P40: PROFICIENCY TESTING ACTIVITIES; EXPERIENCE OF A LABORATORY FOR FOOD QUALITY CONTROL

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Abstract – The Institute of Public Health is the first public health institution in the FYROM that has established Quality Management System according to ISO/IEC 17025. The accreditation has been granted by the National Institute for Accreditation in December 2006. Since then, the maintenance and improvement of the Quality Management System remains a key element in our policy.

It is well-known that one of the elements by which laboratories can demonstrate their competence is through participation in Proficiency Testing (PT) activities. Therefore, our laboratory adopted practice to participate in at least one PT scheme relating to each sub-discipline per a year. Since the first accreditation, our laboratory participated in 15 PT schemes, organized by different providers, such as: Muva Kempten Quality Management & Laboratory Services; FAPAS; DRRR Deutsches Referenzbüro für Lebensmittel-Ringversuche und Referenzmaterialien; LGC; Asia Pacific laboratory Accreditation Cooperation etc.

The most frequently analyzed matrices were: cream, powdered milk, yoghurt butter, cheese, oily dressing, soft drinks, cabbage puree; while the most analyzed parametres were: fat and water content, dry matter, protein content, pH, benzoic acid, nitrates, caffeine, saccharine, potassium acesulfame, citric acid, fructose, glucose, sucrose, total sugars.

Total of 60 parameters were tested, and 49 from the obtained results (81.7%) were evaluated as satisfactory, 2 results (3.3%) were assessed as questionable, while 9 (15.0%) were evaluated as unsatisfactory. The investigation shown that unsatisfactory performance is most frequently due to the systematic errors, such as: improper matrix homogenization, incomplete extraction or inadequate drying and cooling of glassware to constant temperature.

Key words: Proficiency Testing Schemes; Food Quality Control; ISO/IEC 17025

1. INTRODUCTION

The National Institute of Public Health in Skopje is a leading institution of tertiary level for preventive healthcare in the country. Within the Institute there are laboratories – they control the quality and safety of food, water, air, radiation, epidemiology (microbiology and virology) as well as a laboratory for quality control of medicines. The Institute was the first public health institution in Macedonia which set up a quality management system according to ISO/IEC 17025. The accreditation certificate (LT-005, from 25.12.2006) has been granted by the National Institute for Accreditation of RM.

One of the technical requirements of the ISO/IEC 17025 for testing laboratories is to have "quality control procedures for monitoring the validity of tests." In other words, in order to prove the compliance with the requirement stated in 5.9 of ISO/IEC 17025, the laboratory has to perform quality control on every test within the scope of accreditation [1, 2].

Another critical requirement for accredited laboratory is to take part in relevant and obtainable proficiency testing study (PTS). Moreover, ILAC P9 requires that "Accreditation bodies (ABs) seeking to sign or seeking to maintain their status as a signatory to the ILAC Multilateral Recognition Arrangement (MRA) shall demonstrate the technical competence of their accredited calibration and testing laboratories. The satisfactory participation in PT activities, where such activities are available and appropriate, is one of the elements by which accredited laboratories could demonstrate technical competence" [1-3].

Additionally, the satisfactory results from PTS are an attestation of a laboratory's competence and, hence, an essential part of the assessment and accreditation process [2, 3].

Therefore, our laboratory adopted practice to participate in at least one PT scheme relating to

each sub-discipline per a year" [3, 5, 6]. Since the first accreditation, our laboratory participated in 15 PT schemes, organized by different providers, such as: Muva Kempten Quality Management & Laboratory Services, FAPAS, DRRR Deutsches Referenzbüro für Lebensmittel-Ringversuche und Referenz materialien, LGC, and Asia Pacific laboratory Accreditation Cooperation.

The matrixes analyzed were: coffee cream, cream, powdered milk, yoghurt butter; cheese; oily dressing, cola drink, cabbage puree, fresh cheese, orange juice, potable water, water real sample, animal feed cereal based, fish oil, maize flour, corn flakes, graham flour, synthetic food of vegetable and animal origin, homogenized canned fruit, powdered rice, dietetic product etc.

The parameters that were analyzed were: fat content, dry matter, water content, benzoic acid, nitrate content, caffeine, saccharine, K – acesulfame, protein content, pH, citric acid, fructose, glucose, sucrose, total sugars etc.

2. RESULTS AND DISCUSSION

2.1. Analysis of Fatty Food Matrixes

In the PT studies we have analyzed different fatty food matrices, most frequently: Coffee cream, cream, oily dressing, yoghurt butter; cheese with different fat content, powdered milk with different fat content, edible oil. We have tested 9 different parameters, such as: dry matter, fat content, free fatty acids, iodine value, peroxide value, protein content, saponification value, unsaponifiable matter and water content in 15 different samples. The total number of measurements was 38.

