Results of Proficiency Test
Migration of Elements EN71-3
Category 2
April 2021

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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Report: iis21V02B

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1 Introduction

Toy safety is the practice of ensuring that toys, especially those made for children, are safe, usually through the application of set safety standards. In many countries, toys must be able to pass safety tests in order to be sold. Many regions model their safety standards on the EU's EN71 standard, either directly, or through adoption of the ISO8124-3 standard which in itself is modelled on EN71. In Europe, toys must meet the criteria set by the EC Toy Safety Directive (Council Directive 88/378/EEC). This directive has been superseded by Council Directive 2009/48/EC, which applies to toy imports into the EU since 20 July 2011. There is an exception for the chemical requirements under part III of Annex II of this directive. These chemical requirements became into force on 20 July 2013.

The test methods EN71-3:2019 and ISO8124-3:2020 both describe the determination of Migration of Elements (metals that are considered hazardous) when a toy gets into contact with an acid solution (0.07 n HCl, simulating a gastric acid solution).

Since 2010 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Migration of Elements EN71-3 every year. During the annual proficiency testing program 2020/2021, it was decided to continue the proficiency test for the analysis of the Migration of Elements. This year it was decided to publish the proficiency test results for each category separately.

In the interlaboratory study Migration of Elements EN71-3 for category 2 samples 27 laboratories in 15 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the proficiency test of category 2 samples are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.

It was decided to send one sample of 8 mL Fingerpaint labelled #21570 and one sample of 8 mL Slime labelled #21571.

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 Organization, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

A batch of green colored Fingerpaint was purchased in a local shop. To this batch the elements Cobalt, Chromium and Nickel were added as salts. After mixing thoroughly the batch of Fingerpaint was divided over 78 small PE bottles of 10mL and labelled #21570. The homogeneity of the subsamples was verified by measuring the content of the elements Cobalt, Chromium and Nickel in accordance with EN71-3 on 8 stratified randomly selected subsamples.

	Cobalt in mg/kg	Chromium in mg/kg	Nickel In mg/kg
sample #21570-1	23.3	7.09	38.5
sample #21570-2	22.2	7.40	37.0
sample #21570-3	23.2	8.39	38.0
sample #21570-4	23.2	7.56	37.8
sample #21570-5	22.9	7.45	37.8
sample #21570-6	22.8	6.93	37.6
sample #21570-7	23.5	7.35	38.7
sample #21570-8	22.4	6.97	37.3

Table 1: homogeneity test results of subsamples #21570

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

	Cobalt in mg/kg	Chromium in mg/kg	Nickel In mg/kg
r (observed)	1.3	1.30	1.6
reference test method	EN71-3:19	EN71-3:19	EN71-3:19
0.3 x R (reference test method)	3.9	1.24	6.4

Table 2: evaluation of the repeatabilities of subsamples #21570

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

A batch of red colored Slime was obtained from a third party. This batch was found positive and was rejected from the market. After thorough mixing the batch of Slime was divided over 78 small PE bottles of 10mL and labelled #21571.

The homogeneity of the subsamples was verified by measuring the content of the element Boron in accordance with EN71-3 on 8 stratified randomly selected subsamples.

	Boron in mg/kg
sample #21571-1	831
sample #21571-2	851
sample #21571-3	870
sample #21571-4	850
sample #21571-5	849
sample #21571-6	874
sample #21571-7	832
sample #21571-8	874

Table 3: homogeneity test results of subsamples #21571

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.

	Boron in mg/kg
r (observed)	49
reference test method	EN71-3:19
0.3 x R (reference test method)	108

Table 4: evaluation of the repeatabilities of subsamples #21571

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

To each of the participating laboratories one sample of 8mL Fingerpaint labelled #21570 and one sample of 8mL Slime labelled #21571 was sent on March 24, 2021.

2.5 ANALYZES

The participants were requested to determine the migration of nineteen elements applying the analysis procedure that is routinely used in the laboratory. It was also requested to report if the laboratory was accredited for the determination Migration of Elements and to report some analytical details.

It was explicitly requested to treat the samples as if they were routines sample and to report the test results using the indicated units on the report form and not to round the test results but report as much significant figures as possible. It was also requested not to report "less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods 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 samples receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 and 2 of this report. The laboratories are presented by their code numbers.

