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DIAGNOSTIC USE OF PROFICIENCY TESTING IN DAIRY LABORATORY

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ABSTRACT

The composition of raw milk (RM) is important for milk recording, herd improvement, payment of milk and for quality evaluation. The reliability of routine analytical results is therefore significant. A mistake occurrence could jeopardize the efficiency of results. Milk laboratories (ML) perform the systems of analytical quality assurance. Proficiency tests are carried out usually by 10 RM samples with modified variability of components. At evaluation the system of Euclidian distance ($Re=(\bar{d}^2+sd^2)^{0,5}$) for participants order according to their analytical result reliability is used. Two specificaly modified types (designs) of systematic diagnostical graphs were constructed on the Re basis. The first type of flow diagnostical diagram for RM analysators is based on Shewhart´s diagram principles for Re parameter (alternating system of result rendering before and after calibrations). The second type is based on analysator set comparisons before (proficiency test results) and after calibration (success of calibration) by Re. The opposite comparison is possible and valuable as well. It makes possible the diagnosis and incidental co-ordinating of the corrections in the case of the unconformity occurrence. The positive contributions of the developed diagnostic graphics system are expected to: – improvement of estimation of unconformity sources of RM routine analytical results, their origin and character; – determination and improvement of incidental result corrections and instrument repairs; – improvement of reliability of routine results of RM analyses in general.

Key words: milk / dairy laboratories / proficiency testing / quality

DIAGNOSTIČNA UPORABA PREVERJANJA USPOSOBLJENOSTI MLEKARSKIH LABORATORIJEV

IZVLEČEK

Sestava surovega mleka (SM) je pomembna za kontrolo proizvodnje, selekcijo, plačevanje in vrednotenje kakovosti mleka. Zanesljivost rutinskih metod je ključnega pomena, ker bi napake ogrozile verodostojnost rezultatov. Mlekarski laboratoriji vpeljujejo sisteme za zagotavljanje kakovosti analitskih postopkov. Preverjanje usposobljenosti se izvaja navadno na 10 vzorcih SM z različno vsebnostjo osnovnih sestavin. Za preverjanje zanesljivosti analitskih rezultatov se pogosto uporablja sistem Evklidskih distanc ($Re=(\bar{d}^2+sd^2)^{0,5}$). Na osnovi Re sta bili zasnovani dve modifikaciji modela diagnostičnih grafov. Prvi tip diagramov poteka za analizo SM temelji na Shewhart ovem diagramu za parameter Re (sistem za določitev rezultata pred in po kalibraciji). Drugi tip temelji na primerjavi serij rezultatov pred in po kalibraciji z uporabao Re. Primerjava omogoča diagnozo in korekcije v primeru neskladja rezultatov. Od razvoja diagnostičnega grafičnega sistema si obetamo izboljšano določitev virov in narave neskladnosti analitskih rezultatov rutinske kontrole sestave SM, izboljšanje in korekcij rezultatov in instrumentov ter izboljšanje zanesljivosti rutinski rezultatov analize SM na splošno.

Ključne besede: mlekarstvo / laboratoriji / usposobljenost / preverjanje / mleko / kakovost

INTRODUCTION

The main composition and properties of raw milk and their testing systems are very important: – for performance of the milk recording; – for monitoring of dairy cow health state and controlling (prevention and treatments of the dairy cow production disorders); – for milk quality evaluation; – for animal genetic appreciation and improvement (cattle breeding); – for milk quality payment; – for dairy processing; – for foodstuff chain safety in general. The milk foodstuff chain is one of the most monitored and controlled foodstuff chains in terms of: – the number of routinely checked milk parameters (microbiological, compositional, technological); – regularity and relatively high frequency of the mentioned investigations; – mostly biological character of the mentioned investigations. In accordance with such information, there are assumptions, that the milk foodstuff chain could be one of the most safe of all known foodstuff chains. Therefore, the reliability of the referential and routine milk analytical results is very important as well. Incidental mistake occurrence could jeopardize the efficiency of the dairy production.

