 9 and Basic Violet 14 observed in this PT are close to or in line with the requirements mentioned in the target.

#### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

The test method DIN54231 is used by about 87% of the reporting participants.

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

- About 85% of the participants mentioned that they are accredited for the determination of banned dyes in textile.
- About 3% of the participants used less than 0.5 grams as sample intake, about 65% used 0.5 grams and about 30% used about 1 gram as sample intake.

No effect has been observed of the difference in sample intake

#### 5 DISCUSSION

All reporting participants were able to detect in Disperse Blue 102 and Disperse Orange 76 in sample #20555 and Basic Red 9 and Basic Violet 14 in sample #20556. No other banned colorants were detected.

When the results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU and with Bluesign® BSSL (see table 9), it was noticed that not all participants would make identical decisions about the acceptability of the textiles for the determined components.

Ecolabel	baby clothes	in direct skin contact	no direct skin contact	
bluesign® BSSL	<20 mg/kg	<20 mg/kg	<20 mg/kg	
Oeko-Tex standard	<20 mg/kg	<20 mg/kg	<20 mg/kg	

Table 9: Bluesign® BSSL and Ecolabelling Standards and Requirements for Textiles in EU

For sample #20555 all reporting laboratories would have rejected the sample for containing too much Disperse Orange 76 for all categories. For sample #20556 all reporting laboratories would have rejected the sample for containing too much Basic Violet 14 for all categories. For Disperse Blue 102 and Basic Red 9 in total four laboratories would have rejected the sample.

#### 6 CONCLUSION

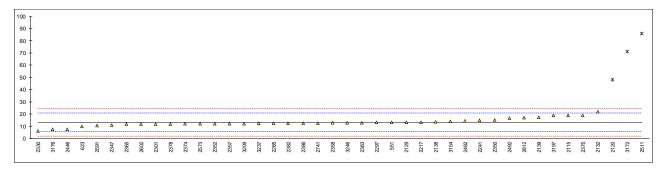
The variation in this interlaboratory study is clearly not caused by just one critical point in the analysis. Almost all participants reported to have used DIN 54231. However, the detection technique and the purity of the various calibration standards that are used may vary strongly. Each participating 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.

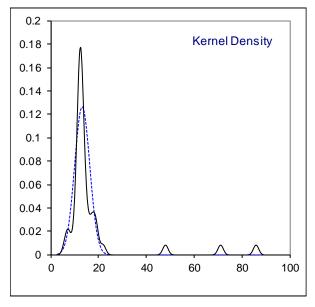
## **APPENDIX 1**

Determination of Disperse Blue 102 (CASno. 12222-97-8) in sample #20555; results in mg/kg

	nination of Disperse Blue 10				
lab	method	value	mark	z(targ)	remarks
210					
230 362					
36∠ 551	In house	13.1877		-0.03	
623	DIN54231	10.07		-0.85	
840	DIN54231	n.d.			
841	DIN54231	<15			
2115	DIN54231	18.92		1.48	
2120	ISO16373-2	48	R(0.01)	9.13	
2121					
2129	DIN54231	13.2		-0.02	
2132	In house	22 		2.29	
2135 2138	DIN54231	13.82		0.14	
2139	BS EN 71-10	17.4		1.08	
2165	DIN54231	ND			
2213					
2217	DIN54231	13.2106		-0.02	
2241	DIN54231	14.8		0.40	
2247					
2265	DIN54231	12.6		-0.18	
2284	DIN54231	<15			
2290 2293	DIN54231	< 15 			
2295					
2297	DIN54231	13.13		-0.04	
2301		11.84	С	-0.38	first reported 70.49
2310					•
2311					
2320					
2330	DIN54231	6.24		-1.85	
2347	DIN54231	11		-0.60	
2350 2352	DIN54231 DIN54231	15.15 12.2		0.49 -0.29	
2357	DIN54231	12.2		-0.29	
2358	DIN54231	12.76		-0.14	
2363	DIN54231	13		-0.08	
2365	DIN54231	11.7		-0.42	
2366	DIN54231	<15			
2370	DIN54231	<15			
2374	DIN54231	12.04		-0.33	
2375	DIN54231	19 11.9		1.50	
2378 2379	DIN54231 DIN54231	Not detected		-0.36 	
2380	D1110-1201				
2382	DIN54231	12.62		-0.18	
2386	DIN54231	12.62		-0.18	
2390					
2425		 7.050			
2446	In house	7.650		-1.48	
2452 2462	DIN54231&GB/T20382/20383	 -15			
2462 2482	DIN54231&GB/120382/20383 DIN54231	<15 14.5		0.32	
2492	In house	16.8		0.92	
2511	DIN54231	85.76	C,R(0.01)	19.06	first reported 74.33
2522	DIN54231	<20.0	/		•
2532					
2538	DIN54231	detected			
2549					
2560 2561					
2573	DIN54231	12.13		-0.30	
2590	D.1107201	12.13		-0.30	
2591	In house	10.7		-0.68	
2602	DIN54231	11.7		-0.42	
2618					
2643					
2644					
2668					
2672 2674	DIN5/1231	 ∠DI			
2674 2678	DIN54231	<rl </rl 			
2689	DIN54231	<15	С		first reported ND
2741	DIN54231	12.73	-	-0.15	
2743					