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

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

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

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by F(0.01) for the Rosner's test. Stragglers are marked by F(0.01) for the Dixon's test, by F(0.01) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

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

3.3 Z-SCORES

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

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

When a laboratory did use a test method with a reproducibility that is significantly different

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

The z-scores were calculated according to:

```
z_{\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 problems occurred with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. All participants reported at least one test result. Four participants reported the test results after the reporting deadline. Not all laboratories were able to report all elements requested.

Finally, 27 reporting laboratories submitted 94 numerical test results. Observed were 5 outlying test results, which is 5.3%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

All original data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER ELEMENT

In this section the reported test results are discussed per sample and per element. 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.

EN71-3 method is considered to be the official test method for the determination of Elements migrated from different matrices. In April 2019 the CEN committee published a new version of EN71-3. In this 2019 test method of EN71-3 new precision data are given in table 4 and in table C.1. Table 4 contains precision data from an interlaboratory study. The committee was not able to obtain precision data for all elements for each category via an interlaboratory study. In order to compensate for missing data for certain element and category combinations estimations for the reproducibility have been considered by the committee based on table 4 and input from experts. These precision data are given in table C.1 of EN71-3:19 and are used to evaluate the performance of the group of participants in this PT.

Sample #21570

<u>Chromium (III):</u> This determination was problematic. Three statistical outliers were

observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the target reproducibility based on

EN71-3:19.

Cobalt: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in good agreement with the

target reproducibility based on EN71-3:19.

Nickel: This determination was not problematic. One statistical outlier was

observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the target reproducibility based on EN71-3:19.

The majority of the participants agreed on a concentration near or below the limit of detection for all other reported elements, see appendix 2.

Sample #21571

<u>Boron:</u> This determination was not problematic. One statistical outlier was

observed. The calculated reproducibility after rejection of the statistical outlier is good in agreement with the target reproducibility based on

EN71-3:19.

