

**Results of Proficiency Test  
Colorants in textile  
(Allergenic & Carcinogenic)  
February 2016**

**Organised by:** Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## 1 INTRODUCTION

Coloured fabrics, when in contact with human skin, may cause Allergic Contact Dermatitis. Twenty dyestuffs are therefore classified as allergenic. Textiles are not allowed to contain more than 50 mg/kg of the 20 below listed dyes according to Öko-tex Standard 100 edition 01/2012, of which 9 dyes are mentioned in DIN54231:

• C.I. Disperse Blue 1	CASno 2475-45-8	C.I.no 64 500	(in DIN54231)
• C.I. Disperse Blue 3	CASno 2475-46-9	C.I.no 61 505	(in DIN54231)
• C.I. Disperse Blue 7	CASno 3179-90-6	C.I.no 62 500	
• C.I. Disperse Blue 26	CASno 3860-63-7	C.I.no 63 305	
• C.I. Disperse Blue 35	CASno 12222-75-2 (*)		(in DIN54231)
• C.I. Disperse Blue 102	CASno 12222-97-8		
• C.I. Disperse Blue 106	CASno 12223-01-7		(in DIN54231)
• C.I. Disperse Blue 124	CASno 61951-51-7		(in DIN54231)
• C.I. Disperse Brown 1	CASno 23355-64-8		
• C.I. Disperse Orange 1	CASno 2581-69-3	C.I.no 11 080	
• C.I. Disperse Orange 3	CASno 730-40-5	C.I.no 11 005	(in DIN54231)
• C.I. Disperse Orange 37/76	CASno 13301-61-6	C.I.no 11 132	(in DIN54231)
• C.I. Disperse Red 1	CASno 2872-52-8	C.I.no 11 110	(in DIN54231)
• C.I. Disperse Red 11	CASno 2872-48-2	C.I.no 62 015	
• C.I. Disperse Red 17	CASno 3179-89-3	C.I.no 11 210	
• C.I. Disperse Yellow 1	CASno 119-15-3	C.I.no 10 345	
• C.I. Disperse Yellow 3	CASno 2832-40-8	C.I.no 11 855	(in DIN54231)
• C.I. Disperse Yellow 9	CASno 6373-73-5	C.I.no 10 375	
• C.I. Disperse Yellow 39	CASno 12236-29-2		
• C.I. Disperse Yellow 49	CASno 54824-37-2		

\* Disperse Blue 35 consists of a mixture of components, of which the monomethylated 1,8-diamino-4,5-dihydroxyanthraquinone (CASno 56524-77-7) and the dimethylated 1,8-diamino-4,5-dihydroxyanthraquinone (CASno 56524-76-6) are responsible for the sensitizing potency of Disperse Blue 35, see also report iis09A04X of May 2009.

The Öko-tex Standard 100 also lists a number of carcinogenic dyes, like for example:

• C.I. Acid Red 26	CASno 3761-53-3	C.I. 16 150
• C.I. Basic Blue 26	CASno 2580-56-5	
• C.I. Basic Green 4 (oxalate)	CASno 2437-29-8, 18015-76-4	
• C.I. Basic Green 4 (chloride)	CASno 569-64-2	
• C.I. Basic Green 4 (free)	CASno 10309-95-2	
• C.I. Basic Red 9	CASno 569-61-9	C.I. 42 500
• C.I. Basic Violet 3	CASno 548-62-9	
• C.I. Basic Violet 14	CASno 632-99-5	C.I. 42 510
• C.I. Direct Black 38	CASno 1937-37-7	C.I. 30 235
• C.I. Direct Blue 6	CASno 2602-46-2	C.I. 22 610
• C.I. Direct Red 28	CASno 573-58-0	C.I. 22 120

The ban on the above dyes has become a widely publicised 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. In this context and in response to requests from several laboratories, the Institute for Interlaboratory Studies (iis) organises a proficiency test for allergenic dyes in textile in the annual proficiency test program since 2003. The scope was extended with carcinogenic dyes in 2016. In the 2016 interlaboratory study 85 laboratories in 24 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the 2016 proficiency test are presented and discussed. This report is also electronically available through the iis internet site [www.iisnl.com](http://www.iisnl.com).

## **2 SET UP**

The Institute for Interlaboratory Studies in Spijkensisse was the organizer of this proficiency test. It was decided to use 2 different textile samples, one treated with banned allergenic dye-stuffs and the other treated with banned carcinogenic dyestuffs. The samples were prepared by a third party. Analysis for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. The participants were requested to report test results using the indicated units and to report rounded and unrounded results. The unrounded 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 PT 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). This protocol is electronically available through the iis internet site [www.iisnl.com](http://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

Two different bulk materials, prepared by a third party, were used in this proficiency test. The first bulk sample, a polyester fabric dyed with two banned allergenic dyes, was cut in small pieces and homogenized. From this batch, 120 subsamples were prepared of 3 grams each and labelled #16525.

The second bulk sample, another polyester fabric dyed with one banned dye, listed both as carcinogenic and as allergenic dye, was cut in small pieces and homogenized. From this batch 120 subsamples were prepared of 3 grams each and labelled #16526.

The homogeneity of the subsamples #16525 was checked by determination of Disperse Yellow 1 and Disperse Red 17 in accordance with DIN54231:05 on 8 stratified randomly selected samples.

The homogeneity of the subsamples #16526 was checked by determination of Disperse Yellow 3 in accordance with DIN54231:05 on 8 stratified randomly selected samples.

See the following tables for the test results.

	Disperse Yellow 1 in mg/kg	Disperse Red 17 in mg/kg
sample #16525-1	104.2	95.4
sample #16525-2	93.9	86.0
sample #16525-3	97.9	87.4
sample #16525-4	91.5	83.7
sample #16525-5	97.3	88.3
sample #16525-6	104.7	89.6
sample #16525-7	102.6	85.2
sample #16525-8	107.4	93.2

table 1: homogeneity test results on subsamples #16525

	Disperse Yellow 3 in mg/kg
sample #16526-1	92
sample #16526-2	94
sample #16526-3	106
sample #16526-4	98
sample #16526-5	96
sample #16526-6	98
sample #16526-7	104
sample #16526-8	92

table 2: homogeneity test results on subsamples #16526

From the above test results, the repeatabilities were calculated and subsequently compared with the corresponding repeatabilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Disperse Yellow 1 #16525 in mg/kg	Disperse Red 17 #16525 in mg/kg	Disperse Yellow 3 #16526 in mg/kg
r(calc)	16	11	15
Reference method	DIN54231:05	DIN54231:05	DIN54231:05
r(reference)	27	24	26

table 3: repeatabilities of subsamples #16525 and #16526

The repeatabilities of the test results of the determined disperse dyes were all in good agreement with the repeatability mentioned in DIN54231:05. Therefore homogeneity of the subsamples was assumed.

To each of the participating laboratories, one sample #16525 and one sample #16526 were sent on February 10, 2016.

## 2.5 ANALYSES

The participants were asked to determine the concentrations of 20 banned allergenic dyes on sample #16525 and of 14 banned carcinogenic dyes on sample #16526, applying the analysis procedure that is routinely used in the laboratory. To get comparable results a detailed report form, on which the units were prescribed as well as the reference standard and a letter of instructions were prepared and made available on the data entry portal [www.kpmd.co.uk/sgs-iis-cts/](http://www.kpmd.co.uk/sgs-iis-cts/).

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

## 3 RESULTS

During five weeks after sample dispatch, the results of the individual laboratories were gathered via the data entry portal [www.kpmd.co.uk/sgs-iis-cts/](http://www.kpmd.co.uk/sgs-iis-cts/). The original data are tabulated per determination in the appendix 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 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) 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 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. 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) for the Rosner General ESD 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 analysis 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 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. 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  $z_{(\text{target})}$ -scores were calculated according to:

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

The  $z_{(\text{target})}$ -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 whether the reported test result is fit-for-use.

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 problems occurred with the delivery of the samples to a laboratory in Bangladesh. This laboratory received the samples late. Seven participants reported test results after the deadline and five participants did not report any test results at all.

Finally, 80 participants reported 233 numerical results. Observed were 7 outlying test results, which is 3.0% of the numerical 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 to 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

In this section the results are discussed per sample. All test results reported on the textile samples are summarised in appendix 1. As in previous PTs almost all participants reported to have used DIN54231 as test method, only a small number of test details were requested to be reported (see appendix 3).

In DIN54231 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 #16525: This polyester fabric was dyed with two allergenic dyes: Disperse Red 17 and Disperse Yellow 1. The test results reported by the participating laboratories vary strongly (from 59.25 mg/kg – 794.5 mg/kg for Disperse Red 17 and from 70.77 mg/kg – 780.5 mg/kg for Disperse Yellow 1).

