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RESEARCH ARTICLE **(Open Access)**

Total solids and fat determination in milk; Interlaboratory testing

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Abstract

In the framework of quality management systems, proficiency tests become an important and useful tool for comparing results and so verifying technical competence of laboratories objectively. Additionally, in the accreditation requirements of ISO/IEC 17025 laboratories are asked to participate in proficiency tests regardless of other quality assurance activities for checking validity of tests results. Under the frame of IPA 2008 Project "Regional Quality Infrastructure in the Western Balkans and Turkey", the Dairy Laboratory, part of Food Quality Department, in the Institute of Food Safety and Veterinary in Tirana, took part in a Proficiency Testing for comparison on fat, proteins and total solids in heat-treated milk (UHT) as part of the Proficiency Testing Protocol 5 (PT5) organized by the Dairy Laboratory, Biotechnical Faculty- Podgorica, Montenegro. There were 8 laboratories that participated in PT. 7 samples were sent to each participant laboratories and after getting back the results, the statistical calculations were done by the organizing body. This PT was the first one our laboratory took part in. Our results achieved encouraged us to present the work done and to analyze the factors we think have influenced. In this paper we have presented our work for total solids and fat content determination in seven milk samples analyzed according to the respective method presented in following. The results for total solids and fat content for each sample analyzed are showed in the respective tables as well as the results for accuracy assessment, i.e, comparability with the reference results and repeatability achieved by each laboratory.

Keywords: Proficiency test, UHT milk, total solids, total fat, standard deviation, systematic errors.

1. Introduction

This article describes the analytical results in total solids and fat content determination in UHT milk performed in the Analytical Laboratory of the Food Quality Department in the Institute of Food Safety and Veterinary in Tirana.

The Dairy Laboratory in Biotechnical Faculty-Podgorica, Montenegro. was nominated as "Pilot Laboratory" by the Laboratory of Metrology and Quality, Faculty of Electrical Engineering-Ljubljana, Slovenia, as coordinator of IPA 2008 Project "Regional Quality Infrastructure in the Western Balkans and Turkey". The participant laboratories, 8 at all, were all asked to carry out three tests in the same 7 samples: fat, protein and total solids. Each laboratory had its individual code. The code of our laboratory was L8. After getting back the results, the Pilot Laboratory did the calculations and statistical evaluation of all the results. The reference value used for these calculation and mentioned through out the paper, represents the average value of the eight participant laboratories. In calculating the averages, the organizing body first made the test for excluding the outliers according to the Cochran and Grubb's statistical tests. This article gives and analyses only the results achieved in total solids and fat content determination. Discussion of our results for total solids and fat content determinations are presented at the end of this paper together with our interpretation and explanation for the possible causes for the errors and deviations from the correct contents. We should underline that this PT helped us in identifying sources of errors even in such simple procedures such as fat determination in milk serving as a tool for analytical performance improvement under the frame of quality assurance management system. That was the aim of participation in this PT and the purpose of this paper as well.

2. Material and method

2.1 Test material

Seven samples of heat –treated milk (UHT milk) collected from the domestic market, preserved and stored at $+4^0$ C before dispatch to the participating laboratories were the test materials for this Proficiency Test (PT). The sample quantity was 100 ml/each. Organizing body of the PT sent samples to each participant after its homogeneity evaluation. Integrity of the samples during the whole distribution process

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up to the dispatch to the testing laboratories was guaranteed by packing in polyethylene capped vials in insulated boxes with cooling packs.

2.2 Submission of the results. The timeframe for reporting the analytical results was 5 working days starting from the moment of their arrival to the lab.

2.3 Distribution of samples and documents. Samples were accompanied by documents of general instructions for the participants, sample receipt form, report form of final results. Each laboratory was assigned a laboratory code, ours was L8.The participants were asked to carry out tests in duplicate for each sample and to report both of them as well as the analytical method used.

2.4 Analytical method

Total fat determination. Our laboratory used the routine Method "Gerber" (1,5) for the determination of total fat content in the 7 samples of milk. The method consists of introducing 10 ml of H_2SO_4 (d= 1.818 g/ml) into butyrometer followed then by pipetting 10.75 ml of milk sample into butyrometer carefully in order not to mix the milk with the sulphuric acid. Later 1 ml of amyl alcohol (d= 0.811 g/ml) is pipetted onto the milk. The butyrometer is closed with a stop without mixing the liquids. Shake the butyrometer vigorously until the liquids are thoroughly mixed. Then the butyrometer is placed in Gerber centrifuge (two tubes in two opposite sides). At the end of the proper time approx. 5 min the butyrometer is placed in heated water bath in 65°C for 5 min. Finally the scale reading is made.

Total solids determination. We used the Gravimetric Method (drying at 102 °C) for total solid content determination (2,4). An accurate quantity approx 5 ml of milk is weighed into pre - weight round flat bottom glass dish providing with a lid (5 cm diameter). The uncovered dish was placed on a boiling water bath until most of the moisture was driven off. The dish was transferred to a well ventilated oven at 102 $\pm$ °C. The dish with the lid apart were dried for 2 h in the oven and then the dish was covered with the lid cooled for 30 min in a desiccator and weighed. The dish and the lid was heated again for 30 min periods in the oven, cooled and weighed until the difference between the two successive weightings did not exceed 1 mg.

3. Results and discussion

3.1 Fat content

The results for fat content determination achieved from our lab are shown in the Table 1.The results of the samples showed that our lab had a good performance in precision better than half of the 8 labs, but there were evidence for some systematic errors. All samples results presented lower value compared to the reference values more or less at the same level showing problems with accuracy of our determinations. Table 2 gives other data necessary to interprete our results regarding repeatibility and accuracy. Regarding the accuracy, the statistical Student Test for $P < 0.001$(t_{test} =7.913> t_{tab}=5.96) showed the presence of significant systematic errors (Table 2).

