Quality Assurance by Means Laboratory Proficiency Testing. A practical Case of Wheat Flour Proficiency Testing

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Abstract – **Due to the development of technical methods that are used in laboratories, but also due to the increased importance given to the laboratory tests, the quality assurance concept becomes more prominent. There are several ways in which quality assurance can be handled: reproducibility tests, use of reference materials, calibrations, multiple determinations using a different technical method. In the recent years, the proficiency testing has been used more and more as a method for proving results' quality assurance. That is because mainly because the communication infrastructure, data processing capabilities increased. Proficiency testing rounds are organized by specialized entities for a wide variety of purposes: starting from for-profit activities to scientific research. In order to increase the credibility of the proficiency test, the organizing entity should have the proficiency testing round organized and implemented according to EN ISO 17043:2010. This paper introduces the concept of quality assurance by means of proficiency testing and metrological traceability in a proficiency testing round for food industry labs.**

Keywords – quality assurance, metrological traceability, proficiency testing, EN ISO 17043:2010

I. INTRODUCTION

Very simply put, participation at proficiency testing rounds is a means by which a laboratory can validate its technical working methods. Since the assigned value of the test item is not known in advance by the participating laboratories, it is established either by a special laboratory with high degree of competence and credibility (master laboratory) which provides results traceable to I.S. or by statistical computations of the result values provided by the participating laboratories. The size of the participation scheme (number of participation laboratories) can be anywhere between tens (a classical example being the niche-activity laboratories like anti-dopping labs) and several hundreds (local medical laboratories). There are cases with proficiency testing schemes which have about 25,000 participants with global coverage (cases of international chains of medical laboratories). Proficiency testing organization is done by 3rd party entities and they select the participant laboratories (based on their experience, technical methods they use), prepare and deliver the test items, process the results provided by participating laboratories and issue concluding reports. EN ISO 17043:2010 [8] is the applicable accreditation standard for proficiency testing organizers. The document contains 2 set of requirements: management and technical. One of the critical aspects with respect to proficiency testing organization is statistical design. There are some established statistical methods that are accepted (described in ISO 13528) but other mathematical methods can be used it was demonstrated they were suitable for the needs of the scheme. Also the metrological compatibility plays an important role. The need for having metrological comparability also extends in the time domain.

Topolnicka et al [1] present a proficiency testing scheme for charcoal and coke (fuel). The test item was a batch of charcoal of 5 kg. Homogeneity and stability tests consisted of 10 sub-samples that were separately analyzed. 45 laboratories participated in the round. Data processing (quantitative data) was done according to ISO 13528:2005. Only 2 labs had unsatisfactory results (z-score > 3). Zhu et al [2] introduce a proficiency testing scheme for analysis of window glass performance. The scheme distributed the 74 participants in 3 groups (26, 24, 24) based on the geographical coverage. The homogeneity tests were conducted on 10 arbitrarily selected samples. Statistical design was done using the robust analysis. As a result, 19 labs (25% of all) had unsatisfactory results. Interpretation of these results generated warning signals regarding the competency of technicians working in the labs given the fact that glass analysis process is fully automated. Luca et al [3] introduce a bilateral comparison scheme between 2 laboratories of ionizing radiation metrology. The test item consisted of 4 ionizing radiation emission pieces (2 with 226Ra, 60Co and 137Cs). The results showed minimal

difference between the results of the two labs, being in the measurement uncertainty limit. This proved the technical competency of the labs and calculation efficiency of the method for radionuclide activity. Lopez - Rioboo [4] introduces a proficiency testing scheme in the clothing industry. The purpose of the proficiency testing scheme is to check the spreading of dyes on clothing materials. 33 laboratories participated. The testing object was a piece of cloth which was stained with a dye that simulates staining the real clothing. A qualitative analysis was performed (unlike in the previous schemes presented before). The laboratories could select one of 9 values that characterized the sta (from "stainless" to "deep stain"). The assigned value was the mode of the reported values. Neves et al [5] introduce a proficiency scheme which evaluates the reproducibility of geometrical, mechanical and physical tests of aggregated materials. 9 laboratories participated in this round. The reproductibility formula used was the one defined by ISO 5725, part 2 [11]. The results obtained show the fact that determinations (evaluated for a time period of 10 years) are consistent. This proves confidence in the results produced by laboratories. Liu [6] and Massih et al [7] present the importance of saving a 2nd set of samples in case of proficiency testing in the field of sea water and the legal risks associated when no such sets are saved.