For Disperse Red 17, only two statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility of DIN54231:05.

For Disperse Yellow 1, three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility of DIN54231:05.

Only laboratory 2553 would not have rejected this sample for containing too much carcinogenic dyestuffs (acc. to the Öko-tex Std.100 limit of 50 mg/kg). All other laboratories would have rejected this sample.

sample #16526: This polyester fabric was dyed with only one carcinogenic dye: Disperse Yellow 3. The test results reported by the participating laboratories vary strongly (from 49.07 mg/kg – 271.8 mg/kg). However, only two statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility of DIN54231:05.

Only laboratory 551 would not have rejected this sample for containing too much carcinogenic dyestuffs (acc. to the Öko-tex Std.100 limit of 50 mg/kg). All other laboratories would have rejected this sample.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities as declared by the relevant standard method and the reproducibilities as found for the group of participating laboratories. The number of significant results, the average results, the calculated reproducibilities (standard deviation\*2.8) and the target reproducibilities, derived from the official test method DIN54231:05 are compared in the next tables.

Parameter	unit	n	average	2.8 * sd	R (target)
Disperse Red 17	mg/kg	78	214	169	172
Disperse Yellow 1	mg/kg	70	161	108	129

table 4: reproducibilities for sample #16525

Parameter	unit	n	average	2.8 * sd	R (target)
Disperse Yellow 3	mg/kg	78	114	95	92

table 5: reproducibilities for sample #16526

Without further statistical calculations it can be concluded that for the three evaluated banned dyestuffs the group of participating laboratories may not have any difficulties with the analysis. See also the discussion in paragraphs 4.1 and 5.

## 4.3 EVALUATION OF THE PROFICIENCY TEST OF FEBRUARY 2016 WITH PREVIOUS PTS

The uncertainties in the test results of the evaluated dyes in the iis16A03 PT are listed in table 6 and are compared with previous proficiency tests.

	Feb. 2016	March 2015	March 2014	March 2013	March 2012	2011 – 2006	target DIN54321
Disperse Blue 1	n.e.	n.e.	n.e.	n.e.	n.e.	43%	27%
Disperse Blue 3	n.e.	n.e.	n.e.	56%	42%	36 - 51%	27%
Disperse Blue 26	n.e.	n.e.	n.e.	n.e.	68%	47 - 56%	27%
Disperse Blue 35	n.e.	31%	n.e.	n.e.	n.e.	57 - 84%	27%
Disperse Blue 106	n.e.	n.e.	28%	n.e.	n.e.	n.e.	27%
Disperse Brown 1	n.e.	n.e.	33%	n.e.	n.e.	n.e.	27%
Disperse Orange 1	n.e.	42%	n.e.	47%	n.e.	44%	27%
Disperse Orange 3	n.e.	n.e.	31%	n.e.	n.e.	24 – 54%	27%
Disperse Red 1	n.e.	n.e.	n.e.	n.e.	n.e.	36 - 63%	27%
Disperse Red 11	n.e.	41%	n.e.	n.e.	65%	45 - 56%	27%
Disperse Red 17	28%	33%	n.e.	n.e.	n.e.	n.e.	27%
Disperse Yellow 1	24%	n.e.	n.e.	n.e.	n.e.	n.e.	27%
Disperse Yellow 3	30%	n.e.	n.e.	29%	n.e.	28%	27%
Disperse Yellow 9	n.e.	n.e.	n.e.	n.e.	n.e.	31%	27%
Disperse Yellow 49	n.e.	n.e.	n.e.	n.e.	n.e.	54%	27%

table 6: development of uncertainties over the last years

From the above table it is clear that for Disperse Red 17 investigated in this PT, a small quality improvement is observed. Disperse Yellow 1 was not evaluated before and the performance for Disperse Yellow 3 is in agreement with previous findings.

## 5 DISCUSSION

From the reported details (see appendix 3), it is clear that the majority of the participants (85-88%) purchased the dyes for calibration as powder and dissolves the powder to prepare the stock solutions.

About half of the laboratories (45-48%) will check the stock solutions by measurement and 31-39% of the laboratories checks visually whether all powder is dissolved. The stock solutions are kept cool (about 4-10°C) by 40-42% of the participants, while another 43-45% stores the stock solutions at very low temperatures (<4°C) and 8% of the laboratories keeps the stock solutions at room temperature.

In previous PT iis15A03, it was suspected that storage of the stock solution(s) at very low temperatures, and subsequently precipitation of the dye from the solution, might be a possible cause for deviating test results. However, the results in this PT do not give any proof for this assumption.

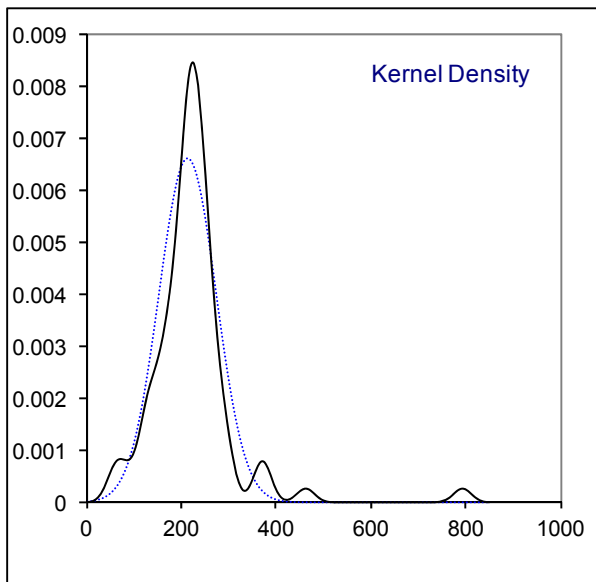
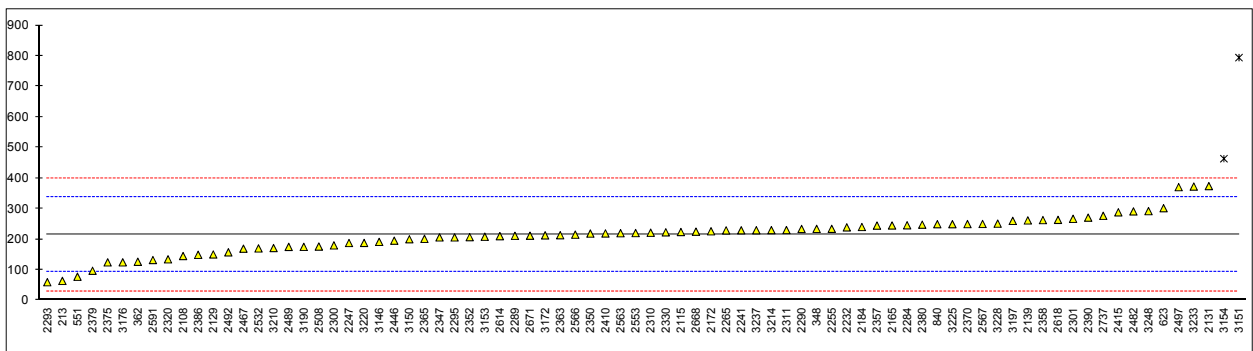
**APPENDIX 1**

Determination of Disperse Red 17 (CASno.3179-89-3) in sample #16525; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	DIN54231	63.5		-2.46	
348	DIN54231	233.96		0.32	
362	DIN54231	126.3		-1.44	
551	DIN54231	77.29346		-2.23	
623	DIN54231	301.56		1.42	
840	DIN54231	249.21		0.57	
2108	DIN54231	144.97		-1.13	
2115	DIN54231	223.68		0.15	
2129	DIN54231	150.2		-1.05	
2131	In house	374		2.60	
2139	DIN54231	261.6		0.77	
2165	DIN54231	245		0.50	
2172	DIN54231	226.1		0.19	
2184	DIN54231	240		0.42	
2232	DIN54231	238.87	C	0.40	first reported 538.87
2241	DIN54231	229.3		0.24	
2247	DIN54231	187.62		-0.44	
2255	DIN54231	234.1		0.32	
2265	DIN54231	228.973		0.24	
2284	DIN54231	245.7		0.51	
2289	DIN54231	211.2		-0.05	
2290	DIN54231	233.63		0.31	
2293	DIN54231	59.25		-2.53	
2295	DIN54231	206		-0.14	
2300	INH-11	179.9		-0.56	
2301	DIN54231	266.94	C	0.85	first reported 533.88
2310	DIN54231	221.3		0.11	
2311	DIN54231	230.1		0.25	
2320	DIN54231	134.2194		-1.31	
2330	DIN54231	222.7		0.13	
2347	DIN54231	206		-0.14	
2350	DIN54231	218.6		0.07	
2352	DIN54231	206.9		-0.12	
2357	DIN54231	244.7		0.49	
2358	DIN54231	262.59		0.78	
2363	DIN54231	213.2		-0.02	
2365	DIN54231	201.3		-0.21	
2370	DIN54231	249.5		0.57	
2375	DIN54231	124.22		-1.47	
2379	DIN54231	96.80		-1.92	
2380	DIN54231	247.6		0.54	
2386	DIN54231	149		-1.07	
2390	DIN54231	270.70685		0.92	
2410	DIN54231	219.01		0.07	
2415	DIN54231	288.18		1.20	
2446	DIN54231	195.30		-0.31	
2467	In house	169.09		-0.74	
2482	DIN54231	291.09		1.25	
2489	DIN54231	175		-0.64	
2492	DIN54231	157.2		-0.93	
2497	DIN54231	370.60	C	2.54	first reported 535.52
2508	DIN54231	176.07		-0.63	
2532	DIN54231	170		-0.72	
2538		----		----	
2546		----		----	
2553	DIN54231	220.23		0.09	
2563	DIN54231	219.8		0.09	
2566	DIN54231	215		0.01	
2567	DIN54231	250.0		0.58	
2590		----		----	
2591	DIN54231	131.85		-1.35	
2614	DIN54231	210.1		-0.07	
2618	DIN54231	263.40		0.80	
2644		----		----	
2668	DIN54231	224.76		0.17	
2671	DIN54231	211.5		-0.05	
2723		----		----	
2737	DIN54231	276.69		1.01	
3146	DIN54231	191.3		-0.38	
3150	DIN54231	200.0		-0.24	
3151	DIN54231	794.5	C,R(0.01)	9.45	first reported 637.5