Table 1 Results of fat content achieved from our lab in g/100 g milk for 7 samples

Sample	*1*	*2*	*3*	*4*	*5*	*6*	*7*
Test 1, g fat/100 g milk	2.60	3.40	1.50	1.10	2.70	3.20	1.50
Test 2, g fat/100 g milk	2.65	3.40	1.50	1.10	2.70	3.20	1.50
Average value	2.63	3.40	1.50	1.10	2.70	3.20	1.50
Reference value	2.74	3.57	1.57	1.17	2.82	3.30	1.65

Table 2 Data used for statistical evaluation of our PT results for fat content

Number of samples	*Difference between duplicates, min*	*Difference between duplicates, max*	*Absolute difference between duplicates*	*Repeatability standard deviation*	*Average value of all our duplicates*	*Average difference between our lab and reference value*
7	-0.05	0.00	-0.007	0.013	2.31	-0.1132

Anyway no outliers resulted based on Grubb and Cochran statistical tests for any samples tested. Repeatibility standard deviation was at 0.013 compared to 0.02 according to the required test method standard data (Figure 1).

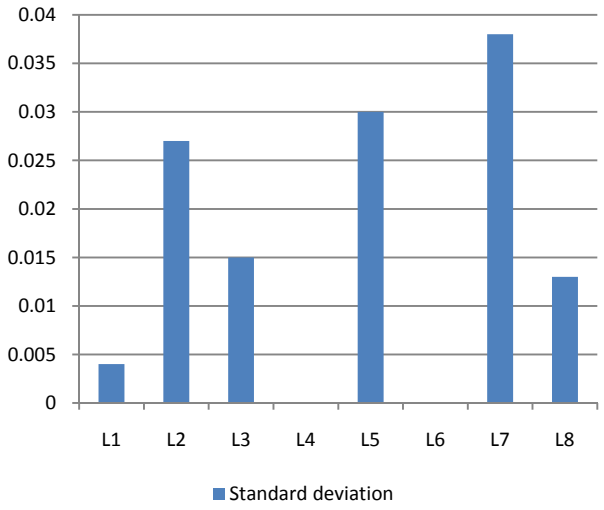

Figure 1. Graphic presentation of standard deviation for fat determination for 8 labs

3.2 Total solid content

The results for total solids content determination achieved from our lab are shown in the Table 3.The results of the samples show that our lab had a very good performance in precision better than half of the 8 labs, and what's more important there were no evidence for any systematic errors.All samples results presented slight higher or lower value compared to the reference values more or less at the same level showing a neglected presence of any random errors. Table 4 gives other data necessary to interprete our results regarding repeatability and accuracy.No outliers resulted based on Grubb and Cochran statistical tests for any samples tested. Repeatibility standard deviation was at 0.02 compared to 0.036 according to the required test method standard data (Figure 2)

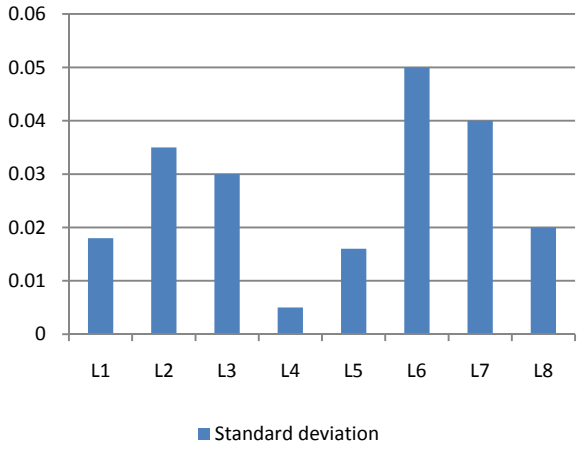

Figure 2. Graphic presentation of standard deviation of total solids determination for 8 labs.

Table 3 Results of total solids content achieved from our lab in g/100 g milk for 7 samples

Sample	*1*	*2*	*3*	*4*	*5*	*6*	*7*
Test 1, g total solids/100 g milk	11.30	12.0	10.25	10.29	11.60	12.05	9.73
Test 2, g total solids/100 g milk	11.30	12.03	10.22	10.24	11.57	12.03	9.74
Average value	11.30	12.02	10.24	10.27	11.59	12.04	9.74
Reference value	11.30	12.13	10.32	10.19	11.51	11.97	9.66

Table 4 Data used for statistical evaluation of our PT results for total solids content

Number of samples	*Difference between duplicates min*	*Difference between duplicates max*	*Absolute difference between duplicates*	*Repeatability standard deviation*	*Average value of all our duplicates*	*Average difference between our lab and value of reference*
7	-0.03	0.05	0.013	0.02	11.03	0.0152

Regarding the accuracy, the statistical Student Test did not show the presence any of significant systematic errors which is an optimistic result for this test. By analysing the Proficiency testing results for both tests, we concentrated ourselves in the systematic errors during fat content analysis in particular. In order to identify the possible causes we checked all the steps of the procedure. We noticed that all the results for fat content were lower than the reference values more or less at the same level. We checked the reagents used during the procedure, especially the concentration of sulphuric acid as the most probable cause for the problem. The concentration of the acid used was slightly lower than required. Another problem may have been the inexact temperature of the

water bath, both factors have influences in lower systematic results.

4. Conclusions

The Proficiency Tests are a very important tool for analytical laboratories to assure reliable and confident results.That PT our lab participated, proved to be very useful in understanding the possible sources of errors in total solids and fat determination by respective methods which served as well as internal reference methods because we used to determine fat content and total solid content in milk by Milk Analyser.

5. Acknowledgment

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