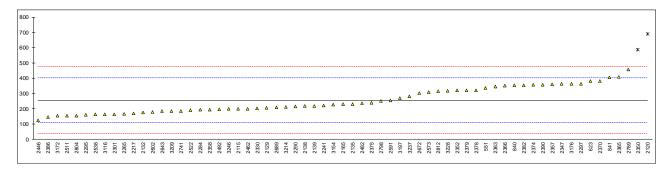
lab	method	value	mark	z(targ)	remarks
2773					
2789					
2798					
2804	DIN54231	<30			
2812	DIN54231	17.12		1.01	
2827					
2912					
3116					
3146					
3154	DIN54231	14.23		0.25	
3172	DIN54231	70.995	R(0.01)	15.17	
3176	ISO16373-2	7.50		-1.52	
3197	DIN54231	18.9		1.48	
3200					
3209	DIN54231	12.20		-0.29	
3210					
3214	DIN54231	<15			
3228	DIN54231	N.D.			
3233					
3237	DIN54231	12.57		-0.19	
3246	DIN54231	12.88	С	-0.11	first reported n.d.
	normality	suspect			
	n	39			
	outliers	3			
	mean (n)	13.288			
	st.dev. (n)	3.1606	RSD=24%		
	R(calc.)	8.850			
	st.dev.(DIN54231:05)	3.8029			
	R(DIN54231:05)	10.648			
	st.dev. (n) R(calc.) st.dev.(DIN54231:05)	8.850 3.8029	RSD=24%		

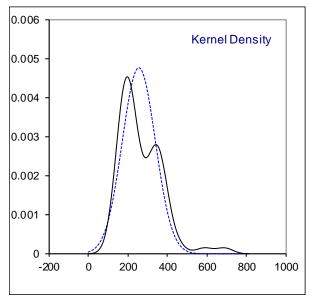




Determination of Disperse Orange 76 = 37 (CASno. 13301-61-6) in sample #20555; results in

mg/kg					
lab	method	value	mark	z(targ)	remarks
210					
230					
362					
551	In house	337.1950		1.12	
623	DIN54231	380.49		1.71	
840	DIN54231	353.0		1.34	
841	DIN54231	405.57		2.06	
2115	DIN54231	200.43		-0.75	
2120	ISO16373-2	689	R(0.01)	5.94	
2121	B.11.5.45.4				
2129	DIN54231	206		-0.67	
2132	In house	176		-1.08	
2135	DINE 4004	231.5		-0.33	
2138	DIN54231	218.51		-0.50	
2139	BS EN 71-10	219.4		-0.49	
2165	DIN54231	229.7		-0.35 	
2213 2217	DIN54231	168.3543		-1.19	
2241	DIN54231	222.1		-0.45	
2247	DIN34231			-0.45	
2265	DIN54231	167.0		-1.21	
2284	DIN54231	192.725		-0.86	
2290	DIN54231	214.8		-0.55	
2293	DIN34231			-0.55	
2295	DIN54231	161.0		-1.29	
2297	DIN54231	364.2		1.49	
2301	2.1.0.120.	165.07		-1.23	
2310					
2311					
2320					
2330	DIN54231	203.37		-0.71	
2347	DIN54231	362		1.46	
2350	DIN54231	585.81	R(0.05)	4.53	
2352	DIN54231	320.4	, ,	0.89	
2357	DIN54231	361.1		1.45	
2358	DIN54231	194.27		-0.83	
2363	DIN54231	346		1.24	
2365	DIN54231	408.0		2.09	
2366	DIN54231	350		1.30	
2370	DIN54231	381		1.72	
2374	DIN54231	356.89		1.39	
2375	DIN54231	240	С	-0.21	first reported 519
2378	DIN54231	322.4		0.92	
2379	DIN54231	322.34		0.92	
2380					
2382	DIN54231	355.52		1.37	
2386	DIN54231	145.82		-1.50	
2390	DIN54231	358.01		1.41	
2425	Labarra	400.004		4.04	
2446	In house	123.201		-1.81	
2452	DINE 4224 9 CD/T20202/20202	 200 F		0.75	
2462 2482	DIN54231&GB/T20382/20383 DIN54231	200.5 237		-0.75 -0.25	
2402	In house	197.1		-0.25	
2511	DIN54231	155.1924		-1.37	
2522	DIN54231	190.17		-0.89	
2532	51110-1201				
2538	DIN54231	163.74		-1.25	
2549	2.1.0.120.				
2560					
2561					
2573	DIN54231	310.04		0.75	
2590					
2591	In house	253.9		-0.02	
2602	DIN54231	177.5		-1.06	
2618					
2643	DIN54231	183.7		-0.98	
2644					
2668					
2672	DIN54231	302		0.64	
2674	DIN54231	N.A.			
2678	B.11.5.400.4				
2689	DIN54231	210		-0.62	
2741	DIN54231	186.38		-0.94	
2743					