3153	DIN54231	208.13		-0.10
3154	DIN54231	463.64	R(0.01)	4.06
3172	DIN54231	212.72		-0.03
3176	DIN54231	124.463		-1.47
3190	DIN54231	175.04		-0.64
3197	DIN54231	260		0.74
3210	DIN54231	171.243		-0.70
3214	DIN54231	229.88		0.25
3220	DIN54231	188.0		-0.43
3225	DIN54231	249.327		0.57
3228	DIN54231	251		0.60
3233	DIN54231	372.46		2.57
3237	DIN54231	229.47		0.24
3248	DIN54231	292.01		1.26

normality suspect  
n 78  
outliers 2  
mean (n) 214.464  
st.dev. (n) 60.3823  
R(calc.) 169.070  
R(DIN54231:05) 171.863

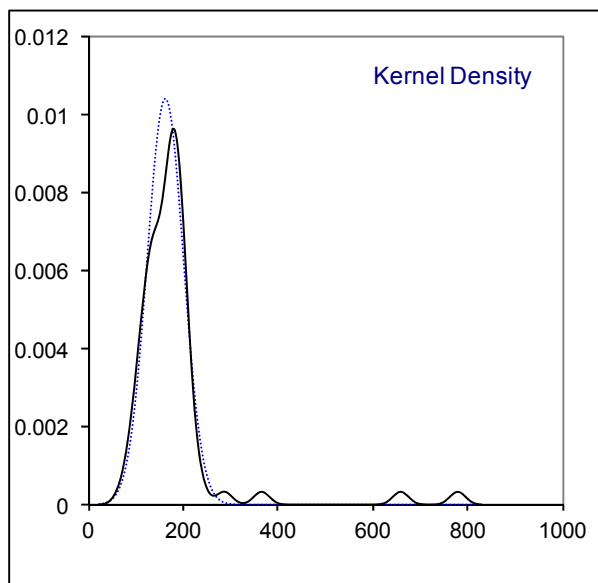
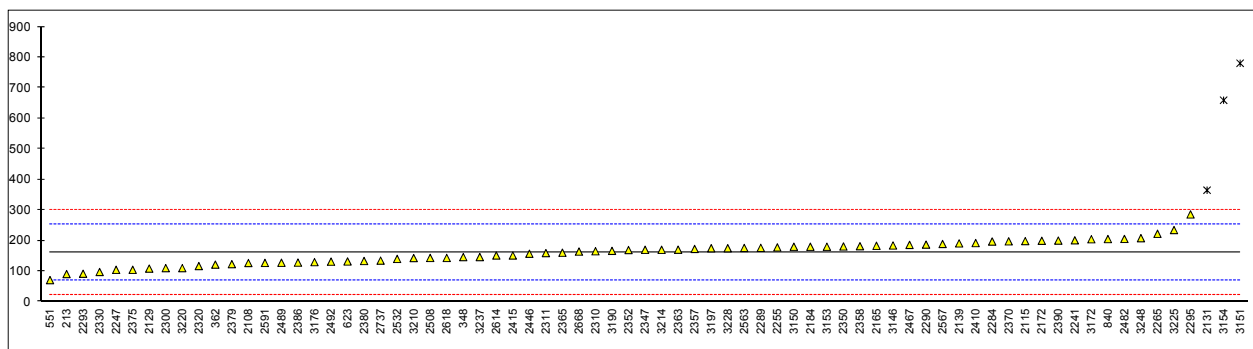


## Determination of Disperse Yellow 1 (CASno.119-15-3) in sample #16525; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	DIN54231	90.5		-1.53	
348	DIN54231	146.12		-0.32	
362	DIN54231	121.4		-0.86	
551	DIN54231	70.76784		-1.96	
623	DIN54231	131.80		-0.63	
840	DIN54231	205.14		0.96	
2108	DIN54231	126.69		-0.74	
2115	DIN54231	198.42		0.82	
2129	DIN54231	108.4		-1.14	
2131	In house	365	R(0.01)	4.43	
2139	DIN54231	191.0		0.65	
2165	DIN54231	183		0.48	
2172	DIN54231	199.4		0.84	
2184	DIN54231	180		0.42	
2232		----		----	
2241	DIN54231	201.0		0.87	
2247	DIN54231	104.37		-1.23	
2255	DIN54231	177.8		0.37	
2265	DIN54231	222.276		1.33	
2284	DIN54231	197.0		0.78	
2289	DIN54231	176.3		0.34	
2290	DIN54231	186.92		0.57	
2293	DIN54231	91.5		-1.51	
2295	DIN54231	285.7		2.71	
2300	INH-11	110		-1.10	
2301		----		----	
2310	DIN54231	165.2		0.09	
2311	DIN54231	159.1		-0.04	
2320	DIN54231	116.6321		-0.96	
2330	DIN54231	97.3		-1.38	
2347	DIN54231	170		0.20	
2350	DIN54231	180.8		0.43	
2352	DIN54231	169.2		0.18	
2357	DIN54231	172.8		0.26	
2358	DIN54231	181.59		0.45	
2363	DIN54231	170.3		0.20	
2365	DIN54231	160.4		-0.01	
2370	DIN54231	197.6		0.80	
2375	DIN54231	104.53		-1.22	
2379	DIN54231	123.04		-0.82	
2380	DIN54231	133.3		-0.60	
2386	DIN54231	128		-0.71	
2390	DIN54231	200.08879		0.85	
2410	DIN54231	192.05		0.68	
2415	DIN54231	151.29		-0.21	
2446	DIN54231	157.01		-0.08	
2467	In house	186.00		0.55	
2482	DIN54231	205.45		0.97	
2489	DIN54231	127.5		-0.72	
2492	DIN54231	131.1		-0.65	
2497		----		----	
2508	DIN54231	143.29		-0.38	
2532	DIN54231	140		-0.45	
2538	§64LFGBB82.02-10	detected		----	see appendix 2
2546		----		----	
2553	DIN54231	ND		----	false negative test result?
2563	DIN54231	176.0		0.33	
2566		----		----	
2567	DIN54231	189.0		0.61	
2590		----		----	
2591	DIN54231	126.90		-0.74	
2614	DIN54231	151.2	C	-0.21	first reported <15
2618	DIN54231	143.33		-0.38	
2644		----		----	
2668	DIN54231	164.11		0.07	
2671		----		----	
2723		----		----	
2737	DIN54231	134.52		-0.57	
3146	DIN54231	184.2		0.51	
3150	DIN54231	179.99		0.42	
3151	DIN54231	780.5	C,R(0.01)	13.46	first reported 642.9
3153	DIN54231	180.00		0.42	
3154	DIN54231	659.58	R(0.01)	10.83	
3172	DIN54231	204.51		0.95	

3176	DIN54231	129.710	-0.68
3190	DIN54231	166.50	0.12
3197	DIN54231	175	0.31
3210	DIN54231	142.966	-0.39
3214	DIN54231	170.04	0.20
3220	DIN54231	110.0	-1.10
3225	DIN54231	234.524	1.60
3228	DIN54231	175	0.31
3233		----	----
3237	DIN54231	146.21	-0.32
3248	DIN54231	208.01	1.02

normality OK  
 n 70  
 outliers 3  
 mean (n) 160.868  
 st.dev. (n) 38.4514  
 R(calc.) 107.664  
 R(DIN54231:05) 128.914



