

## FOOD CONTACT ARTICLES: IMPACT OF UNCERTAINTY CONTRIBUTIONS IN COMPLIANCE TESTING

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**Abstract** – Food contact articles must be compliant to applicable regulations on food safety. In the migration testing different sources of uncertainties can be characterized. In a case study on disposable gloves the contributions to the uncertainties to the final results are analysed.

**Keywords:** food contact materials, disposable gloves, validation, compliance, uncertainty, food safety.

### 1. INTRODUCTION

Food contact articles are covered by the Framework Regulation on Food Contact Materials [1] as regards food safety. In this field, any article has to comply to community legislation or, when not existing, to specific national legislations. Disposable gloves to handle foods are covered by this legislation. These articles can be used both at the industrial (food processing, catering, etc). If gloves are made of plastic they are under the EU Regulation n. 10/2011[2], while, if made of rubber (nitrile, latex) gloves are covered in Italy by a specific legislation [3] on rubber laying down migration limits. In both the cases the results of the migration tests should be reported together with their calculated uncertainty, to be subtracted before comparison with the legal limits. No official migration test is available for overall migration from rubber gloves and therefore a “in house” test was developed, analysing and calculating the uncertainty factors affecting the final results. This paper presents a case-study for the calculation of uncertainties associated to the determination of the overall migration from disposable rubber gloves. However, the approach is the same as for other materials constituting the gloves (eg. vinyl, etc.).

### 2. EXPERIMENTAL

#### **2.1. Overall Migration: contact test, expression of the results**

In order to expose to the contact with the food simulants the surface that come into contact with foods, gloves are tested by article filling, reversing them before filling with the selected simulant. The contact surface is measured by weighing standard printer paper cutted around the shape of the glove.

To perform the contact, the gloves are reversed, rapidly filled with preheated food simulant, immediately introduced in a preheated oven and kept into contact for predefined time and temperature. The volume of the simulant should reflect the average volume of the hand of an adult and it is 250-300 ml of food simulant, depending on the size of the gloves. The simulant is 3% acetic acid, being the most severe in migration test for rubber gloves. However, any other aqueous based simulant can be tested by this method. In fact, overall migration in aqueous based simulants is determined gravimetrically as required by the legislation. The gravimetric procedure is the same as for any other determination of non volatile residue. In short, the simulant from the contact test, potentially containing the migrated substance(s), is evaporated in tared dishes and weighed ( $s=0.1$ ) until constant weight is reached. In this field, EN1186:2003 [4] standard norms consider the weight as constant when the difference from two consecutive weighings is not higher than 0.5mg. The overall (or global, or total) migration is in fact the cumulative migration of non volatile substances, detected by this simple procedure. The identification of the migrants in the dry residue is not required in the determination of overall migration. It is indeed a parameter to indicate the inertness degree of a FCM with respect to its contact with a food.

For food contact articles (e.g. gloves) 3-4 items are submitted to the test.

It is important to underline that the final results are expressed as single article and not averaged for the following reasons:

- These are **not** specimens from the same article
- Not known the intra/inter lot variation of the manufacturer
- Calculating the associated expanded uncertainty considering the average would comprise also the industrial unhomogeneity of the production

Therefore:

- Consider each sample (glove) as unique and unrepeatably and calculate uncertainty individually for each sample
- Consider **ONLY** the analytical uncertainties impacting the results.

From practical point of view, to calculate uncertainties, two different steps are distinguished

- migration part: contact of the glove or a specimen with food simulants,
- measurement part: gravimetric determination of the migrant into the simulants.

In the next chapters each contribution is discussed, highlighting the approach that can be used in the laboratory.