The majority of the participants agreed on a concentration near or below the limit of detection for all other reported elements, see appendix 2.

4.2 Performance evaluation for the group of Laboratories

A comparison has been made between the reproducibilities as declared by the reference test method and the reproducibilities as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibilities (2.8 * standard deviation) and the target reproducibilities, derived from the official test method EN71-3:2019 are presented in the next table.

Element	unit	n	average	2.8 * sd	R(target)
Chromium (III) as Cr	mg/kg	17	5.05	3.51	2.83
Cobalt as Co	mg/kg	24	19.6	4.2	11.0
Nickel as Ni	mg/kg	24	31.5	5.1	17.6

Table 5: reproducibilities on sample #21570 (Fingerpaint)

Element	unit	n	average	2.8 * sd	R(target)
Boron as B	mg/kg	24	912	239	383

Table 6: reproducibility on sample #21571 (Slime)

Without further statistical calculations, it can be concluded that the group of participating laboratories has no difficulties with the determination of the migration of the evaluated elements in plaster in accordance with EN71-3:19, except for Chromium (III).

4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2021 WITH PREVIOUS PTS

The uncertainties determined in this PT are compared with the relative standard deviations as found in previous years and with the target requirements in the next tables.

Element	April 2021	April 2020	April 2019	EN71-3:19 table C.1
Aluminum		9%		15%
Antimony				30%
Arsenic				20%
Barium	-		22%	15%
Boron	9%			15%
Cadmium	-	6%		20%
Chromium (III)	25%			20%
Chromium (VI)				50%
Cobalt	8%			20%
Copper	ŀ			15%
Lead		6%	22%	20%
Manganese	ŀ			15%
Mercury	1			30%
Nickel	6%		19%	20%
Selenium	ŀ			20%
Strontium				15%
Tin				20%
Organic Tin				50%
Zinc				15%

Table 7: development of uncertainties over the years for category 2 materials

The performance of the group is in general smaller than the precision requirements of EN71-3:19 table C.1.

4.4 EVALUATION OF ANALYTICAL DETAILS

A vast majority of the registered participants mentioned that they are ISO/IEC17025 accredited for category 2 determination of Migration of Elements EN71-3.

Furthermore, the participants were asked to provide several analytical details which are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

In EN71-3:19 it is emphasized that maintaining the pH between 1.1 and 1.3 is very important for the migration of the elements. All reporting participants have used a solution with a pH between 1.1 and 1.3 (with or without adjustment of HCl solution) to measure the metals. All reporting participants mentioned to have used for sample intake at least 100 mg or more.

For the migration, (almost) all reporting participants mentioned to have used a volume ratio of 5 mL of HCl solution per 100 mg sample intake.

When the analytical details where investigated separately, it appeared that the effect on the determination of metals in Fingerpaint and Slime is negligible.

5 DISCUSSION

In this PT it appeared that version of EN71-3:19 has been followed well by most of the participants.