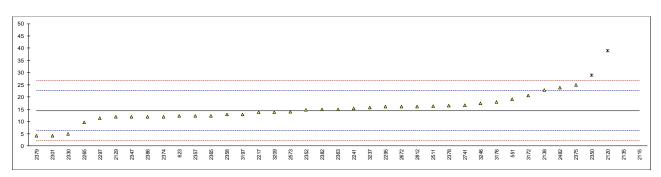


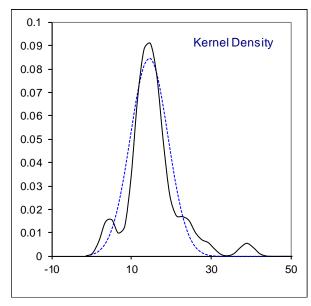
## Determination of Basic Red 9 (CASno. 569-61-9) in sample #20556; results in mg/kg

lah	moth od	value	moule	=/tora\	- vamaula
210	method	value	mark	z(targ)	remarks
230					
362					
551	In house	19.06	С	1.10	first reported N.D.
623	DIN54231	12.32		-0.52	
840	DIN54231	n.d.			
841 2115	DIN54231	<15	R(0.01)	94.07	
2113	DIN54231 ISO16373-2	404.44 39	R(0.01) R(0.01)	5.91	
2121	10010073 2		11(0.01)		
2129	DIN54231	12.0		-0.60	
2132	In house	<10			
2135		341.4	R(0.01)	78.86	
2138	DIN54231	22.85		2.02	
2139 2165	DIN54231	 ND			
2213	DIN34231				
2217	DIN54231	13.8111		-0.16	
2241	DIN54231	15.4		0.22	
2247					
2265	DIN54231	9.6		-1.18	
2284	DIN54231 DIN54231	<15 < 15			
2290 2293	DIN34231	< 15 			
2295	DIN54231	16.0		0.37	
2297	DIN54231	11.33		-0.76	
2301	DIN54231	4.17		-2.49	
2310					
2311					
2320	DINE4224	4.86	С	-2.32	first reported ND
2330 2347	DIN54231 DIN54231	12	C	-2.32 -0.60	first reported ND
2350	5.110.120.1	28.92	R(0.01)	3.48	
2352	DIN54231	14.8	( /	0.08	
2357	DIN54231	12.4		-0.50	
2358	DIN54231	12.89		-0.38	
2363	DIN54231	15		0.12	
2365 2366	DIN54231 DIN54231	12.4 <15		-0.50 	
2370	DIN54231	<15			
2374	DIN54231	12.03		-0.59	
2375	DIN54231	25		2.54	
2378	DIN54231	16.4		0.46	
2379	DIN54231	4.16		-2.49	
2380 2382	DIN54231	 14.95		0.11	
2386	DIN54231	12.02		-0.59	
2390	21020.				
2425					
2446					
2452	DINE 4004 8 CD/T00000/00000	.4.5			
2462 2482	DIN54231&GB/T20382/20383 DIN54231	<15 23.8		2.25	
2492	D111034231			2.23	
2511	DIN54231	16.2603		0.43	
2522	DIN54231	NA			
2532					
2538		see additional remark *)			
2549 2560					
2560 2561					
2573	DIN54231	13.97		-0.12	
2590					
2591					
2602					
2618 2643					
2644					
2668					
2672	DIN54231	16	С	0.37	first reported 40.5
2674	DIN54231	<rl< td=""><td></td><td></td><td></td></rl<>			
2678	DINE 1004	.45	0		first new autout NID
2689 2741	DIN54231 DIN54231	<15 16.72	С	0.54	first reported ND
2741	DINOTEO	10.72		0.54	
2.40					

lab	method	value	mark	z(targ)	remarks
2773					
2789					
2798					
2804	DIN54231	<30			
2812	DIN54231	16.12		0.39	
2827					
2912					
3116					
3146					
3154					
3172	DIN54231	20.668		1.49	
3176	ISO16373-2	18.0		0.85	
3197	DIN54231	12.9		-0.38	
3200					
3209	DIN54231	13.83		-0.16	
3210					
3214					
3228	DIN54231	N.D.			
3233					
3237	DIN54231	15.77		0.31	
3246	DIN54231	17.47	С	0.72	first reported N.D.
	normality	OK			
	n	35			
	outliers	4			
	mean (n)	14.485			
	st.dev. (n)	4.7073	RSD=33%		
	R(calc.)	13.180			
	st.dev.(DIN54231:05)	4.1455			
	R(DIN54231:05)	11.607			

\*) Due to same spectra and retention time like the first peak of Basic Violet 14 it is not possible to decide if Basic Red 9 is also present in the sample or not.

