PROTOCOL OF QUALITY ASSURANCE AND ORGANIZATION OF PROFICIENCY TESTING IN DAIRY LABORATORY

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Abstract

Quality assurance of the analytical results is an obligation of all analytical laboratories which tend to be credible. In order to obtain accuracy and precision of results each laboratory implements suitable programmes according to EN ISO/IEC 17025:2006. Since 2005, Reference Laboratory of the Dairy Science Department is accredited according to the above mentioned norm. Validation of analytical methods and verification of validation parameters are part of good laboratory practice. Quality of analytical results is assured by use of reference methods and certified calibration standards and by participation in international proficiency trials which are organized by dairy laboratories from Germany, Italy, France, Switzerland and Slovenia. The Reference Laboratory of Dairy Science Department has its own protocol of quality control which is based on analytical procedures and methods which are traceable, accurate, reliable and applicable for certain purpose. At the same time, our laboratory organizes proficiency testing for other dairy laboratories. Results of analyses are statistically tested and on the basis of z-score we estimate the successfulness of measurements.

The aim of this paper is to demonstrate the organisation of proficiency testing (Ring control) of analytical results for milk fat, protein, lactose and somatic cells count in milk for other dairy laboratories in Croatia, Bosnia and Herzegovina and Macedonia.

Key words:
Quality assurance, proficiency testing, milk
1 INTRODUCTION

Milk quality depends on the number of different parameters which must be
determined. For each parameter there are often several different methods. Minor
errors in laboratory routines or errors of interpretations are usually found in results
when different methods are used for the same parameter. For that reason unification
of techniques and application of standard methods for determination of milk
components must be accepted by local dairy laboratories and the National Central
Laboratory for Milk. Those standard methods for determining microbiological and
chemical components are crucial for estimating the price of milk. Therefore, quality
assurance becomes necessary to ensure that the analytical results of milk
components are accurate and very similar from one laboratory to another which is
accomplished by the calibration of instruments with traceable calibration standards
and participation in proficiency tests [1].

The aim of this paper is to demonstrate the organisation of proficiency testing (Ring
control) of results for milk fat, protein, lactose and somatic cells count in milk for other
dairy laboratories in Croatia, Bosnia and Herzegovina and Macedonia.

2 EXPERIMENTAL

Individual cow’s milk samples for the Ring test must be fresh. Their chemical
components content must comply with an ordinary range, from minimum to maximum
values, depending on lactation stage. The number of somatic cell count and bacteria
count in milk samples should comply with the National Milk Quality Regulation [2].

2.1 Materials

The Reference Laboratory of the Dairy Science Department prepares secondary
reference material for the Ring control once a month and 20 to 25 high quality cow’s
milk samples are needed for preparing Ring samples. The quantity of samples
depends on the number of registered participants in Ring control. An average number
of participants is 20, and therefore about 3 litres per high quality sample is needed.

2.2 Procedures

Samples of milk are analysed by the infrared spectrometry method (using the
instrument Milkoscan FT 120, according to ISO 9622:1999) [3] to determine the
content of milk fat, protein and lactose, the fluoro-opto-electronic method is used to
determine somatic cell count (using the instrument Fossomatic Minor, according to
ISO 13366-2:2006) [4], whereas the flow citometry method is used to determine an
overall bacteria count (using the instrument Bactoscan FC, according to ISO
21187:2004) [9]. Based upon obtained results, 7 samples are chosen for Ring control
for milk fat, protein and lactose using IR instruments as well as 7 samples for somatic
cell count instruments using the fluoro-opto-electronic method. Samples must be of
high hygienic quality [2] and must comprise a specific content range. After that
chosen samples are preserved by bronopol, and as soon as the preservative is
dissolved, samples must be homogenized in the homogenisator. Then samples are
portioned in bottles of approximately 100 millilitres. Each participant of Ring control is
given one set which consists of 7 samples whereas the Reference Laboratory retains at least 3 to 5 same sets. These sets are used to determine reference values for the content of milk fat, protein and lactose (for Ring test, IR instruments) and somatic cell count (for Ring test, flouro-opto-electronic instruments). Reference values are determined on the basis of repeated sample measurements by the instrumental-routine methods (Milkoscan FT 120 and Fossomatic Minor) and the result accuracy is checked by the reference methods [5, 6, 7, 8, 10]. According to obtained reference values, when Ring control participants' results are received, statistical analysis is made to compare those results with reference values obtained in the Reference Laboratory. Prepared milk samples are kept in the refrigerator (5 ± 3°C) until they are sent over to Ring control participants. In transportation of samples, a portable refrigerator (5 ± 3°C) is used and participants must keep the samples under the same conditions until further analyses. The validity of milk samples is 5 days from sampling. The analysis procedure is as following: first of all samples must be left until they gradually reach the room temperature, then they must be heated in a water bath until they reach 41-43°C. After that the sample must be gently shaken and then analysed.

3 RESULTS AND DISCUSSIONS

For each laboratory involved in the inter laboratory trial, an annual report is made. It refers to how successful all participants have been in analyzing milk fat, protein and lactose (Figure 1) and somatic cell count (Figure 2). In the analytical report, z-score is presented for each parameter. The z-score should not exceed ±2.

![Figure 1 - Annual report of laboratory analyses of milk fat, protein and lactose content](image)
3.1 Statistical analysis

The statistical analysis is made according to AOAC Protocol [11]. The first stage in producing a score from a result $x$ is obtaining an estimate of the bias which is defined as:

$$ \text{bias} = x - X $$

where $X$ is true value or in practice the best estimate of $X$ and $x$ is measured value.

Next step is comparing the bias estimate with a target value for standard deviation that forms the criterion of performance. A $z$-score is formed from:

$$ z = (x-X) / \sigma $$

where $\sigma$ is the target value for standard deviation.

If $X$ and $\sigma$ are good estimates of the population mean and standard deviation, then $z$ will be approximately normally distributed with a mean of zero and unit standard deviation. As $z$ is standardized, it is comparable for all analytes and methods. Values of $z$ are combined to give a composite score for a laboratory in one round of proficiency test (Figure 1, Figure 2).

The $z$–scores can therefore be interpreted as follows:
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\[ z \leq 2 \quad \text{result is satisfactory} \]
\[ 2 < z < 3 \quad \text{result is questionable} \]
\[ z \geq 3 \quad \text{result is unsatisfactory} \]

4 CONCLUSIONS

Involving analytical laboratories in Ring control, we can control their accuracy of measured results on the instruments which are used for determining the content of milk fat, protein, lactose and somatic cell count in milk.

REFERENCES