II. MATERIALS AND METHODS

The wheat quality plays an important role in the food industry. Ensuring that only high quality wheat is used in the manufacturing process, the relevance of food industry testing laboratories is particularly increased. Since, according to the ISO 17025, participation with good results in laboratory comparisons, proficiency testing schemes are an accepted means also in the field of food industry.

The proficiency testing scheme assessed the performance of food industry testing laboratories. The used test item was a batch of 2 kg of wheat flour, each being extracted from a bulk of 50 kg wheat flour which was deposited for 30 days at 20 Celsius after mixing. After extracting, each batch was packed and labeled accordingly. Relative humidity was 60%. Homogeneity was assured by mixing the wheat flour for 30 minutes at room temperature. Samples were sent to participating laboratories within 7 days from preparation.

The proficiency test organized did not run stability tests since wheat flour retains its properties for 6-8 months while the time elapsed from preparation to receiving the results from participating laboratories was just 10 days. During this proficiency test round, the items in Table 1 were assessed.

Table 1. Assessed items during the proficiency test round.

Cr. No.	Properties	Measurand	Unit of measurement
0	1	2	3
1		Humidity	%
2		Ashes content	%s.u.
3		Humid gluten (manual technique)	%
4		Humid gluten (mechanical technique)	%
5		Deformation index	mm
6		Drop-off Index	s
7		Proteic substaces content	%s.u.
8		Acidity	%
9	Chemical and physical properties	Farinograma: - hydration capacity - dough formation - stability - elasticity -moisture - power	 ml/100g min min UF UF -
10		Alveograph: - tenacity (P) - extensibility (L) - inflation index (G) - mixing power (W) - ratio P/L - elasticity index (Ie)	 mm H_2O mm - 10^4*J - -
11		Zeleny Index	ml
12	Microbiology properties	Yeast and Molds	UFC/g
13		Bacillus mezentericus	UFC/g
14		Bacillus cereus	UFC/g

For the processing, the algorithm described in [9] was used:

do
$x^* <-$ *median* $(x_1, x_2, \ldots\ldots\ldots x_n)$, where n – no. of reported results
$s^* <- 1,483 *$ *median* $(|x_i - x|)$
delta $<- 1,5 * s^*$
for each i = 1,n do
    if $x_i < x^* - delta$ *then* $x^*_i <- x^* - delta$;
    else if $x_i < x^* + delta$ *then* $x^*_i <- x^* + delta$;
    else $x^*_i <- x^*$;
end_for
sum = 0;
for each i = 1,n do
    sum = sum + x^*_1 / n;
end_for
$x^* <- sum;$
for each i = 1,n do
    sum = sum + sqr($x_i^* - x^*$);
end_for
$s\_new^* <- sqrt \ (sum \ / \ (n - 1))$
while ($s\_new^* < s^*$)

The convergence stop condition was more severe than in the standard, namely 0.001 instead of 0.01. This was done to ensure a higher convergence corresponding to the competence level declared by the participating laboratories. The algorithm above implements the robust statistics method for the following formulas:

$$u(x) = u(x^*) = (1.25 \cdot s^*) / \sqrt{n} ,\qquad (1)$$

where:
s – standard deviation calculated from the results reported by laboratories;
n – number of participating laboratories;
x – mean value of the reported lab results;
u – uncertaintly associated to attributed to test item, and

$$z = \frac{x_i - x^*}{\hat{\sigma}} ,\qquad (2)$$

where:
x_i – reported result by the laboratory;
x^* – value attributed to the test item (in this scheme was calculated as consensus value from the values reported by participating laboratories);
$\hat{\sigma}$ – standard deviation of the proficiency scheme.

