

INTERLABORATORY COMPARISON EXERCISE TO MEASURE THE CONTACT SURFACE AREA OF PRODUCTS WITH DIFFICULT GEOMETRICAL SHAPES IN MIGRATION TESTS

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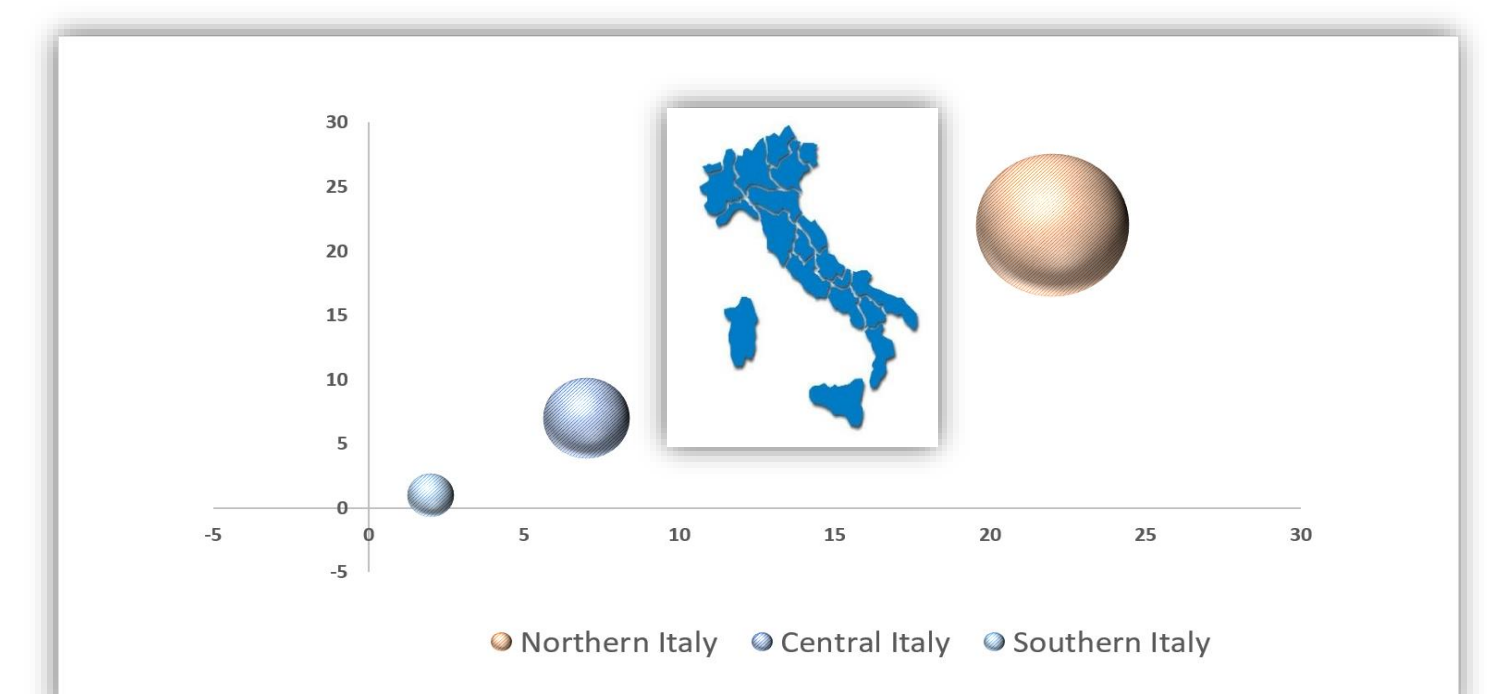
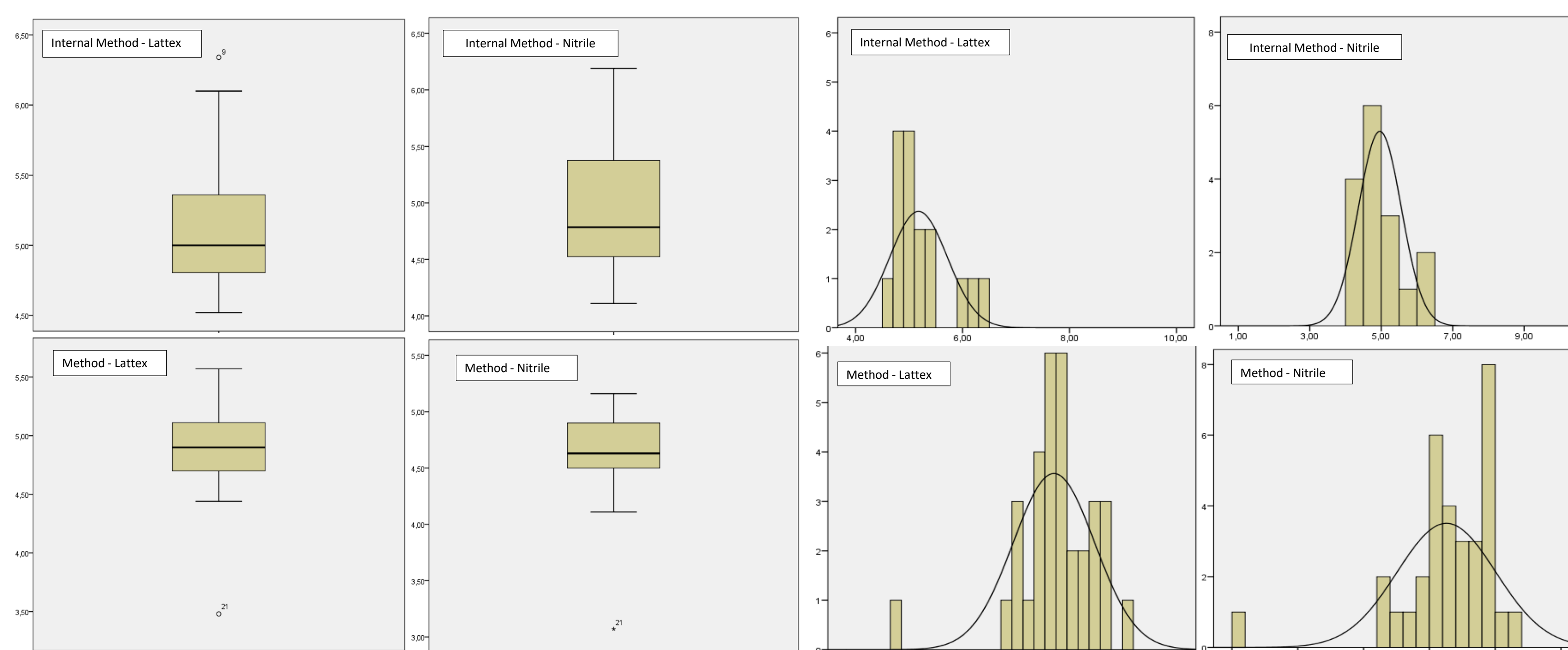