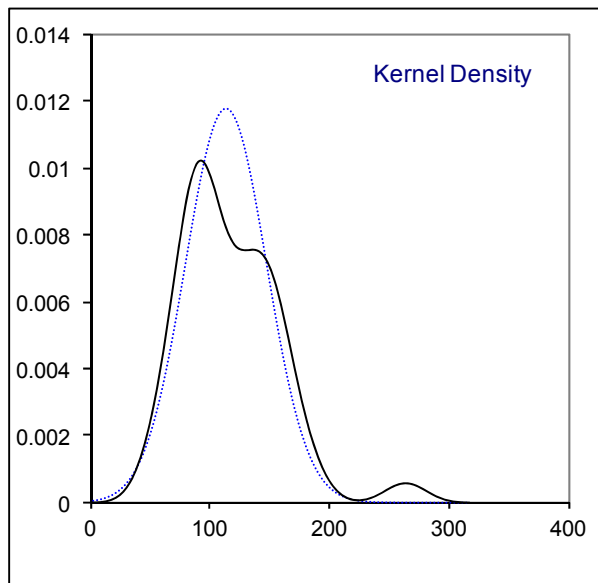
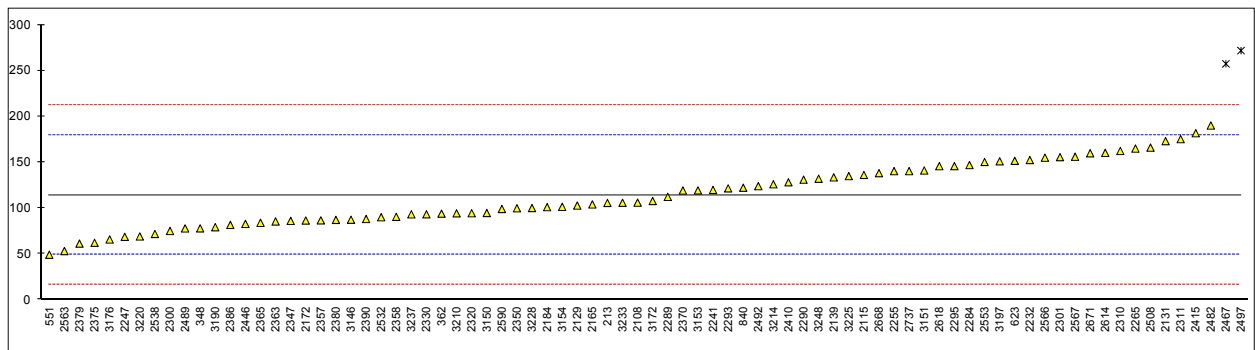
## Determination of Disperse Yellow 3 (CASno.2832-40-8) in sample #16526; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	DIN54231	105.6		-0.26	
348	DIN54231	77.85		-1.11	
362	DIN54231	93.9		-0.62	
551	DIN54231	49.07302		-1.99	
623	DIN54231	151.52		1.14	
840	DIN54231	122.20		0.25	
2108	DIN54231	105.93		-0.25	
2115	DIN54231	136.19		0.67	
2129	DIN54231	102.7		-0.35	
2131	In house	173		1.80	
2139	DIN54231	133.5		0.59	
2165	DIN54231	104		-0.31	
2172	DIN54231	86.40		-0.85	
2184	DIN54231	101		-0.40	
2232	DIN54231	152.44		1.17	
2241	DIN54231	119.8		0.17	
2247	DIN54231	68.62		-1.39	
2255	DIN54231	140.3		0.80	
2265	DIN54231	164.9099		1.55	
2284	DIN54231	146.9		1.00	
2289	DIN54231	112.2		-0.06	
2290	DIN54231	131.01		0.52	
2293	DIN54231	121.5		0.22	
2295	DIN54231	145.8		0.97	
2300	INH-11	75		-1.20	
2301	DIN54231	155.57		1.27	
2310	DIN54231	162.3		1.47	
2311	DIN54231	175.2		1.87	
2320	DIN54231	94.375125		-0.61	
2330	DIN54231	93.2		-0.64	
2347	DIN54231	86		-0.86	
2350	DIN54231	99.75		-0.44	
2352		----		----	
2357	DIN54231	86.6		-0.84	
2358	DIN54231	90.46		-0.73	
2363	DIN54231	85.4		-0.88	
2365	DIN54231	83.9		-0.93	
2370	DIN54231	119.2		0.15	
2375	DIN54231	62.12		-1.59	
2379	DIN54231	61.25		-1.62	
2380	DIN54231	87.1		-0.83	
2386	DIN54231	81.6		-1.00	
2390	DIN54231	88.0556		-0.80	
2410	DIN54231	128.14		0.43	
2415	DIN54231	181.66		2.07	
2446	DIN54231	82.68		-0.96	
2467	In house	257.39	C,R(0.01)	4.38	
2482	DIN54231	190.07		2.32	
2489	DIN54231	77.8		-1.11	
2492	DIN54231	123.9		0.30	
2497	DIN54231	271.80	C,R(0.01)	4.82	
2508	DIN54231	165.95		1.58	
2532	DIN54231	90		-0.74	
2538	§64LFGBB82.02-10	71.74		-1.30	
2546		----		----	
2553	DIN54231	150.34		1.11	
2563	DIN54231	53.1		-1.87	
2566	DIN54231	155		1.25	
2567	DIN54231	156.0		1.28	
2590	DIN54231	99.01		-0.46	
2591		----		----	
2614	DIN54231	160.23		1.41	
2618	DIN54231	145.73		0.97	
2644		----		----	
2668	DIN54231	138.11		0.73	
2671	DIN54231	159.8		1.40	
2723		----		----	
2737	DIN54231	140.32		0.80	
3146	DIN54231	87.3		-0.82	
3150	DIN54231	94.6		-0.60	
3151	DIN54231	141		0.82	
3153	DIN54231	119.25		0.16	
3154	DIN54231	101.39		-0.39	
3172	DIN54231	107.71		-0.20	



3176	DIN54231	65.750	-1.48
3190	DIN54231	79.12	-1.07
3197	DIN54231	151	1.13
3210	DIN54231	94.259	-0.61
3214	DIN54231	125.95	0.36
3220	DIN54231	69.0	-1.38
3225	DIN54231	134.853	0.63
3228	DIN54231	100	-0.43
3233	DIN54231	105.76	-0.26
3237	DIN54231	93.15	-0.64
3248	DIN54231	132.01	0.55

normality OK  
n 78  
outliers 2  
mean (n) 114.168  
st.dev. (n) 33.9317  
R(calc.) 95.009  
R(DIN54231:05) 91.490



Determination of Disperse Blue 1 (CASno. 2475-45-8), Disperse Blue 3 (CASno. 2475-46-9), Disperse Blue 7 (CASno. 3179-90-6), Disperse Blue 26 (CASno. 3860-63-7) and Disperse Blue 35 (CASno. 12222-75-2) in sample #16525; results in mg/kg

Lab	method	Disperse Blue 1	Disperse Blue 3	Disperse Blue 7	Disperse Blue 26	Disperse Blue 35
213		----	----	----	----	----
348	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
362		----	----	----	----	----
551		----	----	----	----	----
623	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
840	DIN54231	ND	ND	ND	ND	ND
2108		----	----	----	----	----
2115		----	----	----	----	----
2129		----	----	----	----	----
2131		----	----	----	----	----
2139		----	----	----	----	----
2165	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2172	DIN54231	<15	<15	<15	<15	<15
2184	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2232		----	----	----	----	----
2241		----	----	----	----	----
2247		----	----	----	----	----
2255	DIN54231	nd	nd	nd	nd	nd
2265		----	----	----	----	----
2284		----	----	----	----	----
2289	DIN54231	ND	ND	ND	ND	ND
2290	DIN54231	<15	<15	<15	<15	<15
2293		----	----	----	----	----
2295	DIN54231	ND	ND	ND	ND	ND
2300	INH-11	ND	ND	ND	ND	ND
2301		----	----	----	----	----
2310	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2311	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2320	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	DIN54231	<15	<15	<15	<15	----
2347	DIN54231	<15	<15	<15	<15	<15
2350	DIN54231	<15	<15	<15	<15	<15
2352		----	----	----	----	----
2357	DIN54231	<15	<15	<15	<15	<15
2358	DIN54231	N.D.	N.D.	N.D.	N.D.	N.D.
2363	DIN54231	<15	<15	<15	<15	<15
2365	DIN54231	<15	<15	<15	<15	<15
2370	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2375		----	----	----	----	----
2379		----	----	----	----	----
2380		----	----	----	----	----
2386	DIN54231	<5	<5	<5	<5	<5
2390	DIN54231	N.D	N.D	N.D	N.D	N.D
2410	DIN54231	<20	<20	<20	<20	<20
2415	DIN54231	ND	ND	ND	ND	ND
2446		----	----	----	----	----
2467		----	----	----	----	----
2482		----	----	----	----	----
2489	DIN54231	ND	ND	ND	ND	ND
2492		----	----	----	----	----
2497		----	----	----	----	----
2508		----	----	----	----	----
2532	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2538	§64LFGBB82.02-10	n.d.	n.d.	n.d.	n.d.	n.d.
2546		----	----	----	----	----
2553	DIN54231	ND	ND	ND	ND	ND
2563		----	----	----	----	----
2566		----	----	----	----	----
2567	DIN54231	ND	ND	ND	ND	ND
2590	DIN54231	< L.O.Q.	----	----	----	< L.O.Q.
2591	DIN54231	<1.0	<1.0	<1.0	<1.0	<1.0
2614	DIN54231	<15	<15	<15	<15	<15
2618		----	----	----	----	----
2644		----	----	----	----	----
2668	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2671		----	----	----	----	----
2723		----	----	----	----	----
2737		----	----	----	----	----
3146		----	----	----	----	----
3150		----	----	----	----	----
3151		----	----	----	----	----