For the Fingerpaint and Slime sample (#21570 and #21571, category 2) the influence (e.g. adjusting the pH and a minimum amount of intake) is visible when following the test method correctly. The relative standard deviation of the group for the measured metals, except for Chromium (III) is small compared to the relative standard deviation mentioned in EN71-3:19.

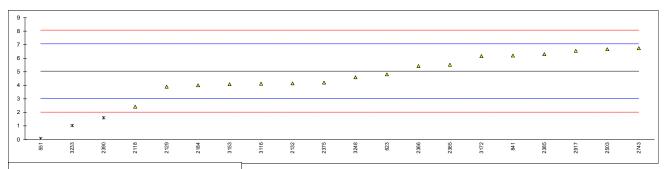
6 CONCLUSION

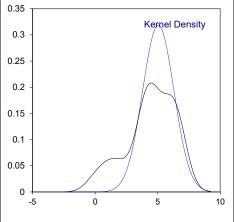
Each participating laboratory should evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of migration of Chromium (III) on Fingerpaint sample #21570; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
551	EN71-3	0.06676	G(0.01)	-4.93	
623	EN71-3	4.81		-0.24	
841	EN71-3	6.2		1.14	
2118	EN71-3	2.42		-2.60	
2129	EN71-3	3.898		-1.14	
2132	EN71-3	4.14		-0.90	
2137					
2184	EN71-3	4.00		-1.04	
2250					
2256	EN71-3	ND			
2365	EN71-3	5.5195		0.47	
2366	EN71-3	5.417		0.37	
2375	EN71-3	4.2		-0.84	
2385	EN71-3	6.30		1.24	
2390	EN71-3	1.6	C,G(0.05)	-3.42	First reported 1.44
2503	EN71-3	6.663	, ,	1.60	·
2743	EN71-3	6.7449		1.68	
2864					
2917	EN71-3	6.55		1.49	
3116	EN71-3	4.12		-0.92	
3153	EN71-3	4.08		-0.96	
3172	EN71-3	6.16	С	1.10	First reported 8.47
3176					·
3195					
3233	EN71-3	1.03	C,G(0.01)	-3.98	First reported 0.73
3248	EN71-3	4.589	, ,	-0.45	·
8005					
	normality	OK			
	n	17			
	outliers	3			
	mean (n)	5.0477			
	st.dev. (n)	1.25423	RSD = 25%		
	R(calc.)	3.5119			
	st.dev.(EN71-3:19)	1.00955			
	R(EN71-3:19)	2.8267			



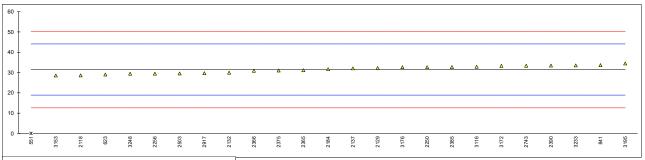


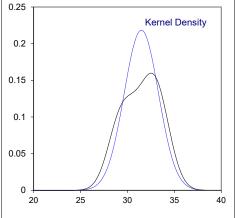
Determination of migration of Cobalt as Co on Fingerpaint sample #21570; results in mg/kg

lab	method	value	mark	z(targ)	remarks											
551	EN71-3	Not detected	IIIai K	2(tary)	Possibly		e nea	ative	test re	esult?						
623	EN71-3	18.18		-0.37	1 0001019	a lalo	o nog	auro	.00. 10	ouit.						
841	EN71-3	20.9		0.33												
2118	EN71-3	18.61		-0.26												
2129	EN71-3	20.126		0.13												
2132	EN71-3	19.61		0.00												
2137	EN71-3	19.85		0.06												
2184 2250	EN71-3 EN71-3	19.82 21.219		0.05 0.41												
2256	EN71-3 EN71-3	19.04		-0.15												
2365	EN71-3	19.4721		-0.13												
2366	EN71-3	19.50		-0.03												
2375	EN71-3	19.6		-0.01												
2385	EN71-3	21.1		0.38												
2390	EN71-3	21.65		0.52												
2503	EN71-3	18.20		-0.36												
2743	EN71-3	17.6812		-0.50												
2864 2917	EN71-3	 18.63		 -0.25												
3116	EN71-3 EN71-3	20.98		0.35												
3153	EN71-3	19.12		-0.13												
3172	EN71-3	21.80		0.55												
3176	EN71-3	20.41		0.20												
3195	EN71-3	22.0		0.61												
3233	EN71-3	16.08		-0.90												
3248	EN71-3	17.4		-0.57												
8005																
	normality	OK														
	n	24														
	outliers	0														
	mean (n)	19.624														
	st.dev. (n)	1.4990	RSD =	- 8%												
	R(calc.)	4.197														
	st.dev.(EN71-3:19)															
	st.dev.(EN71-3:19) R(EN71-3:19)	3.9248 10.989														
35 T																
35 -																_
_																
30 +		10.989							Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ
30		10.989	Δ	Δ Δ	Δ Δ				Δ	Δ	Δ	Δ	Δ	Δ	Δ	
30 +	R(EN71-3:19)	10.989	Δ	Δ Δ	Δ Δ			Δ.	Δ	Δ	Δ	Δ	Δ	Δ	Δ	
30	R(EN71-3:19)	10.989	Δ	Δ Δ	Δ Δ			A	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ
30	R(EN71-3:19)	10.989	Δ	Δ Δ	Δ Δ				Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ
30	R(EN71-3:19)	10.989	Δ	Δ Δ	Δ Δ		Δ	<u> </u>	Δ	Δ	Δ	Δ	Δ	Δ	Δ	
30	R(EN71-3:19)	10.989	△	5366	2375	2164	2137	2129	3176	Δ		4 ▲	2550	2390		
30	R(EN71-3:19)	10.989	△	2 2 3 9 6 P	2375	2184	2137	2129	3176		9 110 2 110 2 110 2 110 2 110 2 110 2 110 2 110 2 110 2 110 2 1 1 1 1				3172	3195 A
30	R(EN71-3:19)	10.989	8516	2 3% S S S S S S S S S S S S S S S S S S	2375	2/84	21637	2.129	3176							
30	R(EN71-3:19)	10.989	S 551 C	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2375 2132	2184	2.137	5/25	3176							
30	R(EN71-3:19)	10.989	8516	2 398 E 2 398 E 2 398 E	2375	2184	2(37	5,129	4							
30	R(EN71-3:19)	10.989	8516	2 3% S S S S S S S S S S S S S S S S S S	2375	2184	2137	2129	9178							
30	R(EN71-3:19)	10.989	3163	2 398 S S S S S S S S S S S S S S S S S S S	2375	2164	2137	2129	376							
30	R(EN71-3:19)	10.989	3163	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2375	2164	2157	2159	3776							
30	R(EN71-3:19)	10.989	A	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2375	2164	2157	2129	3776							
30	R(EN71-3:19)	10.989	S5162	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2375	2164	2157	2729	3776							
0.25 - 0.15 - 0.15 -	R(EN71-3:19)	10.989	85916	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2375	2164	2137	2129	3176							
30	R(EN71-3:19)	10.989	8516	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2375	2184	2157	2159	3776							
0.25 - 0.2 - 0.15 - 0.1 -	R(EN71-3:19)	10.989	85916	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2375	2164	2157	2159	3776							
0.25 - 0.15 - 0.15 -	R(EN71-3:19)	10.989	S516	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2375	2164	2157	2159	3776							
0.25 - 0.2 - 0.15 - 0.1 -	R(EN71-3:19)	10.989	A	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2375	2164	2137	2159	3776							
0.25 - 0.2 - 0.15 - 0.1 -	R(EN71-3:19)	10.989 A A A A A A A A A A A A A A A A A A A	ESS E	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2375	2164	2137	2159	3176							

Determination of migration of Nickel as Ni on Fingerpaint sample #21570; results in mg/kg

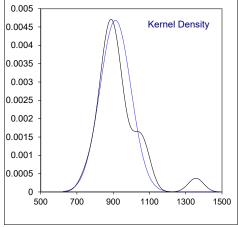
method	value	mark	z(targ)	remarks
EN71-3	0.0373	R(0.01)	-4.99	
EN71-3	29.06		-0.39	
EN71-3	33.6		0.34	
EN71-3	28.60		-0.46	
EN71-3	32.230		0.12	
EN71-3	29.94		-0.25	
EN71-3				
EN71-3	31.67		0.03	
EN71-3	32.623		0.18	
EN71-3	29.45		-0.32	
EN71-3	31.1121		-0.06	
EN71-3	30.9		-0.09	
EN71-3	31		-0.08	
EN71-3	32.7		0.19	
EN71-3	33.3774		0.30	
EN71-3	29.4		-0.33	
normality	OK			
•				
` '		RSD = 6%		
		070		
(=)				
	EN71-3 EN71-3 EN71-3 EN71-3 EN71-3 EN71-3 EN71-3 EN71-3 EN71-3 EN71-3 EN71-3 EN71-3	EN71-3 29.06 EN71-3 33.6 EN71-3 28.60 EN71-3 29.94 EN71-3 32.030 EN71-3 32.05 EN71-3 31.67 EN71-3 32.623 EN71-3 32.623 EN71-3 31.1121 EN71-3 30.9 EN71-3 31.1121 EN71-3 32.7 EN71-3 32.7 EN71-3 33.42 EN71-3 33.42 EN71-3 33.42 EN71-3 33.42 EN71-3 33.42 EN71-3 33.29 EN71-3 32.85 EN71-3 32.85 EN71-3 32.85 EN71-3 32.85 EN71-3 32.85 EN71-3 33.29 EN71-3 33.29 EN71-3 32.60 EN71-3 33.52 EN71-3 59.4	EN71-3	EN71-3





Determination of migration of Boron as B on Slime sample #21571; results in mg/kg

E E 4	method		value	9	n	nark	Z	(targ)	r	emarl	(S_											
551	EN71-3			ested																		