In the framework of supporting of the mentioned facts the milk laboratories carry out the accreditation according to the international standard (EN ISO/IEC 17025) in the Czech Republic. The referential and routine milk laboratories perform**s** the systems of the analytical quality assurance of the calibrations and measure functions of the master and routine milk analysators as well. It means, laboratories improve the analytical result**s** reliability runningly. A similar situation is at using of all referential and routine milk analytical methods in general. The last development works about above mentioned topics are introduced in many scientific and professional papers (Grappin, 1985, 1993; Hanuš and Ficnar, 1990; Hanuš and Kaššovicová, 1992; Leray, 1993; Wood, 1994; Golc-Teger, 1996, 1997; Hanuš *et al.*, 1996, 1998, 2000, 2001, 2002; Klopčič *et al.*, 1999). Mentioned systems are currently developed and improved also in the framework of the projects MZe-ČR, NAZV, QF 3019 and MŠMT-ČR, INGO, LA103.

MATERIAL AND METHODS

The Czech milk referential and routine laboratories (laboratories of the milk recording = individual milk samples; central laboratories = bulk milk samples) take part regularly in the interlaboratory proficiency testing on the national and international levels as well. RICB Rapotin organizes proficiency testing of the routine milk recording laboratories for some raw milk components: fat (F); protein (P); lactose (L); urea (U). Tests are performed on the basis of ten raw milk samples with necessary modified variability of the relevant introduced components.

The calculation system of the Euclidian distance to the origin (Re; according to Leray, 1993; Fig. 1) is used. Euclidian distance represents the distance of each laboratory to the origin (0,0) corresponding to the reference. Re is used for evaluation of the proficiency testing results and for order of the participants (laboratories) according to accuracy and reliability of their analytical results for the mentioned purposes. The Euclidian distance (Leray, 1993) from the origin calculation system is prefered for the mentioned purposes in comparison for instance to the often used Z-score system (Wood, 1994) because of its advantage to separate an incidental analytical mistake into two parts: – systematic error part and random error part. Such a differentiation ability could be important for suggested necessary diagnostical purposes at analytical mistake investigation, identification and specification.

The design of diagnostical diagram system for a better identification of mistakes in milk analysis and specification was proposed, created and developed according to: – results of twenty two proficiency tests performed during the year (2002 and 2003) by RICB referential milk laboratory; – knowledge of result variability of reliability parameter (Re) in regularly (monthly) performed proficiency tests; – knowledge of result dynamics of reliability parameter in regularly performed proficiency tests; – knowledege and consideration about interpretation efficiency of different evalution systems (such as Re, Z-score, Youden plot, Shewhart´s diagram) for interlaboratory proficiency testing results in terms of their real graphical ability to analytical mistake investigation, identification and specification.