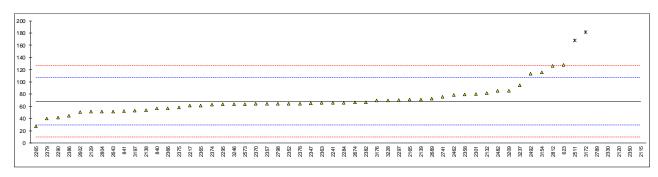


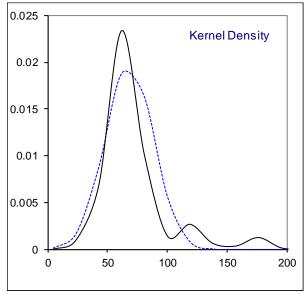


## Determination of Basic Violet 14 (CASno. 632-99-5) in sample #20556; results in mg/kg

lah	method	value	mork	T/tora)	romorko
210	metriod	value	mark	z(targ)	remarks
230					
362					
551					
623	DIN54231	127.73		3.03	
840	DIN54231	57.0		-0.58	
841 2115	DIN54231 DIN54231	52.12 4848.44	R(0.01)	-0.83 244.12	
2120	ISO16373-2	603	R(0.01)	27.30	
2121	100 100 10 2		11(0.01)		
2129	DIN54231	51.2		-0.88	
2132	In house	81.6		0.67	
2135	DINIE 4004		•		f
2138 2139	DIN54231	53.64 71.4	С	-0.75 0.15	first reported 197.12
2165	BS EN 71-10 DIN54231	71.4		0.13	
2213	D11104201				
2217	DIN54231	61.664		-0.34	
2241	DIN54231	66.0		-0.12	
2247	DINE 1004				
2265	DIN54231	27.7 66.168		-2.08	
2284 2290	DIN54231 DIN54231	41.6		-0.11 -1.37	
2293	D11134231			-1.57	
2295	DIN54231	63.7	С	-0.24	first reported 190.0
2297	DIN54231	70.23		0.09	·
2301	DIN54231	80.03		0.59	
2310					
2311 2320					
2320	DIN54231	420.89	R(0.01)	18.00	
2347	DIN54231	65	11(0.01)	-0.17	
2350		1521.16	R(0.01)	74.19	
2352	DIN54231	64.3		-0.21	
2357	DIN54231	64.0		-0.23	
2358	DIN54231	79.85		0.58	
2363 2365	DIN54231 DIN54231	66 61.7		-0.12 -0.34	
2366	DIN54231	57		-0.58	
2370	DIN54231	64.0		-0.23	
2374	DIN54231	62.49		-0.30	
2375	DIN54231	58	С	-0.53	first reported 325
2378 2379	DIN54231	64.4 40.20		-0.21 -1.44	
2379	DIN54231	40.20		-1.44	
2382	DIN54231	66.32		-0.11	
2386	DIN54231	44.42		-1.23	
2390	DIN54231	ND	С		first reported 195.01
2425					
2446					
2452 2462	DIN54231&GB/T20382/20383	78.5		0.52	
2482	DIN54231	85.5		0.87	
2492	In house	113.8		2.32	
2511	DIN54231	168.01	C,R(0.01)	5.09	first reported 185.3645
2522	DIN54231	NA			
2532 2538		dotoctod			
2549		detected			
2560					
2561					
2573	DIN54231	63.99		-0.23	
2590					
2591 2602	DIN54231	50.5		-0.91	
2602 2618	DINU4231	50.5		-0.91	
2643	DIN54231	51.7		-0.85	
2644					
2668					
2672	DINE 1004				
2674 2678	DIN54231	66.3 		-0.11 	
2678 2689	DIN54231	73		0.23	
2741	DIN54231	75.58		0.23	
2743					

lab	method	value	mark	z(targ)	remarks
2773					
2789		315.4	C,R(0.01)	12.61	first reported 226.45
2798	GB/T20382	64		-0.23	
2804	DIN54231	51.6		-0.86	
2812	DIN54231	126.1		2.95	
2827					
2912					
3116					
3146					
3154	DIN54231	115.708		2.42	
3172	DIN54231	181.285	R(0.01)	5.76	
3176	ISO16373-2	69.30		0.05	
3197	DIN54231	53.2		-0.78	
3200					
3209	DIN54231	85.53		0.87	
3210					
3214					
3228	DIN54231	69.3		0.05	
3233					
3237	DIN54231	94.27		1.32	
3246	DIN54231	63.9		-0.23	
	normality	not OK			
	n	49			
	outliers	7			
	mean (n)	68.415			
	st.dev. (n)	20.0828	RSD=29%		
	R(calc.)	56.232			
	st.dev.(DIN54231:05)	19.5804			
	R(DIN54231:05)	54.825			





## **APPENDIX 2 Other reported banned colorants**

## Abbreviations and details of allergenic colorants, see also Oekotex 100:

DB1 DB3	: Disperse Blue 1 : Disperse Blue 3	CASno 2475-45-8 CASno 2475-46-9	C.I.no 64 500 C.I.no 61 505
DB7	: Disperse Blue 7	CASno 3179-90-6	C.I.no 62 500
DB26	: Disperse Blue 26	CASno 3860-63-7	C.I.no 63 305
DB35	: Disperse Blue 35*	CASno 12222-75-2 (*)	
DB35a	: Disperse Blue 35a	CASno 56524-77-7	
DB35b	: Disperse Blue 35b	CASno 56524-76-6	
DB102	: Disperse Blue 102	CASno 12222-97-8	
DB106	: Disperse Blue 106	CASno 12223-01-7	
DB124	: Disperse Blue 124	CASno 61951-51-7	
DBr1	: Disperse Brown 1	CASno 23355-64-8	
DO1	: Disperse Orange 1	CASno 2581-69-3	C.I.no 11 080
DO3	: Disperse Orange 3	CASno 730-40-5	C.I.no 11 005
DO76	: Disperse Orange 76=37	CASno 13301-61-6	C.I.no 11 132
DR1	: Disperse Red 1	CASno 2872-52-8	C.I.no 11 110
DR11	: Disperse Red 11	CASno 2872-48-2	C.I.no 62 015
DR17	: Disperse Red 17	CASno 3179-89-3	C.I.no 11 210
DY1	: Disperse Yellow 1	CASno 119-15-3	C.I.no 10 345
DY3	: Disperse Yellow 3	CASno 2832-40-8	C.I.no 11 855
DY9	: Disperse Yellow 9	CASno 6373-73-5	C.I.no 10 37
DY39	: Disperse Yellow 39	CASno 12236-29-2	
DY49	: Disperse Yellow 49	CASno 54824-37-2	