623	EN71-3		921.1					0.06														
841	EN71-3		1026					0.83														
2118	EN71-3		790.6					-0.89														
2129	EN71-3		854.9					-0.42														
2132	EN71-3		891.5	54				-0.15														
2137	EN71-3		867	7				-0.33														
2184	EN71-3		930.7					0.13														
2250	EN71-3		902.0 1081					-0.08														
2256	EN71-3 EN71-3		849.4					1.24 -0.46														
2365 2366	EN71-3 EN71-3		864.7					-0.46														
2375	EN71-3 EN71-3		940					0.20														
2385	EN71-3		1356		ь	(0.01)		3.24														
2390	EN71-3 EN71-3		928.0		г	(0.01))	0.11														
2503	EN71-3		866.6					-0.34														
2743	EN71-3		841.8	, 3891				-0.52														
2864	EN71-3		751.7					-1.17														
2917	EN71-3		880.4					-0.23														
3116	EN71-3		897.4					-0.11														
3153	EN71-3		908.1					-0.03														
3172	EN71-3		952					0.29														
3176	EN71-3		1049	.73				1.00														
3195	EN71-3		1070					1.15														
3233	EN71-3		823.1					-0.65														
3248	EN71-3		1009	.9				0.71														
8005																						
	normality		OK																			
	n		24																			
	outliers		1																			
	mean (n)		912.4		_																	
	st.dev. (n)		85.21		R	SD =	9%															
	- /		7748 6	308																		
	R(calc.)	0.40\	238.6																			
	st.dev.(EN71		136.8	3733																		
				3733																		
300 -	st.dev.(EN71		136.8	3733																		
	st.dev.(EN71		136.8	3733																		
	st.dev.(EN71		136.8	3733																		<u>*</u>
400 -	st.dev.(EN71		136.8	3733																		*
400	st.dev.(EN71		136.8	3733														Δ	Δ	Δ	Δ	*
1600 T 1400 + 1200 + 1000 +	st.dev.(EN71- R(EN71-3:19		136.8 383.2	3733 245				Δ			Δ		Δ.		Δ	Δ	Δ	Δ	Δ	Δ	Δ	*
200	st.dev.(EN71		136.8	3733	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ			Δ	Δ	Δ	Δ	Δ	Δ	Δ	*
1400	st.dev.(EN71- R(EN71-3:19		136.8 383.2	3733 245	Δ	Δ	Δ	Δ	Δ	Δ					Δ	Δ	Δ	Δ	Δ	Δ	Δ	*
400	st.dev.(EN71- R(EN71-3:19		136.8 383.2	3733 245	Δ	Δ	Δ	Δ	Δ	Δ	Δ		^		Δ	Δ	Δ	Δ	Δ	Δ	Δ	*
400	st.dev.(EN71- R(EN71-3:19	Δ	136.8	3733 245				Δ Δ	Δ 91	Δ.	4	4		46	▲	Δ_						
400	st.dev.(EN71- R(EN71-3:19	Δ	136.8 383.2	3733 245	2903	2137	2917	2132	3116	2250	3163	623	2390	2184	2375	3472	3248	P44	3176	3195	5256	x x
400	st.dev.(EN71- R(EN71-3:19	Δ	136.8	3733 245				2132	3116	2250	3153	2 29	2390	2184	2375	3172						xx
400	st.dev.(EN71- R(EN71-3:19	Δ	136.8 383.2	3733 245	2503			2132	3116	2250	3153	S229	2390	2184	2375	3472						2385
400	st.dev.(EN71- R(EN71-3:19	Δ	136.8 383.2	3733 245	2503			Z 132	3116	2250	3153	△	2390	2184	2375	3172						x *
000	st.dev.(EN71- R(EN71-3:19	Δ	136.8 383.2	3733 245	2503			Z (32 Z	3116	2250	3163	△ € 529	2390	2/84	2375	4 3472						K K



APPENDIX 2
Determination of migration of Other Elements on Fingerpaint sample #21570; results in mg/kg

lab	Al	Sb	As	Ва	В	Cd	Cr(VI)	Cu
551	4.6546	Not detected	Not detected	Not detected	Not tested	Not detected	Not tested	0.0329
623	not detected							
841	5.5	<0.1	<0.1	<2.5	<2.5	<0.025	< 0.002	<2.5
2118	5.82	0.14	1.97	0.10	0.0	0.0	0.0146	0.95
2129								
2132	<50	<1	< 0.05	<25	<25	<0.05	< 0.0035	<15
2137								
2184	<10	<1	<0.5	<2.5	<10	<0.1	<0.004	<2.5
2250	<10	<1	<0,1	<1	<10	<0,1		<10
