

## More Rigorous Methodology for Measurement of Corn Size Distribution of Barley is Needed

Dear Editor,

I have recently read the Committee Report titled “Appraisal of the Glasblaserei and the Sortimat Laboratory Screens for the Assessment of the Corn Size Distribution of Barley,” which was published in the *Journal of the Institute of Brewing*, 107(4):199-205.

Since I have been involved in the evaluation and validation of analytical methods during the last years of my professional career, as Quality Assurance Head in a brewery, I would like to offer some comments about the study.

There are two very different kinds of questions I would like to discuss: first, the selection of the methodology used in the appraisal, and second, different conceptual considerations in its implementation.

With regards to the methodology used, I do not see why a scheme used for Interlaboratory Efficiency Comparison should be used for qualifying a laboratory instrument or, more generally speaking, an analytical method, which is pre-known to be different. I am specifically referring to the fact that the MAPS scheme implements a Laboratory Ring Analysis as other schemes already did, but by using a robust statistics methodology. Its aim is not to test or compare new or different methods but to compare the proficiency of the participating laboratories.

It is in that sense that robust statistics present their advantages, where large deviations are going to be expected because of diversity of participating laboratories. However, and because of their usage, some important parameters of value when evaluating an analytical method are lost. I am referring particularly to the determination of the statistical coefficient usually defined in the appraisal of a methodology: coefficients of variations or their expressions as coefficient of variation, both for repeatability and reproducibility, as sound statistical indicators of the advantages of one method over another.

When considering this, it clearly suggests that the scheme offered by MAPS cannot address these questions, as it compares laboratory performances and not methodology performances. Certainly, it could be said that methods are performed by laboratories, but when making a decision so important, that involves the trade of large amounts of grains as claimed in the paper, every effort should be made to discriminate the methodology from its implementation, because even the most precise methodology can be ruined by improper implementation.

I consider the guidelines described in the International Standard ISO 5725 “Accuracy (Trueness and Precision) of Measurement Method and Results,” Part 1 (1994) and Part 2 (1994), the correct way to compare the performance of different methodologies.

This Standard has the clear advantage of providing a consistent, well known and widely adopted basis for designing experiments (in this case the methodology or instruments), establishing the minimum number and recruiting laboratories, preparing the materials, instructing the participant laboratories on a unique methodology to adhere to, collecting and statistically processing the information, and generating the report.

On the other hand, when considering the implementation of appraisal, I found several points that I would like to comment on.

- The first and probably most important remark is that the number of participating laboratories was very low, so no truly reliable statistical values can be inferred from that data. In an extreme situation, the results from only one laboratory have been computed. The comparison with the original methodology, as referred by Analytica-EBC, Method 3.11, “Sieving Test for Barley,” is more striking: the precision values were obtained from a collaborative trial in which 19–25 laboratories participated.
- Even worse, because there was only one laboratory performing the test on sample 12, corns 2.2–2.5 mm, the Robust Standard Deviation was assigned a value of 0.00. That is a conceptual mistake. In the case of a single result, the standard deviation cannot be determined, since it requires at least a degree of freedom for its calculation (G.W. Snedecor and W.G. Cochran, 1989, *Statistical Methods, Eighth Edition*, Iowa State University Press).
- In the paper it has been assumed that all equipment was in good condition and working satisfactorily. Also, it was assumed that screens had been checked for calibration of the slots. Why these assumptions? Why not include in the test protocol, instructions for checking the equipment and the calibrations instead of the assumption?

In the way it was done, the appraisal effectively tested the individual laboratory’s performance, but not the method, since no provisions were taken, in a general sense, to ensure the same conditions existed with the equipment.

Under the facts discussed above I kindly invite you to consider this work as a preliminary report, considering

that provisional and initial conclusions suggest that in uncontrolled conditions the Glasblaserei instrument appears to perform better.

I personally consider that no solid, statistically sound and hence definite conclusions can be drawn from this work.

Please note that I am not saying that the conclusion already drawn is not true. I am saying that it does not have enough data to support it.

Finally, I would like to propose the performance of a new trial, based on the ISO 5725 guidelines mentioned earlier.

Main aspects to consider:

- Recruitment of a reasonable number of laboratories
- Setting of a common methodology that includes proper instrument (frame, sieves, etc.) verification, calibration and operation
- As methodology involves time and weight measurements, properly calibrated instruments are required
- Calculation and report of results
- Procurement of homogeneous samples

- Grains are hygroscopic and humidity content can alter shape and/or size, consequently sample delivery and storage conditions are important
- Because of hygroscopicity, it is recommended that sample containers be opened at a pre-arranged dates, to minimize local environment effects
- Introducing and training the personnel in the methodology before the actual ring analysis is performed.

Thank you in advance for your kind attention to this matter.

Yours sincerely,

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