<sup>\*</sup> Disperse Blue 35 consists of a mixture of components, of which the monomethylated 1,8-diamino-4,5-dihydroxy-anthraquinone (CASno 56524-77-7) and the dimethylated 1,8-diamino-4,5-dihydroxy-anthraquinone (CASno 56524-76-6)

are responsible for the sensitizing potency of Disperse Blue 35, see also report iis09A04X of May 2009.

## Abbreviations and details of carcinogenic colorants, see also Oekotex 100:

AR26	: Acid Red 26	CASno 3761-53-3	C.I. 16 150
BB26	: Basic Blue 26	CASno 2580-56-5	
BR9	: Basic Red 9	CASno 569-61-9	C.I. 42 500
BV3	: Basic Violet 3	CASno 548-62-9	
BV14	: Basic Violet 14	CASno 632-99-5	C.I. 42 510
DBI38	: Direct Black 38	CASno 1937-37-7	C.I. 30 235
DB6	: Direct Blue 6	CASno 2602-46-2	C.I. 22 610
DR28	: Direct Red 28	CASno 573-58-0	C.I. 22 120
DO11	: Disperse Orange 11	CASno 82-28-0	C.I. 60 700

## Abbreviations and details of other banned colorants colorants, see also Oekotex 100:

: Disperse Orange 149	CASno 85136-74-9	
: Disperse Yellow 23	CASno 6250-23-3	C.I. 26 070
: Basic Green 4 (oxalate)	CASno 2437-29-8	
: Basic Green 4 (chloride)	CASno 569-64-2	
: Basic Green 4 (free)	CASno 10309-95-2	
: Navy Blue	EG-no.405-665-4	
	: Disperse Yellow 23 : Basic Green 4 (oxalate) : Basic Green 4 (chloride) : Basic Green 4 (free)	: Disperse Yellow 23 CASno 6250-23-3 : Basic Green 4 (oxalate) CASno 2437-29-8 : Basic Green 4 (chloride) CASno 569-64-2 : Basic Green 4 (free) CASno 10309-95-2

## Other reported allergenic Colorants in sample #20555; results in mg/kg

lab	DB 1	DB 3	DB 7	DB 26	DB 35	DB 35a	DB 35b	DB 106	DB 124	DBr 1	DO 1	DO 3
210												
230												
362												
551 623	N.D. ND	N.D. ND	N.D. ND	N.D. ND	ND	ND	ND	ND	N.D. ND	N.D. ND	N.D. ND	N.D. ND
840	n.d.											
841	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2115												
2120	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
2121 2129												
2132	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2135								9.3				
2138	ND											
2139	ND.	ND.	ND	ND	NID.	NID.	NID.	NID.	ND.	NID.	NID.	ND
2165 2213	ND 											
2217												
2241	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2247		45				45	45		45	45	45	
2265 2284	< 15 <15											
2290	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15
2293												
2295												
2297	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2301 2310												
2311												
2320												
2330	ND											
2347 2350	<15 <7.50											
2352	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50
2357												
2358	n.d.											
2363	ND .15	ND 115	ND -15	ND -15	ND 115	ND 115	ND 115	ND .15	ND 115	ND 11.5	ND	ND 115
2365 2366	<15 <15											
2370	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2374												
2375												
2378 2379	ND											
2380												
2382	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0
2386	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2390 2425								13.95 				
2425												
2452												
2462	<15	<15	<15	<15	<15			<15	<15	<15	<15	<15
2482 2492												
2492 2511												
2522	<20.0	<20.0	<20.0	<20.0	<20.0	NA	NA	<20.0	<20.0	<20.0	<20.0	<20.0
2532												
2538	ND	ND	ND	ND				ND	ND	ND	ND 	ND
2549 2560												
2561												
2573	ND											
2590												
2591 2602												
2618												
2643	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2644												
2668 2672	<10	<10			<10			<10	<10			<10
2674	n.d.	n.d.	n.d.	n.d.	n.d.			n.d.	n.d.	n.d.	n.d.	n.d.
2678												
2689	ND	ND	ND	ND	ND			ND	ND	ND	ND	ND
2741	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2743												

lab	DB 1	DB 3	DB 7	DB 26	DB 35	DB 35a	DB 35b	DB 106	DB 124	DBr 1	DO 1	DO 3
2773												
2789												
2798												
2804	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
2812												
2827												
2912												
3116												
3146												
3154												
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176												
3197	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3200												
3209												
3210												
3214	<15	<15	<15	<15	<15	<15	<15	<15	<15		<15	<15
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.
3233												
3237												
3246	n.d.	n.d.	n.d.		n.d.	n.d.		n.d.	n.d.	n.d.	n.d.	n.d.