2256	6.22	ND						
2365	<50	<1.0	< 0.4	<50	<50	<0.10	< 0.0025	<50
2366	<50	<1.0	<0.4	<50	<50	<0.1	<0.0025	<50
2375	<50	<1	<0.4	<50	<50	<0.1	<0.005	<50
2385	7.38	<0.5	<0.25	<0.25	<0.25	<0.1	<0.005	2.65
2390	n.d							
2503	6.871							11.1
2743	7.5548			2.7399	8.0131			4.1458
2864		not detected						
2917	14.39	<1	<1	48.36	51.01	<1		1.39
3116	6.11	LT2	LT0.15	LT2	LT5	LT0.15	LT0.002	LT2
3153	<100	<1	< 0.3	<10	<50	<0.1	<0.0025	<10
3172	< 50	< 5	< 0.1	< 50	< 50	< 0.05	< 0.005	< 50
3176	6.20							
3195	<10	<0.20	0.027	<5.0	<5.0	<0.010		8.72
3233	5.87	< 5	< 0.05	< 5	< 5	< 0.05	< 0.0025	< 5
3248	<10	<1	<0.45	<10	<100	<0.1	<0.0025	<10
8005		LT2	LT2	LT2		LT2		

lab	Pb	Mn	Hg	Se	Sr	Sn	Org Sn	Zn
551	Not detected	0.0373	Not detected	Not detected	Not detected	Not detected	Not tested	0.3449
623	not detected	not detected	not detected					
841	<0.1	<2.5	<0.1	<0.5	<2.5	<0.025	<0.04	<2.5
2118	0.01	0.04	0.0	0.0	0.09	0.0	not analyzed	0.47
2129							not analyzed	
2132	<0.1	<25	<0.1	<1	<50	not applicable	<0.15	<50
2137								
2184	<0.4	<2.5	<0.5	<2.5	<2.5	<0.060	not detected	<10
2250	<0,1	<1	<0,1	<5	<1	<0,1		<10
2256	ND	ND	ND	ND	ND	ND	ND	ND
2365	<0.20	<50	<0.5	<4	<50	<4.9	<0.2	<50
2366	<0.2	<50	<0.5	<4	<50	< 0.03	<0.2	<50
2375	<0.2	<50	<0.5	<4	<50	<0.08		<50
2385	0.10	<0.25	<0.1	<0.5	0.12	<0.08	<0.02	2.50
2390	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2503								
2743								2.8004
2864	not detected		not detected	not detected				
2917	<1	<1	<1	<1	<1	<1		51.01
3116	LT0.2	LT2	LT0.15	LT2	LT2	LT2	LT0.0164	LT5
3153	<0.1	<10	<0.5	<1	<100	<10	<0.1	<100
3172	< 0.3	< 50	< 0.5	< 1	< 50	< 50	< 0.1	< 50
3176								
3195	0.030	<5.0	<0.025	<0.20	<5.0	<0.20		<5.0
3233	< 0.05	< 5	< 0.5	< 5	< 5	< 0.05	< 0.25	7.65
3248	<0.25	<10	< 0.95	<1	<10	<0.8	<0.2	<10
8005	LT2		LT2	LT2				

Determination of migration of Other Elements on Slime sample #21571; results in mg/kg

lab	Al	Sb	As	Ва	Cd	Cr(III)	Cr(VI)	Со	Cu
551	0.5987	not detected	Not tested	Not detected	Not detected				
623	not detected	0.06	not detected	not detected	not detected				
841	<2.5	<0.1	<0.1	<2.5	< 0.025	<0.2	< 0.002	<0.1	<2.5
2118	0.23	0.08	1.31	0.04	0.0	0.01	0.0003	0.0	0.0
2129									
2132	<50	<1	< 0.05	<25	< 0.05	<1	< 0.0035	<0.1	<15
2137									
2184	<10	<1	<0.5	<2.5	<0.1	<1	< 0.004	<0.5	<2.5
2250	<10	<1	<0,1	<1	<0,1			<0,1	<10
2256	ND								
2365	<50	<1.0	< 0.4	<50	< 0.10	<1.0	< 0.0025	<0.5	<50
2366	<50	<1.0	<0.4	<50	<0.1	<1.0	<0.0025	<0.5	<50
2375	<50	<1	<0.4	<50	<0.1	<1	< 0.005	<0.5	<50
2385	0.28	<0.5	<0.25	<0.25	<0.1	<0.25	< 0.005	<0.25	<0.25
2390	n.d	n.d	n.d	n.d	n.d	0.1	n.d	n.d	n.d
2503									1.264
2743	4.2690			7.4571					
2864		not detected	not detected	not detected	not detected				
2917	5.76	<1	<1	38.87	<1	<1	<1	<1	<1
3116	LT2	LT2	LT0.15	LT2	LT0.15	LT1	LT0.002	LT2	LT2
3153	<100	<1	< 0.3	<10	<0.1	<1	<0.0025	<1	<10
3172	< 50	< 5	< 0.1	< 50	< 0.05	< 4	< 0.005	< 1	< 50
3176									
3195	<10	<0.20	<0.020	<5.0	<0.010			<0.050	<2.0
3233	< 5	< 5	< 0.05	< 5	< 0.05	0.25	0.0051	< 0.5	< 5
3248	<10	<1	< 0.45	<10	<0.1	<1	< 0.0025	<1	<10
8005		LT2	LT2	LT2	LT2				

lab	Pb	Mn	Hg	Ni	Se	Sr	Sn	Org Sn	Zn
551	Not detected	Not detected	0.7919	Not detected	Not detected	Not detected	Not detected	Not detected	0.6090