## 2.2. Uncertainty in overall migration

### Uncertainty sources

Uncertainty sources to be considered when testing food contact articles come from both the article features and the analytical tests. The following sources contributing to uncertainty of the final result are recognized and then combined in a final equation to calculate expanded uncertainty to be associated to the result of the overall migration test [5]. It is to point out that the results are expressed with respect the contact surface (mg/dm<sup>2</sup>)

- Repeatability of the method
- Uncertainty of the volume measurements
- Uncertainty of the weighing
- Uncertainty in the measurement of the contact surface
- Uncertainty of the method

### Uncertainty components

#### Repeatability of the method $u_{(r)}$

To calculate the contribution of repeatability of the method, repeatability tests were performed on the gravimetric part on two concentration levels, one from a food contact article (n= 12 replicated determinations) and the other on a spiked solution (n= 14 replicated determinations). The calculated contribution to uncertainty was:

$$u_{(r)} = 0,42 \text{ mg/dm}^2$$

#### Balance calibration $u_{(M_m)}$

Uncertainty is estimated from the uncertainty limit in use of the balance (0,0003 g/ $\geq\sqrt{3}$ ), the rounding of the figures in the display (0,1 mg, uniform distribution) and the established condition settled in the method to consider the weight as “ constant” (0.5 mg, uniform distribution)

$$u_{(M_m)} = 0.17 \text{ mg}$$

#### Uncertainty from glassware $u_{(V)}$

Uncertainty sources on the measurement of the simulant volume derive from uncertainty of the calibration declared from the glassware producer  $u_{T_a}$  and from uncertainty from the difference of the Temperature between the simulant solution and the calibrated cylinder  $u_{T_e}$ .

$u_{T_a}$ : (A-class glassware), declared uncertainty is divided by  $\sqrt{6}$  assuming a triangular distribution

$u_{T_e}$ : The difference between the T of the calibration of the glassware is reasonably assumed +5°C (20°C+5°C). Uncertainty due to the thermal dilatation of the liquids is certainly higher than that of the glassware. Standard Uncertainty of a volume of a simulant (assuming a rectangular distribution of the T) is

$$u_{T_e} = \frac{2.1 \cdot 10^{-4} \times V \times 5}{\sqrt{3}} \quad (1)$$

Combining both the two contributions:

$$u_{(V)} = \sqrt{u_{T_a}^2 + u_{T_e}^2} \quad (2)$$

In the case of the example of gloves, a volume of simulant of 0.250 liter. was considered for the the calculation

$$u_{(V)} = 0.0083 \text{ liter}$$

Measurement of the contact surface  $u_{(S)}$

This contribution is one of the critical aspects. It depends from the features of the considered article and from the method using for calculating the contact area. Many methods are available for the determination of the food contact surface area, but standardised methods are not available. Some of the most commonly used methods were:

- calculation of the area using mathematical formulas for regular geometric shapes
- wrapping the sample in paper, cut and weigh the paper
- wrapping the sample in aluminium foil, cut and weigh the foil
- drawing the outline of the sample on paper, cut and weigh the paper.

In case of glove the method used to measure the contact area was “drawing the shape” of glove on paper (Fig.1).

To this aim, one glove was placed on standard paper for printer (80 g/m<sup>2</sup>) and its outline was drawn on the paper. Then the shape was cutted and weighed. From the known grammage of the paper the surface area was easy calculated. It is important to note that previously, the laboratory checked the constance of the grammage declared from the producer of the printer paper. To calculate the contact area of glove samples, two different operators performed ten replicated measurements of the surface of the **same** article (eg. glove) and from these data the estimated reproducibility of the laboratory in the measurement of the surface by this method was estimated:

$$u_{(S)} = 0.139 \text{ dm}^2$$



**Fig. 1.** “drawing the shape” on paper method to calculate the surface in the case of glove samples.

Uncertainty of the method  $u_{(M)}$

The Overall migration (mg/dm<sup>2</sup>) from glove sample was calculated in this case study by applying the following equation, that of course is applicable to any kind of gloves, either made of rubber or plastic.

$$M = \frac{(M_{C,1} - M_{C,2}) - (M_{B,1} - M_{B,2})}{S} = \frac{M_n}{S} \quad (3)$$

Where:

M= overall migration in simulant, expressed in mg/dm<sup>2</sup> calculated for the article n;

M<sub>C,1</sub>= mass of the tared dish and the residue after evaporation of the simulant (mg);

M<sub>C,2</sub> = mass of the tared dish before adding the simulant (mg);

M<sub>B,1</sub>= mass of the tared dish and the residue after evaporation of the simulant (mg) from the blank test;

M<sub>B,2</sub>= mass of the tared dish before adding the simulant (mg) from the blank test;

S= surface area (dm<sup>2</sup>);

M<sub>n</sub>= mass of migrated amount in the simulant (expressed in mg with respect to the article n).