Other reported allergenic Colorants in sample #20555; results in mg/kg -- continued --

lab	DR 1	DR 11	DR 17	DY 1	DY 3	DY 9	DY 39	DY 49	AR 26	BB 26	BR 9	BV 3
210						פוט	D1 39	D1 49	AR 20	DD 20	DK 9	DV 3
230												
362												
551	N.D.				N.D.	N.D.						
623	ND.	ND	ND	ND	ND.	ND.						
840	n.d.											
841	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2115												
2120	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
2121												
2129												
2132	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2135												
2138	ND											
2139												
2165	ND											
2213												
2217												
2241	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2247												
2265	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15
2284	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2290	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15
2293												
2295												
2297	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2301												
2310												
2311												
2320												
2330	ND											
2347	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2350	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50
2352												
2357												
2358	n.d.											
2363	ND											
2365	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2366	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2370	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2374												
2375												
2378												
2379	ND											
2380												
2382	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0
2386	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2390												
2425												

lab	DR 1	DR 11	DR 17	DY 1	DY 3	DY 9	DY 39	DY 49	AR 26	BB 26	BR 9	BV 3
2446												
2452												
2462	<15	<15	<15	<15	<15	<15	<15	<15	<15		<15	
2482												
2492												
2511												
2522	<20.0	<20.0	<20.0	<20.0	<20.0	<20.0	<20.0	<20.0	NA	NA	NA	NA
2532												
2538	ND		ND		ND	ND						
2549												
2560												
2561												
2573	ND	ND	ND									
2590												
2591												
2602												
2618												
2643	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2644												
2668												
2672	<10				<10				<10		<10	<10
2674	n.d.	n.d.	n.d.									
2678												
2689	ND	ND	ND									
2741	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2743												
2773												
2789												
2798												
2804	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
2812												
2827												
2912												
3116												
3146												
3154												
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176												
3197	ND	ND	ND									
3200												
3209												
3210												
3214	<15	<15	<15	<15	<15	<15	<15	<15				
3228	N.D.	N.D.	N.D.									
3233												
3237												
3246	n.d.			n.d.	n.d.							

Other reported allergenic Colorants in sample #20555; results in mg/kg -- continued --

lab	BV 14	DBI 38	DB 6	DR 28	DO 11	DO 149	DY 23	BG 4o	BG 4c	BG 4f	NB
210											
230											
362											
551						N.D.	N.D.				
623	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
841	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2115											
2120	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	
2121											
2129											
2132	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2135											
2138	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2139											
2165	ND	ND	ND	ND	ND	ND	ND				
2213											
2217											
2241	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2247											
2265	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15
2284	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2290	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15

lab	BV 14	DBI 38	DB 6	DR 28	DO 11	DO 149	DY 23	BG 4o	BG 4c	BG 4f	NB
2293											
2295											
2297	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2301											
2310 2311											
2320											
2330	ND	NA	NA	ND							
2347	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	
2350	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50
2352											
2357	 n d						 n d	 n d	 n d		 n d
2358 2363	n.d. ND										
2365	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2366	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2370	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2374											
2375 2378											
2379	ND	NB									
2380											
2382	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0
2386	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2390											
2425 2446											
2452											
2462	<15	<15		<15	<15	<15	<15				
2482											
2492											
2511 2522	NA	NA	NA	NA	NA	<20.0	<20.0	NA	NA	NA	NA
2532											
2538	ND										
2549											
2560											
2561 2573	ND										
2590											
2591											
2602											
2618											
2643 2644	< 5 										
2668											
2672											
2674	n.d.										
2678 2689	ND										
2741	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2743											
2773											
2789											
2798 2804	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	
2812											
2827											
2912											
3116											
3146 3154											
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176											
3197	ND										
3200											
3209 3210											
3214											
3228	N.D.										
3233											
3237 3246	n.d.				n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	
3240	n.u.				n.u.	n.u.	n.u.	n.u.	n.u.	II.U.	

## Other reported allergenic Colorants in sample #20556; results in mg/kg

lab	DB 1	DB 3	DB 7	DB 26	BD 35	DB 35a	DB 35b	DB 102	DB 106	DB 124	DBr 1	DO 1
210												
230												
362 551	N.D.	N.D.	N.D.	N.D.				N.D.		N.D.	N.D.	N.D.
623	ND	ND	ND	ND.	ND	ND	ND	ND	ND	ND	ND	ND
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
841	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2115												
2120 2121	< 10 	< 10 	< 10 	< 10 	< 10 	< 10 	< 10 	< 10 	< 10 	< 10 	< 10 	< 10 
2129												
2132	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2135	ND.	ND	ND	ND	ND	ND	ND	ND	ND	ND	NID.	ND
2138 2139	ND 	ND 	ND 	ND 	ND 	ND 	ND 	ND 	ND 	ND 	ND 	ND 
2165	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2213												
2217		-10		-10	-10	-10		-10			-10	-10
2241 2247	<10 	<10 	<10 	<10 	<10 	<10 	<10 	<10 	<10 	<10 	<10 	<10 
2265	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15
2284	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2290	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15
2293 2295												
2297	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2301												
2310												
2311 2320												
2330	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2347	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2350	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50
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.
2363	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2365	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2366 2370	<15	<15 <15	<15	<15	<15	<15 <15	<15 <15	<15 <15	<15	<15 <15	<15	<15 <15
2374	<15 		<15 	<15 	<15 				<15 		<15 	
2375												
2378												
2379 2380	ND 	ND 	ND 	ND 	ND 	ND 	ND 	ND 	ND 	ND 	ND 	ND 
2382	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0
2386	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2390												
2425 2446	>50											
2446	>500											
2462	<15	<15	<15	<15	<15			<15	<15	<15	<15	<15
2482												
2492 2511												
2522	<20.0	<20.0	<20.0	<20.0	<20.0	NA	NA	<20.0	<20.0	<20.0	<20.0	<20.0
2532												
2538	ND	ND	ND	ND				ND	ND	ND	ND	ND
2549 2560												
2561												
2573	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2590												
2591												
2602 2618												
2643	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2644												
2668		-10										
2672 2674	<10 n.d.	<10 n.d.	n.d.	n.d.	<10 n.d.			n.d.	<10 n.d.	<10 n.d.	n.d.	n.d.
2678				11.u. 								
2689	ND	ND	ND	ND	ND			ND	ND	ND	ND	ND
2741	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2743												