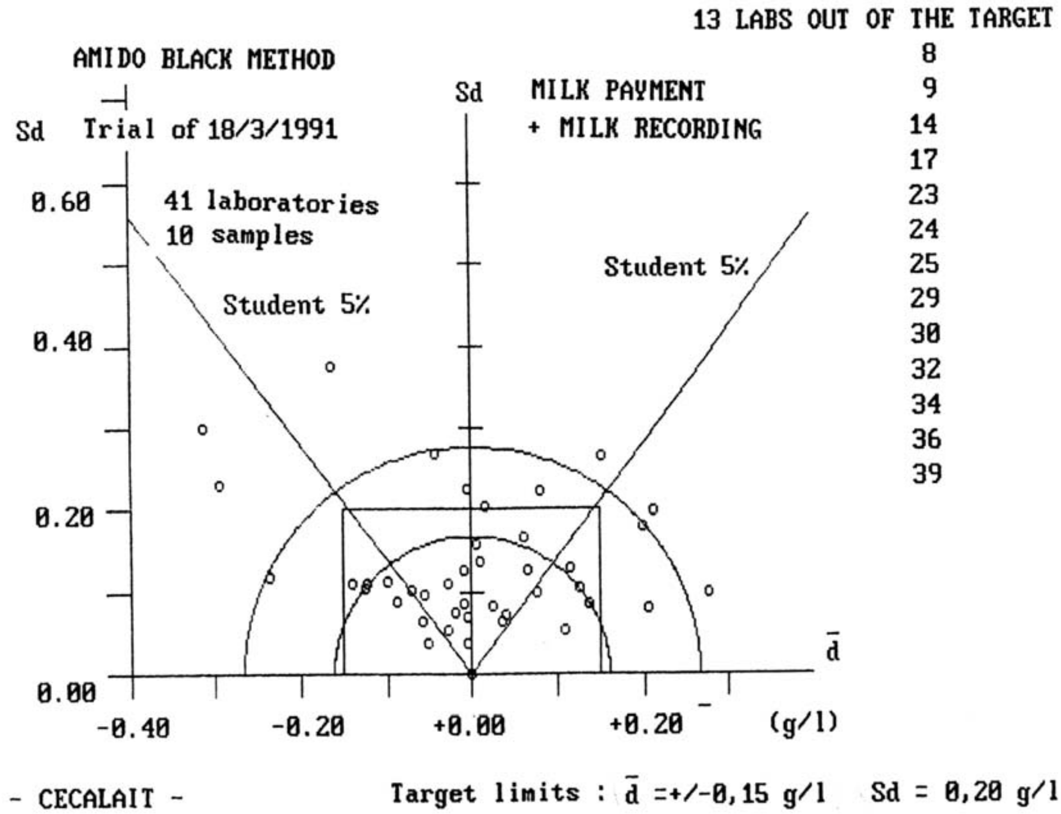

Figure 1. The expression system of Euclidian distance to the origin (Re) as accuracy parameter of milk analytical results in the proficiency testing (according to Leray, 1993).

RESULTS AND DISCUSSION

The diagnostical diagram systems for raw milk analysators were constructed as specific purpose modifications on the basis of the Shewhart´s diagram principles (Kupka, 1997) for the mentioned Re system (Fig. 2) and Re time diagram reciprocal comparisons (Fig. 3). The Re discrimination limits (HDLRe) were calculated for F, P, L and U according to long-term analytical result rows (2 years = 22 tests at month intervals) for Fig. 2 and according to results of instrument sets of proficiency tests and calibration process (Fig. 3; the half circle lines). The graphs of the individual instruments (Fig. 2), and calibration process and proficiency test comparisons (Fig. 3) are accessible for workers of the routine milk laboratories (workers of laboratory network) in the electronical way:

- the Re situations (Fig. 2) before and after calibration are shown by the alternating system of the symbols (○×○×…). It makes possible the diagnose and incidental co-ordinating of the necessary corrections in the case of the result unconformity occurrence. It is possible to show separately two components of the instrument result reliability (Re): the mean difference ($\overline{d}$); the variability of the differences (sd); by the same type of the graph as well. Such projection improves the diagnosis of incidental occurrence of the result error in terms of the effect estimation of the systematical and random error parts on the total value of the unconformity. There are the protein graphs chosen for four types of the instruments (Fig. 2) for instance:

- master instrument of the referential-routine laboratory system (network) with good stability in the referential laboratory;
- routine instrument with good stability in the routine laboratory;
- routine instrument with usual stability in the routine laboratory;
- routine instrument with unconformity occurrence;

- further, the mentioned modificated system shows the situations of the whole set of the analysators according to Re (Re=$(\overline{d}^{2}+sd^{2})^{0,5}$; by the original Re rendering (according to Leray, 1993) Fig. 1) by the mutual comparison of two graphs after the last calibration and before the next calibration (×○). The opposite comparison (○×) is possible and very valuable for relevant analytical mistake diagnoses as well (Fig. 3). Such rendering improves the further diagnostical possibilities for investigation, identification and specification of incidental unconformities.