All the participating laboratories received the proficiency testing report with z-score as a central result value. The interpretation of the z-score is as follows:
➢ $|z| \leq 2$ - satisfactory performance;
➢ $|z| < 3$ – acceptable;
➢ $|z| \geq 3$ - wrong.

III. RESULTS

Table 2 shows the results obtained by each laboratory during the proficiency testing scheme.

Table 2. z-scores obtained by participating labs for different parameters. "-" denotes the fact that lab did not submit a measurand value, therefore the z-score was not computed for that particular value.

Cr. No.	Measurand	Final result values																
		ICF 111	ICF 112	ICF 113	ICF 114	ICF 115	ICF 116	ICF 117	ICF 118	ICF 119	ICF 120	ICF 121	ICF 122	ICF 123	ICF 124	ICF 125	ICF 126	ICF 127
1	Humidity, %	0.91	0.18	-2.10	0.89	2.19	0.76	-1.10	0.84	-0.33	-0.19	-0.03	0.24	-1.94	-0.62	0.75	-0.51	-0.29
2	Ashes content, % s.u.	-	-	-	0.46	-	-3.07	-0.31	-	1.72	0.63	0.10	-1.12	-	-	-	-	-
3	Humid gluten, %																	
	-manual method	1.68	1.09	-0.26	1.13	0.02	-0.35	-1.13	0.46	-	-0.64	1.96	-1.14	0.63	-0.20	-0.84	-0.81	-0.64
4	Deformation index, mm	0.95	-	1.16	-0.85	-0.08	1.21	-	0.08	-	-	-0.34	-	-0.85	-0.85	0.70	-	-1.12
5	Drop-ff index, sec (*)	-	-	-	-	-	-0.02	-1.52	-	-	0.83	-	-0.31	-	-	-	1.02	-
6	Proteic substances content , % s.u.(*)	-	-	-	-1.80	-	-0.20	0.92	-	0.41	-	-	-	-	-	-	-	-
7	Acidity, %	-	-1.13	-0.47	0.03	0.52	-0.79	-	2.24	-	-1.94	0.03	-	0.77	1.26	-0.47	-	0.20
8	Rheological properties																	
	- hydration capacity, ml/100 g	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
9	Alveogram																	
	- tenacity (P), mm H_2O (*)	-	-	-	-	-	-	0.03	-	-	-0.97	-	1.25	-	0.37	-	-0.68	-

	-extensibility (L), mm (*)	-	-	-	-	-	-	-0.22	-	-	1.64	-	0.43	-	-0.29	-	-1.14	-
	- inflation index (G) (*)	-	-	-	-	-	-	-0.20	-	-	1.74	-	0.43	-	-0.26	-	-1.18	-
	- mixture energy (W), 10^4*J (*)	-	-	-	-	-	-	0.05	-	-	-0.54	-	1.44	-	0.13	-	-0.98	-
	- ratio P/L (*)	-	-	-	-	-	-	-0.02	-	-	-3.13	-	3.00	-	0.50	-	-0.45	-
	- elasticity index (Ie), % (*)	-	-	-	-	-	-	0.99	-	-	-0.35	-	-1.89	-	0.61	-	0.02	-
	Microbiology properties																	
10	Yeasts and Molds, UFC/g	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
11	*Bacillus cereus*, UFC/g	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

IV. CONCLUSION

This article introduces a new proficiency testing scheme in the field of food industry. The scheme results is interesting at two levels: firstly, at the application level, because it offers the participating labs the chance of testing their competence (to our knowledge it is the first time such scheme is organized by an accredited proficiency testing provider) and secondly, the convergence condition for the robust calculation was 1 order of magnitude higher and still all the labs on all the determinations (with one exception) obtained z-scores which yields a satisfactory result.

V. ACKNOWLEDGMENT

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