3153	DIN54231	<15.0	<15.0	<15.0	<15.0	<15.0
3154		----	----	----	----	----
3172	DIN54231	<10.00	<10.00	<10.00	<10.00	<10.00
3176		----	----	----	----	----
3190		----	----	----	----	----
3197	DIN54231	ND	ND	ND	ND	ND
3210	DIN54231	<50	<50	<50	<50	<50
3214	DIN54231	N.D	N.D	N.D	N.D	N.D
3220		----	----	----	----	----
3225	DIN54231	<7.5	<7.5	<7.5	<7.5	<7.5
3228	DIN54231	Not detected	Not detected	Not detected	Not detected	Not detected
3233		----	----	----	----	----
3237		----	----	----	----	----
3248		----	----	----	----	----
	normality	n.a.	n.a.	n.a.	n.a.	n.a.
	n	41	41	41	41	41
	outliers	0	0	0	0	0
	mean (n)	not detected	not detected	not detected	not detected	not detected
	st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.
	R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.
	R(DIN54231:05)	n.a.	n.a.	n.a.	n.a.	n.a.

Determination of Disperse Blue 35a (CASno. 56524-77-7), Disperse Blue 35b (CASno. 56524-76-6), Disperse Blue 102 (CASno. 12222-97-8), Disperse Blue 106 (CASno. 12223-01-7) and Disperse Blue 124 (CASno. 61951-51-7) in sample #16525; results in mg/kg

Lab	Method	Disperse Blue 35a	Disperse Blue 35b	Disperse Blue 102	Disperse Blue 106	Disperse Blue 124
213		----	----	----	----	----
348	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
362		----	----	----	----	----
551		----	----	----	----	----
623	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
840	DIN54231	ND	ND	ND	ND	ND
2108		----	----	----	----	----
2115		----	----	----	----	----
2129		----	----	----	----	----
2131		----	----	----	----	----
2139		----	----	----	----	----
2165	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2172	DIN54231	<15	<15	<15	<15	<15
2184	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2232		----	----	----	----	----
2241		----	----	----	----	----
2247		----	----	----	----	----
2255	DIN54231	nd	nd	nd	nd	nd
2265		----	----	----	----	----
2284		----	----	----	----	----
2289	DIN54231	ND	ND	ND	ND	ND
2290	DIN54231	<15	<15	<15	<15	<15
2293		----	----	----	----	----
2295	DIN54231	ND	ND	ND	ND	ND
2300	INH-11	ND	ND	ND	ND	ND
2301		----	----	----	----	----
2310	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2311	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2320	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	DIN54231	<15	<15	<15	<15	<15
2347	DIN54231	<15	<15	<15	<15	<15
2350	DIN54231	<15	<15	<15	<15	<15
2352		----	----	----	----	----
2357	DIN54231	<15	<15	<15	<15	<15
2358	DIN54231	N.D.	N.D.	N.D.	N.D.	N.D.
2363	DIN54231	<15	<15	<15	<15	<15
2365	DIN54231	<15	<15	<15	<15	<15
2370	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2375		----	----	----	----	----
2379		----	----	----	----	----
2380		----	----	----	----	----
2386	DIN54231	<5	<5	<5	<5	<5
2390	DIN54231	N.D	N.D	N.D	N.D	N.D
2410	DIN54231	<20	<20	<20	<20	<20
2415	DIN54231	ND	ND	----	ND	ND
2446		----	----	----	----	----
2467		----	----	----	----	----
2482		----	----	----	----	----
2489	DIN54231	ND	ND	ND	ND	ND
2492		----	----	----	----	----
2497		----	----	----	----	----
2508		----	----	----	----	----
2532	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2538	§64LFGBB82.02-10	n.d.	n.d.	n.d.	n.d.	n.d.
2546		----	----	----	----	----
2553	DIN54231	ND	ND	ND	ND	ND
2563		----	----	----	----	----
2566		----	----	----	----	----
2567		----	----	ND	ND	ND
2590		----	----	----	< L.O.Q.	< L.O.Q.
2591	DIN54231	<1.0	<1.0	<1.0	<1.0	<1.0
2614	DIN54231	<15	<15	<15	<15	<15
2618		----	----	----	----	----
2644		----	----	----	----	----
2668		----	----	n.d.	n.d.	n.d.
2671		----	----	----	----	----
2723		----	----	----	----	----
2737		----	----	----	----	----
3146		----	----	----	----	----
3150		----	----	----	----	----

3151		----	----	----	----	----
3153	DIN54231	<15.0	<15.0	<15.0	<15.0	<15.0
3154		----	----	----	----	----
3172	DIN54231	<10.00	<10.00	<10.00	<10.00	<10.00
3176		----	----	----	----	----
3190		----	----	----	----	----
3197	DIN54231	ND	ND	ND	ND	ND
3210		----	----	<50	<50	<50
3214	DIN54231	N.D	N.D	N.D	N.D	N.D
3220		----	----	----	----	----
3225	DIN54231	<7.5	<7.5	<7.5	<7.5	<7.5
3228	DIN54231	Not detected	Not detected	Not detected	Not detected	Not detected
3233		----	----	----	----	----
3237		----	----	----	----	----
3248		----	----	----	----	----
normality	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
n	41	41	41	41	41	41
outliers	0	0	0	0	0	0
mean (n)	not detected	not detected	not detected	not detected	not detected	not detected
st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
R(DIN54231:05)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

Determination of Disperse Brown 1 (CASno. 23355-64-8), Disperse Orange 1 (CASno. 2581-69-3), Disperse Orange 3 (CASno. 730-40-5), Disperse Orange 76 = 37 (CASno. 13301-61-6) and Disperse Red 1 (CASno. 2872-52-8) in sample #16525; results in mg/kg

Lab	Method	Disperse Brown 1	Disperse Orange 1	Disperse Orange 3	Disperse Orange 76 = 37	Disperse Red 1
213		----	----	----	----	----
348	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
362		----	----	----	----	----
551		----	----	----	----	----
623	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
840	DIN54231	ND	ND	ND	ND	ND
2108		----	----	----	----	----
2115		----	----	----	----	----
2129		----	----	----	----	----
2131		----	----	----	----	----
2139		----	----	----	----	----
2165	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2172	DIN54231	<15	<15	<15	<15	<15
2184	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2232		----	----	----	----	----
2241		----	----	----	----	----
2247		----	----	----	----	----
2255	DIN54231	nd	nd	nd	nd	nd
2265		----	----	----	----	----
2284		----	----	----	----	----
2289	DIN54231	ND	ND	ND	ND	ND
2290	DIN54231	<15	<15	<15	<15	<15
2293		----	----	----	----	----
2295	DIN54231	ND	ND	ND	ND	ND
2300	INH-11	50.1	ND	ND	ND	ND
2301		----	----	----	----	----
2310	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2311	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2320	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	DIN54231	<15	<15	<15	<15	<15
2347	DIN54231	<15	<15	<15	<15	<15
2350	DIN54231	<15	<15	<15	<15	<15
2352		----	----	----	----	----
2357	DIN54231	<15	<15	<15	<15	<15
2358	DIN54231	N.D.	N.D.	N.D.	N.D.	N.D.
2363	DIN54231	<15	<15	<15	<15	<15
2365	DIN54231	<15	<15	<15	<15	<15
2370	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2375		----	----	----	----	----
2379		----	----	----	----	----
2380		----	----	----	----	----
2386	DIN54231	<5	<5	<5	<5	<5
2390	DIN54231	N.D	N.D	N.D	N.D	N.D
2410	DIN54231	<20	<20	<20	<20	<20
2415	DIN54231	ND	ND	ND	ND	ND
2446		----	----	----	----	----
2467		----	----	----	----	----
2482		----	----	----	----	----
2489	DIN54231	ND	ND	ND	ND	ND
2492		----	----	----	----	----
2497		----	----	----	----	----
2508		----	----	----	----	----
2532	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2538	§64LFGBB82.02-10	n.d.	n.d.	n.d.	n.d.	n.d.
2546		----	----	----	----	----
2553	DIN54231	ND	ND	ND	ND	ND
2563		----	----	----	----	----
2566		----	----	----	----	----
2567	DIN54231	ND	ND	ND	ND	ND
2590		----	----	< L.O.Q.	< L.O.Q.	< L.O.Q.
2591	DIN54231	<1.0	<1.0	<1.0	<1.0	<1.0
2614	DIN54231	<15	<15	<15	<15	<15
2618		----	----	----	----	----
2644		----	----	----	----	----
2668	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2671		----	----	----	----	----
2723		----	----	----	----	----
2737		----	----	----	----	----
3146		----	----	----	----	----
3150		----	----	----	----	----