INTRODUCTION

The Italian National Reference Laboratory on Materials and Articles in Contact with Food (IT NRL-FCM) organized an Interlaboratory Comparison (ILC) on the measure of the contact surface area of products with difficult geometrical shapes to be analysed in migration tests. The general aim of the exercise was to assess the capability of Italian official control laboratories to measure the contact surface area and compare the methodologies used. The determination of the surface area (dm²) is an essential step in measurements for FCM migration tests, especially in the phase of interpretation of results for enforcement purposes. In fact, the contact surface area is needed for the expression of the final analytical results. Therefore, an exact and reproducible determination of the surface area is required.

To get a significant number of participating laboratories, in addition to the Official Control Laboratories, also private laboratories were invited to join the task. 33 laboratories participated to the ILC, especially from Northern Italy. Operative instructions were sent together with the samples (nitrile and latex gloves). Besides, two protocols for the surface measurements were possible: a) the one provided by the IT NRL b) any other in house protocol developed by the participating laboratory. The statistical design was based on robust statistical techniques (in conformity with ISO 13528:2015) to assign values. Only obvious incorrect results (aberrant data) were excluded from the statistical evaluation.

MAIN CORE



$$u(x_{ILC}) = 1.25 \times \frac{\sigma_{ILC}}{\sqrt{n}}$$

$$z = \frac{(x_i - x_{ILC})}{\sigma_{ILC}}$$

$$z' = \frac{(x_i - x_{ILC})}{\sqrt{\sigma_{ILC}^2 + u^2(x_{ILC})}}$$

Method type	Result number	Assigned value (robust mean) x_{ILC}	Standard deviation $S(\sigma_{ILC})$	Uncertainty $u(x_{ILC})$
NRL FCM - latex glove	33	4.9	0.31	0.067
NRL FCM - nitrile glove	33	4.67	0.28	0.061
Internal method - latex glove	16	5.11	0.46	0.144
Internal method - nitrile glove	16	4.93	0.62	0.193

Method	Number of z-scores	Number of z-scores with $ z \leq 2$	% z-scores with $ z \leq 2$
NRL FCM	33	31	91
Latex Glove			
NRL FCM	33	31	91
Nitrile Glove			

Method	Number of z-scores	Number of z-scores with $ z \leq 2$	% z-scores with $ z \leq 2$	Number of z'-scores	Number of z'-scores with $ z' \leq 2$	% z'-scores with $ z' \leq 2$
Internal Method	16	13	81	16	13	81
Latex Glove						
Internal Method	16	15	94	16	16	100
Nitrile Glove						

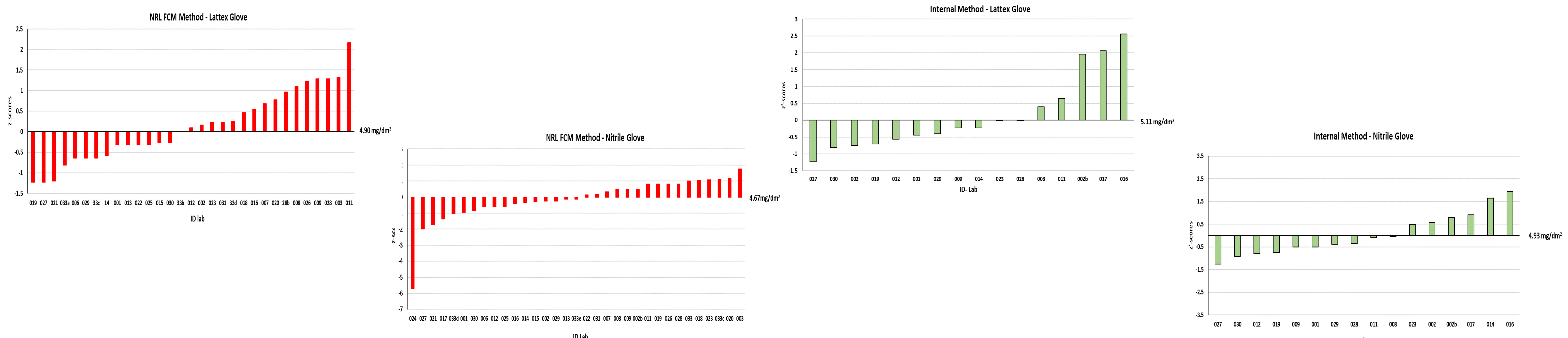
Results and Discussion

The most of participants reported similar results. Only two aberrant data were highlighted and submitted by the same participant: the value of 75.9 dm² and 81.6 dm² for Nitrile NRL method and Lattice NRL method, respectively.

The distribution of results for latex and nitrile rubber glove and for each method (NRL method or in house laboratory methods) was evaluated running a normality test. In our case, for small data sets, were used the Shapiro-Wilk test (p-value=0.05). The test has highlighted that only the combination nitrile rubber glove/in house laboratory method was normally distributed. In these results the null hypothesis states that the data follow a normal distribution because the p-value was 0.301, which was greater than the significance level of 0.05. For the remaining combinations the p-value was smaller than the significance level.

Furthermore, the distribution of the results submitted by laboratories were reported as box plots and frequency histograms (setting the bandwidth to a value of 0.75*σ_{ILC}). The histogram tool is useful in the preliminary analysis stage to check if there is an anomaly such as a large proportion of outliers or unusual skewness. These plots displayed one main mode indicating in general homogeneous data distributions.

A summary of the statistical data obtained is given in Table 1. All values were calculated using the algorithm A as described in the previous section. The z-score results are plotted in histogram graphs. The overwhelming majority (> 80%) of results has been classified with z-scores ≤ 2.



CONCLUSIONS and FUTURE CHALLENGE

To sum up, the obtained results in the meantime discussing the future way to use these results in the calculation of the uncertainty are useful in the harmonization process of the Italian laboratory approaches for the determination of the surface area in term of reproducibility and trueness. A new ILC is in progress on eco-friendly and sustainable green-utensils.

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