lab	DB 1	DB 3	DB 7	DB 26	BD 35	DB 35a	DB 35b	DB 102	DB 106	DB 124	DBr 1	DO 1
2773												
2789												
2798												
2804	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
2812												
2827												
2912												
3116												
3146												
3154												
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176												
3197	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3200												
3209												
3210												
3214	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15		<15
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.
3233												
3237												
3246	n.d.	n.d.	n.d.		n.d.	n.d.		n.d.	n.d.	n.d.	n.d.	n.d.

Other reported allergenic Colorants in sample #20556; results in mg/kg -- continued --

lak	DO 2	DO 76	DD 4	DD 11	DD 47	DY 1	DY 3	DV 0	DV 20	DY 49	AD 26	DD 26
lab	DO 3	DO 76	DR 1	DR 11	DR 17			DY 9	DY 39		AR 26	BB 26
210												
230												
362												
551	N.D.	NID	NID.	NID.								
623	ND	ND.	ND.	ND	ND.	ND.	ND	ND	ND.	ND	ND.	ND
840	n.d.											
841	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2115												
2120	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
2121												
2129												
2132	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2135												
2138	ND											
2139												
2165	ND											
2213												
2217												
2241	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2247												
2265	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15
2284	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2290	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15
2293												
2295												
2297	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2301												
2310												
2311												
2320												
2330	ND											
2347	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2350	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50
2352												
2357												
2358	n.d.											
2363	ND											
2365	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2366	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2370	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2374												
2375												
2378												
2379	ND											
2380												
2382	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0
2386	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2390												
2425												

lab	DO 3	DO 76	DR 1	DR 11	DR 17	DY 1	DY 3	DY 9	DY 39	DY 49	AR 26	BB 26
2446												
2452												
2462	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	
2482												
2492												
2511												
2522	<20.0	<20.0	<20.0	<20.0	<20.0	<20.0	<20.0	<20.0	<20.0	<20.0	NA	NA
2532												
2538	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	
2549												
2560 2561												
2573	ND	ND	ND 	ND 	ND	ND 	ND 	ND 	ND 	ND 	ND 	ND
2590		4.4										
2591		4.1										
2602												
2618												
2643	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2644												
2668	40	40	40				40				40	
2672	<10	<10	<10				<10				<10	
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.
2678	ND	ND	ND			AUD.	AUD.	ND	ND.		AUD.	
2689	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2741	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2743												
2773												
2789												
2798												
2804	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
2812												
2827												
2912												
3116												
3146												
3154												
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176												
3197	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3200												
3209												
3210												
3214	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15		
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.
3233												
3237												
3246	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.		

Other reported allergenic Colorants in sample #20556; results in mg/kg -- continued --

lab	BV 3	DBI 38	DB 6	DR 28	DO 11	DO 149	DY 23	BG 4o	BG 4c	BG 4f	NB
210											
230											
362											
551	N.D.					N.D.	N.D.				
623	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
841	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2115											
2120	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	
2121											
2129											
2132	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2135											
2138	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2139											
2165	ND	ND	ND	ND	ND	ND	ND				
2213											
2217											
2241	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
2247											
2265	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15
2284	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2290	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15

lab	BV 3	DBI 38	DB 6	DR 28	DO 11	DO 149	DY 23	BG 4o	BG 4c	BG 4f	NB
2293											
2295											
2297 2301	<10 										
2310											
2311											
2320	ND.	ND	ND.	ND.	ND.	NID.	ND.	ND.	 ΝΙΔ	 NIA	ND
2330 2347	ND <15	NA <15	NA <15	ND 							
2350	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50	<7.50
2352											
2357 2358	 n d										
2363	n.d. ND										
2365	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2366	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
2370 2374	<15 										
2374											
2378											
2379	ND										
2380 2382	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0
2386	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0
2390											
2425											
2446 2452											
2462		<15	<15	<15	<15	<15	<15				
2482											
2492 2511											
2522	NA	NA	NA	NA	NA	<20.0	<20.0	NA	NA	NA	NA
2532											
2538	ND										
2549 2560											
2561											
2573	ND										
2590 2591											
2602											
2618											
2643	< 5	< 5 									
2644 2668											
2672	<10										
2674	n.d.										
2678 2689	ND										
2741	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
2743											
2773 2789											
2798											
2804	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	
2812											
2827 2912											
3116											
3146											
3154 3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3176											
3197	ND										
3200 3209											
3210											
3214											
3228	N.D.										
3233 3237											
3246	n.d.				n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	