3151		----	----	----	----	----
3153	DIN54231	<15.0	<15.0	<15.0	<15.0	<15.0
3154		----	----	----	----	----
3172	DIN54231	<10.00	<10.00	<10.00	<10.00	<10.00
3176		----	----	----	----	----
3190		----	----	----	----	----
3197	DIN54231	ND	ND	ND	ND	ND
3210	DIN54231	<50	<50	<50	<50	<50
3214	DIN54231	N.D	N.D	N.D	N.D	N.D
3220		----	----	----	----	----
3225	DIN54231	<7.5	<7.5	<7.5	<7.5	<7.5
3228	DIN54231	Not detected	Not detected	Not detected	Not detected	Not detected
3233		----	----	----	----	----
3237		----	----	----	----	----
3248		----	----	----	----	----
normality	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
n	41	41	41	41	41	41
outliers	0	0	0	0	0	0
mean (n)	not detected	not detected	not detected	not detected	not detected	not detected
st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
R(DIN54231:05)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

Determination of Disperse Red 11 (CASno. 2872-48-2), Disperse Yellow 3 (CASno. 2832-40-8), Disperse Yellow 9 (CASno. 6373-73-5), Disperse Yellow 39 (CASno. 12236-29-2) and Disperse Yellow 49 (CASno. 54824-37-2) in sample #16525; results in mg/kg

Lab	method	Disperse Red 11	Disperse Yellow 3	Disperse Yellow 9	Disperse Yellow 39	Disperse Yellow 49
213		----	----	----	----	----
348	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
362		----	----	----	----	----
551		----	----	----	----	----
623	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
840	DIN54231	ND	ND	ND	ND	ND
2108		----	----	----	----	----
2115		----	----	----	----	----
2129		----	----	----	----	----
2131		----	----	----	----	----
2139		----	----	----	----	----
2165	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2172	DIN54231	<15	<15	<15	<15	<15
2184	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2232		----	----	----	----	----
2241		----	----	----	----	----
2247		----	nd	nd	nd	nd
2255	DIN54231	nd	nd	nd	nd	nd
2265		----	----	----	----	----
2284		----	----	----	----	----
2289	DIN54231	ND	ND	ND	ND	ND
2290	DIN54231	<15	<15	<15	<15	<15
2293		----	----	----	----	----
2295	DIN54231	ND	ND	ND	ND	ND
2300	INH-11	ND	110	ND	ND	ND
2301		----	----	----	----	----
2310	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2311	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2320	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	DIN54231	<15	<15	<15	<15	<15
2347	DIN54231	<15	<15	<15	<15	<15
2350	DIN54231	<15	<15	<15	<15	<15
2352		----	----	----	----	----
2357	DIN54231	<15	<15	<15	<15	<15
2358	DIN54231	N.D.	N.D.	N.D.	N.D.	N.D.
2363	DIN54231	<15	<15	<15	<15	<15
2365	DIN54231	<15	<15	<15	<15	<15
2370	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2375		----	----	----	----	----
2379		----	----	----	----	----
2380		----	----	----	----	----
2386	DIN54231	<5	<5	<5	<5	<5
2390	DIN54231	N.D	N.D	N.D	N.D	N.D
2410	DIN54231	<20	<20	<20	<20	<20
2415	DIN54231	ND	ND	ND	ND	ND
2446		----	----	----	----	----
2467		----	----	----	----	----
2482		----	----	----	----	----
2489	DIN54231	ND	ND	ND	ND	ND
2492		----	----	----	----	----
2497		----	----	----	----	----
2508		----	----	----	----	----
2532	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2538	§64LFGBB82.02-10	n.d.	n.d.	n.d.	n.d.	n.d.
2546		----	----	----	----	----
2553	DIN54231	ND	ND	ND	ND	ND
2563		----	----	----	----	----
2566		----	----	----	----	----
2567	DIN54231	ND	ND	ND	ND	ND
2590		----	< L.O.Q.	----	----	----
2591	DIN54231	<1.0	<1.0	<1.0	<1.0	<1.0
2614	DIN54231	<15	<15	<15	<15	<15
2618		----	----	----	----	----
2644		----	----	----	----	----
2668	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2671		----	----	----	----	----
2723		----	----	----	----	----
2737		----	----	----	----	----
3146		----	----	----	----	----
3150		----	----	----	----	----



3151		----	----	----	----	----
3153	DIN54231	<15.0	<15.0	<15.0	<15.0	<15.0
3154		----	----	----	----	----
3172	DIN54231	<10.00	<10.00	<10.00	<10.00	<10.00
3176		----	----	----	----	----
3190		----	----	----	----	----
3197	DIN54231	ND	ND	ND	ND	ND
3210	DIN54231	<50	<50	<50	<50	<50
3214	DIN54231	N.D	N.D	N.D	N.D	N.D
3220		----	----	----	----	----
3225	DIN54231	<7.5	<7.5	<7.5	<7.5	<7.5
3228	DIN54231	Not detected	Not detected	Not detected	Not detected	Not detected
3233		----	----	----	----	----
3237		----	----	----	----	----
3248		----	----	----	----	----
	normality	n.a.	n.a.	n.a.	n.a.	n.a.
	n	41	41	41	41	41
	outliers	0	0	0	0	0
	mean (n)	not detected	not detected	not detected	not detected	not detected
	st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.
	R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.
	R(DIN54231:05)	n.a.	n.a.	n.a.	n.a.	n.a.

Determination of Acid Red 26 (CASno.3761-53-3), Basic Blue 26 (with  $\geq 0.1\%$  Michler's ketone or base) (CASno.2580-56-5), Basic Green 4 (oxalate) (CASno.2437-29-8), Basic Green 4 (chloride) (CASno.569-64-2) and Basic Green 4 (free) (CASno.10309-95-2) in sample #16526; results in mg/kg

Lab	method	Acid Red 26	Basic Blue 26	Basic Green 4 (oxalate)	Basic Green 4 (chloride)	Basic Green 4 (free)
213		----	----	----	----	----
348	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
362		----	----	----	----	----
551		----	----	----	----	----
623	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
840	DIN54231	ND	ND	ND	ND	ND
2108		----	----	----	----	----
2115		----	----	----	----	----
2129		----	----	----	----	----
2131		----	----	----	----	----
2139		----	----	----	----	----
2165	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2172	DIN54231	<15	<15	<15	<15	<15
2184	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2232		----	----	----	----	----
2241		----	----	----	----	----
2247	DIN54231	nd	nd	nd	----	nd
2255	DIN54231	nd	nd	nd	nd	nd
2265		----	----	----	----	----
2284		----	----	----	----	----
2289	DIN54231	ND	ND	ND	ND	ND
2290	DIN54231	<15	<15	<15	<15	<15
2293		----	----	----	----	----
2295	DIN54231	ND	ND	----	ND	ND
2300	INH-11	nd	nd	nd	nd	nd
2301		----	----	----	----	----
2310	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2311	DIN54231	Not Detected	Not Detected	----	----	----
2320	DIN54231	Not Detected	Not Detected	Not Tested	Not Tested	Not Tested
2330	DIN54231	<15	----	----	----	----
2347	DIN54231	<15	<15	<15	<15	<15
2350	DIN54231	<15	<15	<15	<15	<15
2352		----	----	----	----	----
2357	DIN54231	<15	<15	----	----	----
2358	DIN54231	N.D.	N.D.	N.D.	N.D.	N.D.
2363	DIN54231	<15	<15	<15	<15	<15
2365	DIN54231	<15	<15	<15	<15	<15
2370	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2375		----	----	----	----	----
2379	DIN54231	Not detected	Not detected	Not detected	Not detected	Not detected
2380		----	----	----	----	----
2386	DIN54231	<5	<5	<5	<5	<5
2390	DIN54231	N.D	N.D	N.D	N.D	N.D
2410	DIN54231	<20	<20	<20	<20	<20
2415	DIN54231	ND	----	----	----	----
2446		----	----	----	----	----
2467		----	----	----	----	----
2482		----	----	----	----	----
2489	DIN54231	ND	ND	ND	ND	ND
2492		----	----	----	----	----
2497		----	----	----	----	----
2508		----	----	----	----	----
2532	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2538	§64LFGBB82.02-10	n.d.	n.d.	n.d.	n.d.	n.d.
2546		----	----	----	----	----
2553	DIN54231	ND	ND	ND	ND	ND
2563		----	----	----	----	----
2566		----	----	----	----	----
2567	DIN54231	ND	----	----	----	----
2590		----	----	----	----	----
2591		----	----	----	----	----
2614	DIN54231	<15	<15	<15	<15	<15
2618		----	----	----	----	----
2644		----	----	----	----	----
2668	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2671		----	----	----	----	----
2723		----	----	----	----	----
2737		----	----	----	----	----
3146		----	----	----	----	----