The z-score gives a bias estimate of the result. Absolute z-scores less than 2 are acceptable. A zone of doubtful performance exists for absolute z-scores between 2 and 3. An absolute z-score of 3 or more can be interpreted as unacceptable performance, requiring corrective actions. Corrective actions should also be triggered when z-scores are frequently in the doubtful zone or of identical sign" [3].

Table 1. The values for z score obtained in PT schemes with fatty food matrices

Parameter	Number of results	z-score		
		|z|* ≤ 2	2 < |z| <3	|z| ≥ 3
Dry matter	10	8	0	2
Fat content	13	7	1	5
Free fatty acids	1	1	0	0
Iodine value	2	2	0	0
Peroxide value	2	2	0	0
Protein content	5	5	0	0
Saponification value	1	1	0	0
Unsaponifiable matter	1	1	0	0
Water content	2	1	0	1
Ash	1	1	0	0
Total	38	29	1	8

* An absolute z-score

As it is shown in the Table 1, 29 results (76.3 %) have absolute z-score ≤ 2 (satisfactory); 1 of the results (2.6 %) has z score 2 < |z| < 3 and 8 of the results (21.1 %) have z score ≥ 3 (not satisfactory).

The investigation has shown a high percentage of unsatisfactory z scores (21.1 %) obtained in the quality control of fatty food matrices (especially in the determination of the fat content). This could be due to systematic errors, such as: improper matrix homogenization, incomplete extraction or inadequate drying and cooling of glassware to constant temperature.

2.2. Analysis of additive content and some routine components in soft drinks and cabbage puree

Cabbage puree, cola drink, orange juice, and cake mix flour were tested for the content of additives (acesulfame – K; benzoic acid; caffeine; saccharin; artificial colours (allura red, carmoisine, sunset yellow, erythrosine), nitrate content, Brix), while orange juice was also tested on the content on citric acid, fructose, glucose, sucrose, total sugars and pH value.

Number of the samples was 6, while number of the parameters tested was 17.

Table 2. The values for z score obtained in PT schemes with soft drinks and cabbage puree

Parameter	Number of results	z-score		
		|z|* ≤ 2	2 < |z| <3	|z| ≥ 3
Acesulfame Potassium	2	2	0	0
Benzoic acid	1	1	0	0
Sorbic acid	1	1	0	0

Caffeine	1	1	0	0
Saccharin	2	1	1	0
Artificial colours: Allura red Carmoisine Sunset yellow Erythrosine	4	2	0	2
Nitrate content	1	1	0	0
Brix	2	2	0	0
pH	2	2	0	0
Citric acid	2	1	0	1
Fructose	1	1	0	0
Glucose	1	1	0	0
Sucrose	1	1	0	0
Total sugars	1	1	0	0
Total	22	18	1	3

* An absolute z-score

At it is shown in the table 2, 18 (81.8 %) of the results have z score which is satisfactory ($z \leq 2$), only 1 result has z score that shows doubtful performance (4.5 %) and three results are unsatisfactory $2 < z < 3$ (13.6 %).

3. CONCLUSIONS

The laboratory for food quality control in Institute of Public Health, Skopje, for more than one decade, has participated in various proficiency testing schemes with different parameters as: fat content, dry matter, water content, benzoic acid, nitrate content, caffeine, saccharine, acesulfame potassium, protein content, pH, citric acid, fructose, glucose, sucrose, total sugars etc.

Measurements were performed in different matrixes as: coffee cream, cream, powdered milk, yoghurt butter; cheese; oily dressing, cola drink, cabbage puree, fresh cheese, orange juice, animal feed cereal based, fish oil, maize flour, corn flakes, graham flour etc.

The total number of tested parameters was 27. 47 (78.3 %) of the results obtained were with satisfactory z score ($z \geq 2$); 2 (3.3 %) of tested parameters had doubtful z score ($2 < z < 3$) and 11 (18.3 %) of tested parameters had unsatisfactory z score ($z \geq 3$);

The highest percentage of unsatisfactory z scores (38.5 %) and the highest unsatisfactory value for z score (15.7) was obtained for the determination of the fat content in fatty food matrices (coffee cream, cream, powdered milk, yoghurt butter; cheese and powdered milk). This is due to the systematic errors made during analysis performance as: improper homogenization of the matrix, shorter duration of Soxhlet extraction and errors made during drying and cooling of glassware to constant temperature.

The highest percentage of unsatisfactory z scores in analysis of in soft drinks and cabbage puree (50 %) were obtained for the analysis of artificial colors (Sunset yellow and Erythrosine) in soft drinks. The principal problems associated with the determination of colour additives in beverages are the mixtures of colours and the diversity of potential interferences present. The results demonstrated the need to improve the existing HPLC method in order to achieve better selectivity and separation, especially for analysis of sunset yellow and erythrosine.

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