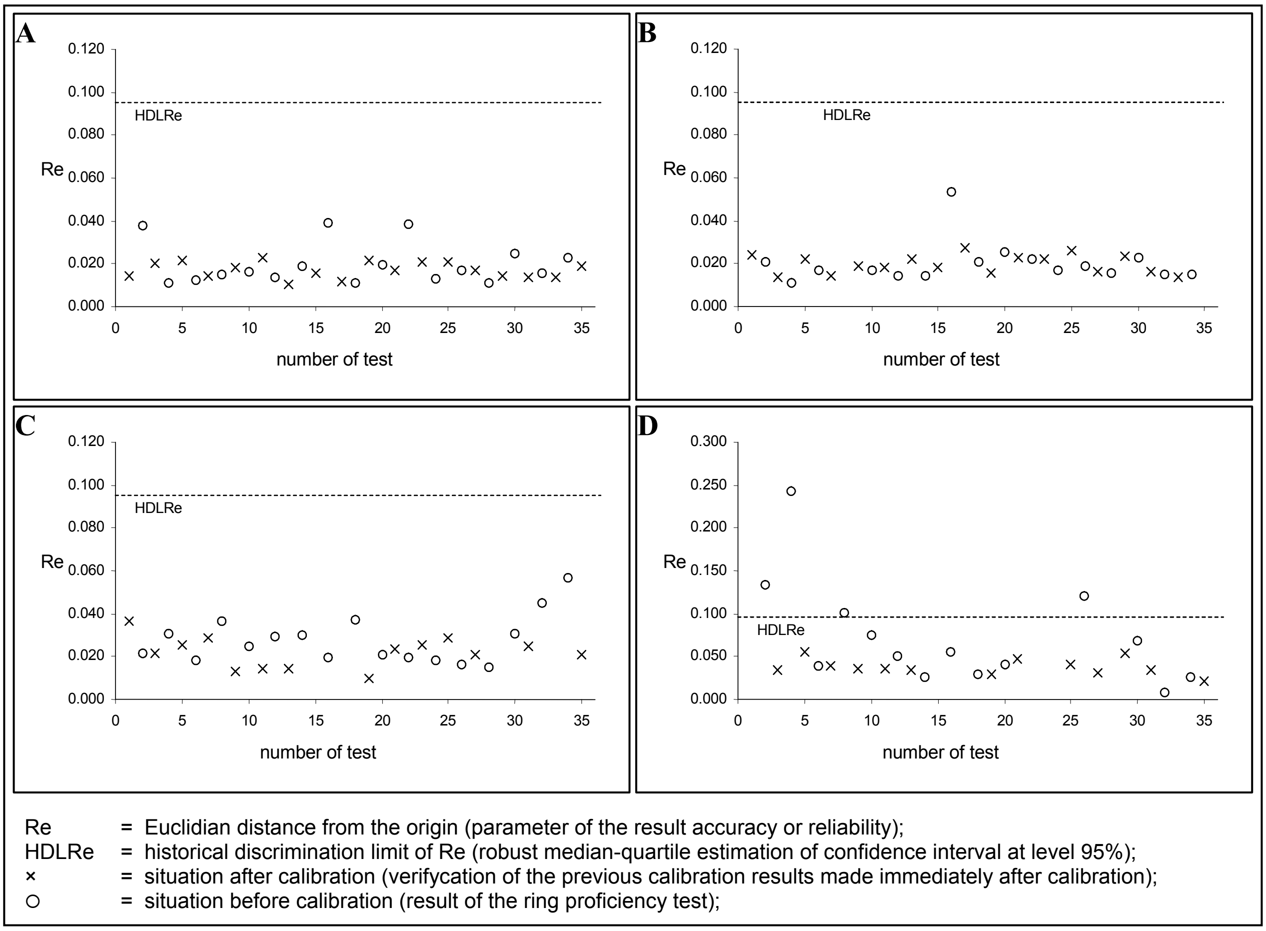

Figure 2. Individual diagnostical flow graphs for protein content at instruments (A,B,C,D) with different measurement stability.

The results of the proficiency testing are anonymous, of course. Nevertheless, if the laboratory managers know the key numbers of their own instruments and further they can compare the result reliability (accuracy) situations by different types of mentioned developped diagnostical graph combinations, they could be able to guess the type, character and source of incidental analytical mistakes according to their own knowledge about analytical method principles and simultaneous laboratory facts.

CONCLUSIONS

It is expected, that the mentioned developped diagnostical graphical system for the analytical mistake identification and speification will contribute positively in the milk referential-routine laboratory networks to:

- improve of the estimation of unconformity sources of the raw milk routine analytical results in terms of their origin and character;
- determine and improve the incidental corrections of the results and necessary reparations of the instruments;
- improve the quality (reliability) of the routine results of the raw milk analyses in general.