3150		----	----	----	----	----
3151		----	----	----	----	----
3153	DIN54231	<15.0	<15.0	<15.0	<15.0	<15.0
3154		----	----	----	----	----
3172	DIN54231	<10.00	<10.00	<10.00	<10.00	<10.00
3176		----	----	----	----	----
3190		----	----	----	----	----
3197	DIN54231	ND	ND	ND	ND	ND
3210	DIN54231	<50	<50	----	----	----
3214	DIN54231	N.D	N.D	N.D	N.D	N.D
3220		----	----	----	----	----
3225	DIN54231	<7.5	<7.5	<7.5	<7.5	<7.5
3228	DIN54231	Not detected	Not detected	Not detected	Not detected	Not detected
3233		----	----	----	----	----
3237		----	----	----	----	----
3248		----	----	----	----	----
normality	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
n	42	42	42	42	42	42
outliers	0	0	0	0	0	0
mean (n)	not detected	not detected	not detected	not detected	not detected	not detected
st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
R(DIN54231:05)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

Determination of Basic Red 9 (CASno.569-61-9), Basic Violet 3 (with  $\geq 0.1\%$  Michler's ketone or base) (CASno.548-62-9) and Basic Violet 14 (CASno.632-99-5) in sample #16526; results in mg/kg

Lab	method	Basic Red 9	Basic Violet 3 (with $\geq 0.1\%$ Michler's ketone or base)	Basic Violet 14
213		----	----	----
348	DIN54231	n.d.	n.d.	n.d.
362		----	----	----
551		----	----	----
623	DIN54231	n.d.	n.d.	n.d.
840	DIN54231	ND	ND	ND
2108		----	----	----
2115		----	----	----
2129		----	----	----
2131		----	----	----
2139		----	----	----
2165	DIN54231	n.d.	n.d.	n.d.
2172	DIN54231	<15	<15	<15
2184	DIN54231	Not Detected	Not Detected	Not Detected
2232		----	----	----
2241		----	----	----
2247	DIN54231	nd	nd	nd
2255	DIN54231	nd	nd	nd
2265		----	----	----
2284		----	----	----
2289	DIN54231	ND	ND	ND
2290	DIN54231	<15	<15	<15
2293		----	----	----
2295	DIN54231	ND	ND	----
2300	INH-11	nd	nd	nd
2301		----	----	----
2310	DIN54231	n.d.	n.d.	n.d.
2311	DIN54231	Not Detected	Not Detected	Not Detected
2320	DIN54231	Not Detected	Not Detected	Not Detected
2330	DIN54231	<15	----	<15
2347	DIN54231	<15	<15	<15
2350	DIN54231	<15	<15	<15
2352		----	----	----
2357	DIN54231	<15	<15	<15
2358	DIN54231	N.D.	N.D.	N.D.
2363	DIN54231	<15	<15	<15
2365	DIN54231	<15	<15	<15
2370	DIN54231	n.d.	n.d.	n.d.
2375		----	----	----
2379	DIN54231	Not detected	Not detected	Not detected
2380		----	----	----
2386	DIN54231	<5	<5	<5
2390	DIN54231	N.D	N.D	N.D
2410	DIN54231	<20	<20	<20
2415	DIN54231	ND	----	ND
2446		----	----	----
2467		----	----	----
2482		----	----	----
2489	DIN54231	ND	ND	ND
2492		----	----	----
2497		----	----	----
2508		----	----	----
2532	DIN54231	Not Detected	Not Detected	Not Detected
2538	§64LFGBB82.02-10	n.d.	n.d.	n.d.
2546		----	----	----
2553	DIN54231	ND	ND	ND
2563		----	----	----
2566		----	----	----
2567	DIN54231	ND	ND	ND
2590	DIN54231	< L.O.Q.	----	< L.O.Q.
2591		----	----	----
2614	DIN54231	<15	<15	<15
2618		----	----	----
2644		----	----	----
2668	DIN54231	n.d.	n.d.	n.d.
2671		----	----	----
2723		----	----	----
2737		----	----	----
3146		----	----	----
3150		----	----	----

3151		----	----	----
3153	DIN54231	<15.0	<15.0	<15.0
3154		----	----	----
3172	DIN54231	<10.00	<10.00	<10.00
3176		----	----	----
3190		----	----	----
3197	DIN54231	ND	ND	ND
3210	DIN54231	<50	<50	----
3214	DIN54231	N.D	N.D	N.D
3220		----	----	----
3225	DIN54231	<7.5	<7.5	<7.5
3228	DIN54231	Not detected	Not detected	Not detected
3233		----	----	----
3237		----	----	----
3248		----	----	----
	normality	n.a.	n.a.	n.a.
	n	42	42	42
	outliers	0	0	0
	mean (n)	not detected	not detected	not detected
	st.dev. (n)	n.a.	n.a.	n.a.
	R(calc.)	n.a.	n.a.	n.a.
	R(DIN54231:05)	n.a.	n.a.	n.a.

Determination of Direct Black 38 (CASno.1937-37-7), Direct Blue 6 (CASno.2602-46-2), Direct Red 28 (CASno.573-58-0), Disperse Blue 1 (CASno.2475-45-8) and Disperse Orange 11 (CASno.82-28-0) in sample #16526; results in mg/kg

Lab	method	Direct Black 38	Direct Blue 6	Direct Red 28	Disperse Blue 1	Disperse Orange 11
213		----	----	----	----	----
348	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
362		----	----	----	----	----
551		----	----	----	----	----
623	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
840	DIN54231	ND	ND	ND	ND	ND
2108		----	----	----	----	----
2115		----	----	----	----	----
2129		----	----	----	----	----
2131		----	----	----	----	----
2139		----	----	----	----	----
2165	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2172	DIN54231	<15	<15	<15	<15	<15
2184	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2232		----	----	----	----	----
2241		----	----	----	----	----
2247	DIN54231	nd	nd	nd	nd	nd
2255	DIN54231	nd	nd	nd	nd	nd
2265		----	----	----	----	----
2284		----	----	----	----	----
2289	DIN54231	ND	ND	ND	ND	ND
2290	DIN54231	<15	<15	<15	<15	<15
2293		----	----	----	----	----
2295	DIN54231	ND	ND	ND	ND	ND
2300	INH-11	nd	nd	nd	nd	nd
2301		----	----	----	----	----
2310	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2311	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2320	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	DIN54231	<15	<15	<15	<15	<15
2347	DIN54231	<15	<15	<15	<15	<15
2350	DIN54231	<15	<15	<15	<15	<15
2352		----	----	----	----	----
2357	DIN54231	<15	<15	<15	<15	<15
2358	DIN54231	N.D.	N.D.	N.D.	N.D.	N.D.
2363	DIN54231	<15	<15	<15	<15	<15
2365	DIN54231	<15	<15	<15	<15	<15
2370	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2375		----	----	----	----	----
2379	DIN54231	Not detected	Not detected	Not detected	Not detected	Not detected
2380		----	----	----	----	----
2386	DIN54231	<5	<5	<5	<5	<5
2390	DIN54231	N.D	N.D	N.D	N.D	N.D
2410	DIN54231	<20	<20	<20	<20	<20
2415	DIN54231	ND	ND	ND	ND	ND
2446		----	----	----	----	----
2467		----	----	----	----	----
2482		----	----	----	----	----
2489	DIN54231	ND	ND	ND	ND	ND
2492		----	----	----	----	----
2497		----	60.21	----	----	----
2508		----	----	----	----	----
2532	DIN54231	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2538	§64LFGBB82.02-10	n.d.	n.d.	n.d.	n.d.	n.d.
2546		----	----	----	----	----
2553	DIN54231	ND	ND	ND	ND	ND
2563		----	----	----	----	----
2566		----	----	----	----	----
2567	DIN54231	ND	ND	ND	ND	ND
2590		----	----	----	< L.O.Q.	< L.O.Q.
2591		----	----	----	----	----
2614	DIN54231	<15	<15	<15	<15	<15
2618		----	----	----	----	----
2644		----	----	----	----	----
2668	DIN54231	n.d.	n.d.	n.d.	n.d.	n.d.
2671		----	----	----	----	----
2723		----	----	----	----	----
2737		----	----	----	----	----
3146		----	----	----	----	----
3150		----	----	----	----	----