According to GUM [6], the function to express the relationship between the uncertainty of the result and  $u_{(M)}$  the uncertainty of each input  $u_{(M_{C,1})}$ ,  $u_{(M_{C,2})}$ ,  $u_{(M_{B,1})}$ ,  $u_{(M_{B,2})}$ ,  $u_{(S)}$  is

$$u_{(M)} = \sqrt{\left(\frac{\partial M}{\partial M_{C,1}}\right)^2 u_{(M_{C,1})}^2 + \left(\frac{\partial M}{\partial M_{C,2}}\right)^2 u_{(M_{C,2})}^2 + \left(\frac{\partial M}{\partial M_{B,1}}\right)^2 u_{(M_{B,1})}^2 + \left(\frac{\partial M}{\partial M_{B,2}}\right)^2 u_{(M_{B,2})}^2 + \left(\frac{\partial M}{\partial S}\right)^2 u_{(S)}^2} \quad (4)$$

$$u_{(M)} = \sqrt{\left(\frac{1}{S}\right)^2 u_{(M_{C,1})}^2 + \left(\frac{1}{S}\right)^2 u_{(M_{C,2})}^2 + \left(\frac{1}{S}\right)^2 u_{(M_{B,1})}^2 + \left(\frac{1}{S}\right)^2 u_{(M_{B,2})}^2 + \left(\frac{M_n}{S^2}\right)^2 u_{(S)}^2} \quad (5)$$

Because  $u_{(M_{C,1})} \cong u_{(M_{C,2})} \cong u_{(M_{B,1})} \cong u_{(M_{B,2})}$

simplification is possible:

$$u_{(M)} = \sqrt{4 \left(\frac{1}{S}\right)^2 u_{(M_n)}^2 + \left(\frac{M_n}{S^2}\right)^2 u_{(S)}^2} \quad (6)$$

Where  $u_{(M)}$  is the uncertainty in weighing.

### Combined Standard Uncertainty

Overall migration from glove sample are expressed in mg/dm<sup>2</sup> food contact surface therefore combining the contributions we obtain the following formula for the calculation of the propagation of the uncertainties:

$$u_{(C)} = \sqrt{4 \left(\frac{1}{S}\right)^2 u_{(M)}^2 + \left(\frac{M_n}{S^2}\right)^2 u_{(S)}^2 + u_{(r)}^2 + u_{(V)}^2} \quad (7)$$

$$u_{(C)} = \sqrt{\left(\frac{2}{S} u_{(M)}\right)^2 + \left(\frac{M_n}{S^2} u_{(S)}\right)^2 + u_{(r)}^2 + u_{(V)}^2} \quad (8)$$

Where:

$$u_{(r)} = 0,42 \text{ mg/dm}^2$$

$$u_{(M)} = 0,17 \text{ mg}$$

$$u_{(S)} = 0,139 \text{ dm}^2$$

$$u_{(V)} = 0.0083 \text{ liter}$$

**Expanded Uncertainty** to be associated to the result is calculated with k=2 and 95% confidence level

$$U = 2u_c$$

### Worked Example

Uncertainty calculated for some representative levels for Overall migration (close to the legislative limits, in mg/dm<sup>2</sup>) for a surface of 1 dm<sup>2</sup> leads to the following results illustrated in Table 1.

In the example in the Table 1 the contribution of the uncertainty from the volume is not reported because negligible with respect the other contributions.