## **APPENDIX 3 Accreditation by ISO17025**

lab	ISO/IEC17025 accr.	Sample intake used (grams)	Sample preparation
210			
230			
362			
551	No	1.0g	Used as received
623	Yes	1 gram	Further Cut
840	Yes	0.5 g	Further Cut
841	Yes	_	Used as received
		0.5g	
2115	No	1 g	Used as received
2120	No	1 g	Further Cut
2121			
2129	Yes	0.5	Used as received
2132	No	0.5g	Used as received
2135	Yes	0.5g	Used as received
2138	Yes	0.5007 g	Further Cut
2139	Yes	1g	Further Cut
2165	Yes	0.5	Used as received
2213			
2217	Yes	0.5	Used as received
2241	Yes	0.5	Used as received
2247	<del></del>		
2265	Yes	0,5g	Further Cut
2284	Yes	0.5g	Used as received
2290		0.59	
2293	 Voo	0.E. grom	Further Cut
2295	Yes	0.5 gram	Further Cut
2297	Yes	0.5g	Used as received
2301	Yes	1 gram	Used as received
2310			
2311			
2320			
2330	Yes	0.5 grams	Further Cut
2347		· ·	
2350	No	1g	Used as received
2352	Yes	1g	Used as received
2357		.9	
2358	Yes	1.0 g	Further Cut
2363	Yes	0.5	Further Cut
2365			
	Yes	0.5g	Further Cut
2366	Yes	0.5g	Used as received
2370	Yes	0.5 g	Further Cut
2374	Yes	1g	Further Cut
2375	Yes	0,66 grams	Further Cut
2378	Yes	1g	Used as received
2379	No	1 grams	Further Cut
2380			
2382	Yes	1g	Used as received
2386	Yes	0,5	Further Cut
2390	Yes	1.00gm	Further Cut
2425		<b>G</b>	
2446	Yes	0,5 g	Used as received
2452		-,- 9	
2462	Yes	0.5g	Further Cut
2482	Yes	0,5	Used as received
2492	Yes	0.25	Used as received
2511		0.20	
2522	No	10	
		1g	Further Cut
2532	 V	0.7	Lload on manifold
2538	Yes	0.7 g	Used as received
2549			
2560			
2561			<del></del>
2573	Yes	0.5g	Used as received
2590			
2591	No	0.5	Used as received
2602	Yes	0,5 g	Used as received
2618		· ·	
2643	Yes	0.5 g	Used as received
2644		<del>J</del>	
2668	<del></del>		
2672		0,5 / 0,25	Used as received
	Yes		
2674	Yes	0.5g	Used as received
2678	 V	0.5~	Fronth on Cost
2689	Yes	0.5g	Further Cut
2741	Yes	0.5	Used as received
2743			

lab	ISO/IEC17025 accr.	Sample intake used (grams)	Sample preparation
2773		·	
2789	Yes	0.5 g	Used as received
2798	Yes	0.5	Used as received
2804	No	0.5	Used as received
2812	Yes	1	
2827			
2912			
3116	Yes	0.5 gram	Used as received
3146		•	
3154	Yes	0,5	Used as received
3172	Yes	2	Further Cut
3176	Yes	0,5 g	Further Cut
3197	Yes	0,5 grams	Further Cut
3200			
3209			
3210			
3214	Yes	0.5 g	Further Cut
3228	Yes		Used as received
3233			
3237	Yes	0,5g	Used as received
3246	Yes	0.2g / 3 mL extraction solution	Used as received

#### **APPENDIX 4**

## Number of participants per country

- 4 labs in BANGLADESH
- 1 lab in BRAZIL
- 1 lab in BULGARIA
- 1 lab in CAMBODIA
- 3 labs in FRANCE
- 11 labs in GERMANY
  - 1 lab in GUATEMALA
- 5 labs in HONG KONG
- 1 lab in HUNGARY
- 9 labs in INDIA
- 2 labs in INDONESIA
- 6 labs in ITALY
- 1 lab in MAURITIUS
- 1 lab in MOROCCO
- 22 labs in P.R. of CHINA
  - 1 lab in PAKISTAN
  - 1 lab in PORTUGAL
- 4 labs in SOUTH KOREA
- 2 labs in SPAIN
- 1 lab in SRI LANKA
- 2 labs in TAIWAN
- 1 lab in THAILAND
- 3 labs in TUNISIA
- 6 labs in TURKEY
- 1 lab in UNITED KINGDOM
- 5 labs in VIETNAM

#### **APPENDIX 5**

#### **Abbreviations**

C = final test result after checking of first reported suspect test result

D(0.01) = outlier in Dixon's outlier test D(0.05) = straggler in Dixon's outlier test G(0.01) = outlier in Grubbs' outlier test G(0.05) = straggler in Grubbs' outlier test

DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test

R(0.01) = outlier in Rosner's outlier test R(0.05) = straggler in Rosner's outlier test

W = test result withdrawn on request of participant ex = test result excluded from statistical evaluation

n.a. = not applicablen.e. = not evaluatedn.d. = not detectedfr. = first reported

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