3151		----	----	----	----	----
3153	DIN54231	<15.0	<15.0	<15.0	<15.0	<15.0
3154		----	----	----	----	----
3172	DIN54231	<10.00	<10.00	<10.00	<10.00	<10.00
3176		----	----	----	----	----
3190		----	----	----	----	----
3197	DIN54231	ND	ND	ND	ND	ND
3210	DIN54231	<50	<50	<50	<50	<50
3214	DIN54231	N.D	N.D	N.D	N.D	N.D
3220		----	----	----	----	----
3225	DIN54231	<7.5	<7.5	<7.5	<7.5	<7.5
3228	DIN54231	Not detected	Not detected	Not detected	Not detected	Not detected
3233		----	----	----	----	----
3237		----	----	----	----	----
3248		----	----	----	----	----
normality	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
n	42	42	42	42	42	42
outliers	0	0	0	0	0	0
mean (n)	not detected	not detected	not detected	not detected	not detected	not detected
st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
R(DIN54231:05)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

**APPENDIX 2**

Summary of all other reported dyes in samples #16525; results in mg/kg

Lab	Other reported Disperse Dyes
---	none

Summary of all other reported dyes in samples #16526; results in mg/kg

Lab	Other reported Disperse Dyes
---	none

Comments on samples #16525

lab	Comments Allergenic dyes
362	The result for Red 17 is 8.42mg/l, according to the ratio 1:15 (DIN 54231) we calculate the result 126.3mg/kg The result for Yellow 3 is 8.09mg/l, according to the ratio 1:15 (DIN 54231) we calculate the result 121.4mg/kg
2538	Disperse Yellow 3 is not part of the analysis method (detected but not quantified).
2590	We don't have all required dyes standards. Standards we have are from Dr. Ehrenstorfer, with declared purity.

Comments on samples #16526

lab	Comments Carcinogenic dyes
362	The results is 6.26mg/l, according to the ratio 1:15 (DIN54231) we calculate the result 93.9mg/kg
2390	Basic Green 4 (oxalate) CAS no. 2437-29-8, Basic Green 4 (chloride) CAS no. 569-64-2 & Basic Green 4 (free) CAS no. 10309-95-2 is qualitatively analyzed.
2590	We don't have all required dyes standards. Standards we have are from Dr. Ehrenstorfer, with declared purity.



**APPENDIX 3**

## Summary of reported analytical details

Lab	What material was purchased for the colorants stock calibration solutions?	How were the freshly prepared stock calibration solutions of the colorants checked?	How were the stock calibration solutions of the colorants stored?	How were the stored stock calibration solutions of the colorants checked before use?
213	---	---	---	---
348	powder	other	<4°C	by measurement
362	powder	visually	at room temperature	visually
551	powder	by measurement	<4°C	other
623	powder	by measurement	between 4°C and 10°C	by measurement
840	powder	by measurement	between 4°C and 10°C	by measurement
2108	powder	by measurement	at room temperature	by measurement
2115	ready to use solution	by measurement	between 4°C and 10°C	by measurement
2129	powder	by measurement	at room temperature	by measurement
2131	stock solution	visually	between 4°C and 10°C	by measurement
2139	stock solution	by measurement	between 4°C and 10°C	visually
2165	powder	visually	between 4°C and 10°C	visually
2172	powder	visually	<4°C	by measurement
2184	powder	visually	between 4°C and 10°C	visually
2232	powder	by measurement	<4°C	visually
2241	powder	visually	<4°C	by measurement
2247	powder	visually	<4°C	visually
2255	powder	by measurement	<4°C	by measurement
2265	powder	by measurement	between 4°C and 10°C	by measurement
2284	powder	by measurement	<4°C	by measurement
2289	powder	by measurement	between 4°C and 10°C	by measurement
2290	stock solution	other	other	other
2293	powder	not checked	between 4°C and 10°C	not checked
2295	powder	other	<4°C	by measurement
2300	---	---	---	---
2301	powder	visually	<4°C	visually
2310	powder	by measurement	<4°C	by measurement
2311	powder	visually	<4°C	not checked
2320	powder	visually	between 4°C and 10°C	visually
2330	powder	not checked	between 4°C and 10°C	by measurement
2347	ready to use solution	other	other	other
2350	powder	visually	between 4°C and 10°C	by measurement
2352	powder	visually	between 4°C and 10°C	visually
2357	powder	by measurement	between 4°C and 10°C	by measurement
2358	powder	by measurement	<4°C	by measurement
2363	powder	by measurement	between 4°C and 10°C	by measurement
2365	powder	by measurement	<4°C	by measurement
2370	powder	by measurement	<4°C	by measurement
2375	powder	by measurement	<4°C	by measurement
2379	powder	by measurement	between 4°C and 10°C	by measurement
2380	powder	by measurement	<4°C	by measurement
2386	powder	visually	between 4°C and 10°C	visually
2390	powder	visually	between 4°C and 10°C	by measurement
2410	powder	visually	between 4°C and 10°C	visually
2415	powder	visually	between 4°C and 10°C	visually
2446	powder	by measurement	<4°C	by measurement

Lab	What material was purchased for the colorants stock calibration solutions?	How were the freshly prepared stock calibration solutions of the colorants checked?	How were the stock calibration solutions of the colorants stored?	How were the stored stock calibration solutions of the colorants checked before use?
2467	powder	visually	between 4°C and 10°C	visually
2482	stock solution	by measurement	<4°C	by measurement
2489	powder	visually	<4°C	visually
2492	stock solution	visually	<4°C	visually
2497	stock solution	by measurement	between 4°C and 10°C	by measurement
2508	powder	by measurement	between 4°C and 10°C	by measurement
2532	powder	visually	<4°C	visually
2538	powder	by measurement	between 4°C and 10°C	other
2546	---	---	---	---
2553	powder	other	<4°C	other
2563	powder	visually	at room temperature	by measurement
2566	powder	by measurement	<4°C	visually
2567	powder	visually	<4°C	visually
2590	powder	visually	between 4°C and 10°C	other
2591	powder	visually	between 4°C and 10°C	visually
2614	powder	by measurement	<4°C	by measurement
2618	powder	other	other	other
2644	---	---	---	---
2668	---	---	---	---
2671	powder	other	other	other
2723	---	---	---	---
2737	powder	visually	<4°C	visually
3146	---	---	---	---
3150	stock solution	by measurement	at room temperature	by measurement
3151	powder	by measurement	<4°C	by measurement
3153	powder	by measurement	between 4°C and 10°C	by measurement
3154	powder	visually	at room temperature	visually
3172	powder	visually	between 4°C and 10°C	by measurement
3176	powder	by measurement	<4°C	by measurement
3190	powder	other	other	other
3197	powder	by measurement	<4°C	by measurement
3210	powder	visually	between 4°C and 10°C	visually
3214	powder	by measurement	<4°C	by measurement
3220	---	---	---	---
3225	powder	visually	<4°C	visually
3228	powder	visually	between 4°C and 10°C	visually
3233	powder	visually	between 4°C and 10°C	visually
3237	powder	not checked	between 4°C and 10°C	not checked
3248	powder	by measurement	<4°C	by measurement

## APPENDIX 4

### Number of participants per country

3 labs in BANGLADESH  
1 lab in BRAZIL  
1 lab in BULGARIA  
2 labs in CAMBODIA  
2 labs in FRANCE  
13 labs in GERMANY  
1 lab in GUATEMALA  
6 labs in HONG KONG  
11 labs in INDIA  
2 labs in INDONESIA  
6 labs in ITALY  
3 labs in KOREA  
1 lab in MOROCCO  
13 labs in P.R. of CHINA  
1 lab in PAKISTAN  
1 lab in ROMANIA  
1 lab in SINGAPORE  
2 labs in SPAIN  
2 labs in SRI LANKA  
2 labs in SWITZERLAND  
2 labs in TAIWAN R.O.C.  
1 lab in THAILAND  
5 labs in TURKEY  
3 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's outlier test
R(0.05)	= straggler in Rosner's outlier test
n.e.	= not evaluated
n.d.	= not detected

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