623	not detected	not detected	not detected						
841	<0.1	<2.5	<0.1	<2.5	<0.5	<2.5	<0.025	<0.04	<2.5
2118	0.0	0.05	0.0	0.01	0.0	0.07	0.01	not analyzed	0.01
2129									
2132	<0.1	<25	<0.1	<1	<1	<50	not applicable	<0.15	<50
2137									
2184	<0.4	<2.5	<0.5	<2.5	<2.5	<2.5	<0.060	not detected	<10
2250	<0,1	<1	<0,1	<1	<5	<1	<0,1		<10
2256	ND	ND	ND						
2365	< 0.20	<50	< 0.50	<9	<4	<50	<4.9	<0.2	<50
2366	<0.2	<50	<0.5	<9	<4	<50	< 0.03	<0.2	<50
2375	<0.2	<50	<0.5	<9	<4	<50	<0.08		<50
2385	<0.1	<0.25	<0.1	<0.25	<0.5	<0.25	<0.08	<0.02	2.03
2390	n.d	n.d	n.d						
2503	1.036								
2743						0.4068			3.3113
2864	not detected		not detected		not detected				
2917	<1	<1	<1	<1	<1	<1	<1	<1	31.6
3116	LT0.2	LT2	LT0.15	LT2	LT2	LT2	LT2	LT0.0164	LT5
3153	<0.1	<10	<0.5	<1	<1	<100	<10	<0.1	<100
3172	< 0.3	< 50	< 0.5	< 5	< 1	< 50	< 50	< 0.1	< 50
3176									
3195	<0.010	<5.0	< 0.025	<0.25	<0.20	<5.0	<0.20		<5.0
3233	< 0.05	< 5	< 0.5	< 5	< 5	< 5	< 0.05	< 0.25	< 5
3248	<0.25	<10	< 0.95	<1	<1	<10	<0.8	<0.2	<10
8005	LT2		LT2		LT2				

APPENDIX 3
Analytical details for sample #20570 and #21571

Allaly	Analytical details for sample #20570 and #21571									
	ISO/IEC17025		Amount of 0.07 mol/L HCl solution used in	pH after 1 minute	Was the pH adjusted after 1	pH after				
lab	accredited	Sample intake	mL	shaking	minute of shaking	adjustment				
551	No	1 g	50 mL	1.25-1.27	No					
623	Yes	0.3	15	1.2	No					
841	Yes	0.2 grams	10 mL	> 1.3	Yes	< 1.3				
2118	Yes	0.5	25	1.28	No					
2129	Yes									
2132	Yes	0.1 gram	5 mL	1.28 / 1.27	No					
2137	Yes	0.2	0.613 mL / 100 mL	1.22	Yes	1.2				
2184	Yes	0.1g	5ml	1.14 / 1.13	No					
2250	Yes	0,200	10	1,2 +- 0,1	No					
2256	Yes	118.3 / 106.4 mg	5.92 / 5.32mL	1.290 / 1.340	Yes	1.130 / 1.204				
2365	Yes	0.5g	25mL	1.20	No	NA				
2366	Yes	0.2g / 1g	10ml / 28.5ml (dewax)							
2375	Yes	-	-	-		-				
2385	Yes	0.51 / 0.48	25.5/24.0	1.31/1.31	Yes	1.21/1.15				
2390	Yes	1.0425 / 1.5491	52ml / 77ml	1.2	No					
2503	Yes	0,2532 / 0,2340	12,6 / 11,7	1,14 / 1,2	No	1,14 / 1,12				
2743	Yes	1g	50	1.2	No					
2864	Yes	0.1g	5ml	pH 2.50	Yes	pH 1.20				
2917	Yes				Yes					
3116	Yes	0.5g	25mL	1.1 - 1.2	No	Not applicable				
3153	Yes	0.2g	10mL	1.32	Yes	1.19				
3172										
3176	Yes	0,1	100	1,19	No					
3195	No	0.50	25	1.1	No	1.1				
3233	Yes	0.1013 / 0.1115	5.1 / 5.6	1.24 / 1.25	No	1.24 / 1.25				
3248	Yes	0.2000	10	1.28	No					
8005	Yes	0.5g	25 mL	1.1 - 1.2		Not applicable				

APPENDIX 4

Number of participants per country

- 1 lab in BELGIUM
- 1 lab in BRAZIL
- 1 lab in FRANCE
- 4 labs in GERMANY
- 6 labs in HONG KONG
- 1 lab in INDONESIA
- 2 labs in ITALY
- 3 labs in P.R. of CHINA
- 1 lab in PAKISTAN
- 1 lab in SLOVENIA
- 1 lab in SOUTH KOREA
- 1 lab in TAIWAN
- 2 labs in TURKEY
- 1 lab in U.S.A.
- 1 lab in VIETNAM

APPENDIX 5

Abbreviations

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

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \\ DG(0.05) &= \text{straggler in Double Grubbs' outlier test} \\ \end{array}$

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 applicable n.e. = not evaluated n.d. = not detected

Literature

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- 14 Council Directive 88/378/EEC
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