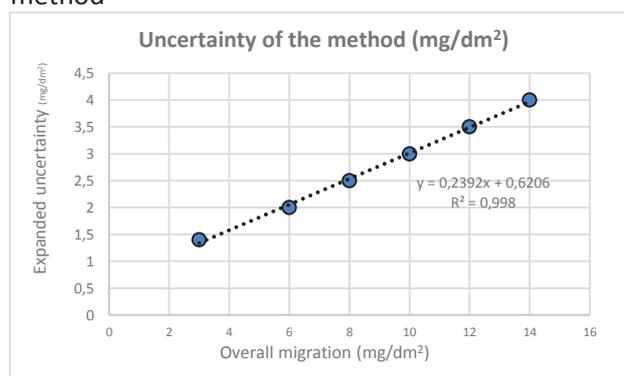
**Table 1.** Example of uncertainty values calculated at representative levels of Overall Migration

Migration mg/dm <sup>2</sup>	Contributions mg/dm <sup>2</sup>			Uncertainty		
	$\frac{2}{S} u_{(M)}$	$\frac{M_n}{S^2} u_{(S)}$	$u_{(r)}^2$	$u_{(C)}$ mg/dm <sup>2</sup>	U (k=2) mg/dm <sup>2</sup>	U (k=2) %
3	0.34	0.42	0.42	0,68	1,4	68
6	0.34	0,83	0,42	0,99	2.0	33
8	0.34	1.11	0,42	1.23	2.5	34
10	0.34	1.39	0,42	1.49	3.0	30
12	0.34	1.68	0,42	1.76	3.5	29
14	0.34	1.94	0,42	2.01	4.0	29

From the data in the Table 1 it is well evident which of the contributions mostly impacts final uncertainty. In fact the contribution from the surface measurement is more than double of the others. This stands for the levels of overall migration around 8 and 10 mg/dm<sup>2</sup>, the Italian legal limits for rubber FCMs and the EU legal limit for plastic FCMs, respectively.

By plotting the calculated expanded uncertainty versus overall migration the linear profile shown in Fig 2 is obtained for uncertainty of the method in the examined range of OM.

**Fig.2** Uncertainty profile of the Overall migration method



### 3.CONCLUSIONS

In the light of what above, it was possible to identify and quantify the contribution most impacting on uncertainty. This was of course the uncertainty of the measurements and calculation of the contact surface, due to the measurement methods that are currently available and commonly used.

To afford this problem and contributing to improve the homogeneity of the analytical results in the field of Food Contact Materials, EURL -FCM organized an Interlaboratory exercise [7]. The most common methods to measure contact surface area were used by 67 EU laboratories (30 NRLs and 37 national official control laboratories). Useful data can be extracted from this exercise to get a sounded figure to be used in the calculations. In fact, these figures could be used as acceptability limits for uncertainties in the measurements of surface to demonstrate good performance of the laboratory giving official results that may have legal impact. However, the laboratory could demonstrate through in house validation tests that may ensure better performance (e.g. by means optical scanners) and use internal data.

#### 4. REFERENCE

- [1] REGULATION (EC) No 1935/2004 of the European parliament and of the council of 27 october 2004 on materials and articles intended to come into contact with food and repealing Directives 80/590/EEC and 89/109/EEC
- [2] COMMISSION REGULATION (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food
- [3] Decreto ministeriale n. 34 del 21.3.1973 Disciplina igienica degli imballaggi, recipienti, utensili, destinati a venire in contatto con le sostanze alimentari o con sostanze d'uso personale pubblicato sulla Gazzetta Ufficiale - supplemento ordinario n. 104 del 20.4.1973 and further amendments
- [4] EN 1186 Materials and articles in contact with foodstuffs – Plastics –
- [5] EURACHEM/CITAC Guide CG 4 Third Edition QUAM:2012.P1
- [6] GUM Evaluation of measurement data — Guide to the expression of uncertainty in measurement JCGM 100:2008 GUM 1995 with minor corrections
- [7] ILC 003 2013 – Food Contact Surface Area of Kitchen Utensils - Report EUR 26477 EN Quantifying Uncertainty in Analytical Measurement