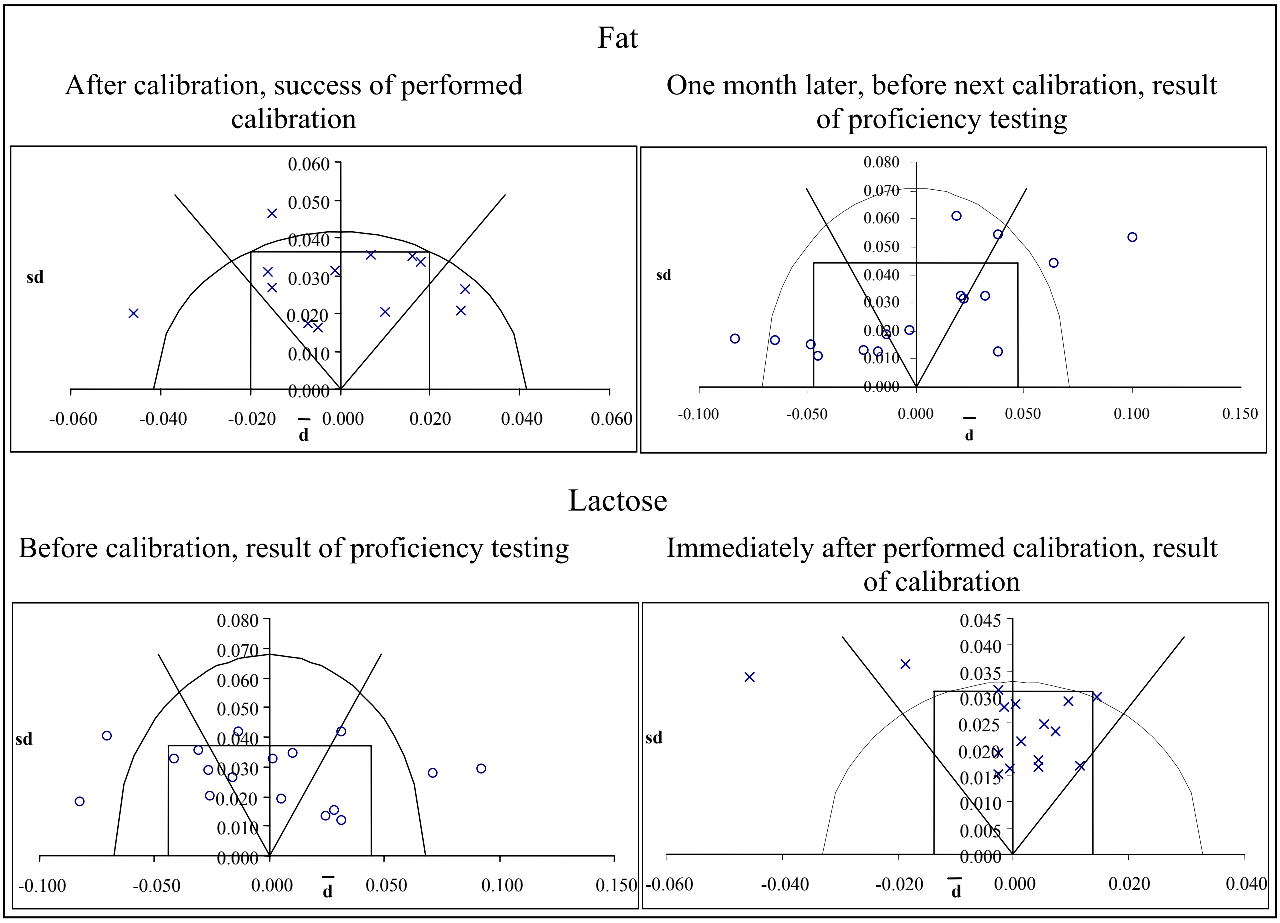

Figure 3. Pairs of group diagnostics comparison graphs (fat: comparison of analysator group after calibration and before next calibration by two different sets of milk referential standard samples; lactose: comparison of analysator group immediately before calibration and after calibration by the same set of milk referential standard samples).

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