ELSEVIER

Food Chemistry 82 (2003) 121–124

Food Chemistry

www.elsevier.com/locate/foodchem

Control of the water content of dairy products—definition of limits, consideration of process variation, official use of autocontrol data

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Abstract

The control of the water content of dairy products is of considerable economic importance. Ideally, it should be possible to evaluate the water content of consignments, or even products, made within an extended time period. In the real world, official control results may often just allow statements on sample units. As the water content of many dairy products is adjusted to a certain level, determined by legislation, control results reflect process conditions and measurement uncertainty. Both sources of variation are to be taken into consideration when defining compliance with legal limits. On the other hand, it is hardly possible to cover these aspects by official analysis. Control authorities must have access to the autocontrol data of the producer. An adequate concept for future official controls includes the use of this information and regular checks of its reliability. Consequently, official control would focus on reliability checks rather than direct controls of the water content of dairy products. When controlling drinking milk, the relevant aspect is not the water content of this product but the percentage of added water. It can be determined by a comparison with the composition of raw milk used for drinking milk production. A statistical approach is required.

Keywords: Dairy products; Water content; Added water; Definition of limits; Measurement error; Process variation; Autocontrol

1. Introduction

At the first glance, legislation regarding the water content of dairy products appears to be a fairly simple exercise: specify a limit and define a reference method. Furthermore, make sure that samples are taken using adequate techniques and that laboratories with proven competence carry out the analysis. Results below the limit show compliance with the legal requirement, results above the limit non-compliance. Measurement error is very often not addressed; however, it will probably be accepted to include this aspect, when drafting relevant future legislation.

A very important aspect is usually neglected, when using this approach: we are interested in the water content (arithmetic mean, variation) of a large amount of product, e.g. a consignment, a product made on a given day or even a product made within an extended time period. When measuring the water content of one or a few sample unit(s), how certain can we be certain that this aspect is adequately covered? The water content of products such as butter or milk powder inevitably shows some variation due to process conditions. When making statements on large quantities of a product, this variation has to be measured or a reasoned assumption on its extent has to be made.

A special problem is the determination of added water in drinking milk. Usually a limit for the freezing point is established to prevent water addition during milk processing. However, as the freezing point of raw milk shows some variation, this is not an adequate approach. Controls should therefore be based on a comparison with the freezing point of raw milk. This principle has been established by legislation in the EU (Council Regulation No. 2597, 1997).

How can compliance with legal requirements be checked under these circumstances? In the following sections an attempt is made to answer this question.

2. Definition of limits

Even if processing conditions are carefully controlled, the water content of dairy products will vary to a certain extent. This variation can be characterized by a process standard deviation. The producer has to take this standard deviation into consideration when adjusting the process (European Commission, 1999). A precise definition of limits established by legislation will help him

0308-8146/03/$ - see front matter
doi:10.1016/S0308-8146(02)00586-1

Table 1
Definition of limits

Compliance with an upper limit (U) for the water content	
$\mu \leqslant U - 1.645\ \sigma_p$	
μ	True arithmetic mean of the water content
σ_p	Long-term process standard deviation of the water content
1.645	Factor to be used if "95% compliance" is required (probability to obtain complying result: 95%)

to do this adequately. As "100% compliance" is an unrealistic requirement which cannot be controlled, a statistical approach should be envisaged. When (in theory) a large sample representative for the product quantity to be controlled is taken and analysed at least x% of the sample units should give acceptable results (Table 1).

Various aspects (consumer protection, production costs, energy consumption) are to be taken into consideration before a suitable value for x can be fixed (Table 2).

The process standard deviation describes the variation of the water content due to slight variations of the process conditions. When measuring the standard deviation, the values obtained reflect process variation and measurement error. Consequently, a correction (elimination of the repeatability variance component) has to be made when determining and reporting the "true" process standard deviation (Table 3).

3. Control aspects

Ideally, official control should provide information on arithmetic mean and standard deviation of the water content. However, it would be unrealistic to assume that proposals to cover this latter aspect would be accepted. A dramatic increase in sample sizes would be the consequence, if the process standard deviation would have to be determined. Furthermore, the interpretation of the figures obtained would not be easy.

As a consequence, a different strategy has to be developed:

Table 2
Upper limit for the arithmetic mean—a function of the compliance criterion

Accepted percentage of non-complying results	Upper limit for x (%)
10%	15.74
5%	15.67
1%	15.53
0.10%	15.38
0.01%	15.26

Example: Butter, water content. Process S.D.: 0.20%.

Table 3
Calculation of the "true" process standard deviation

$\sigma_p = \sqrt{\sigma^2 - \sigma_r^2}$	
σ	Process standard deviation including the repeatability standard deviation, σ_r

Remark: for practical reasons it may be preferable that the producer uses σ when controlling the production process. σ_p is used by the control authority.

- Control of the arithmetic mean using the conventional approach (i.e. analysis of a random sample; composite samples can be used)
- Alternative procedure for the control of the process standard deviation (see next section).

The producer has to adjust the process in a way which guarantees that an upper limit for the arithmetic mean (defined by the process standard deviation and the compliance criteria used for the definition of the legal limit) is not exceeded (Table 4). Control results, not exceeding this upper limit, show compliance with the legal requirement. Results exceeding the upper limit have to be interpreted taking "sampling error" and analytical error into consideration (Table 4). The control principle described here is applied in recent legislation (Commission Regulation No. 970, 2000).

4. Use of autocontrol data

With sample sizes regarded as "realistic", an adequate control of the arithmetic mean may be possible if composite samples can be analysed. The process standard deviations cannot be controlled at all under these circumstances. An alternative strategy is therefore needed. Control data obtained by the dairy should fulfil the requirements regarding sample size.

In principle the source of information needed for an alternative approach is already available. There are three important aspects:

Table 4
Official control

Interpretation of control results taking sampling error and analytical error into consideration	
$\bar{x} \leqslant \mu \Rightarrow$ Compliance	
$\bar{x} > \mu \Rightarrow$ Further investigation	
$\bar{x}$: arithmetic mean of the offical control results	
$\bar{x} \leqslant \mu + 1.645\sigma_{\bar{x}}$	$\sigma_{\bar{x}} = \sqrt{\frac{\sigma_p^2}{n} + \sigma_L^2 + \frac{\sigma_r^2}{n_1}}$
σ_L = between-laboratory standard deviation	n_1 = number of composite samples
$\sigma_L = \sqrt{\sigma_R^2 - \sigma_r^2}$	n = sample size
σ_R = reproducibility standard deviation	
σ_r = repeatability standard deviation	

- the control data are to be made available to the control authority on request
- the control data must be reliable
- a long-term process standard deviation can be determined on condition that the process is under statistical control.

The reliability aspect is of special importance. How can the control authority be sure that the data produced by the dairy reflect reality?

Firstly, the dairy control laboratory must be able to produce reliable results. This capability can be demonstrated by inter-laboratory comparisons. Ideally, it is demonstrated by a regular successful participation in proficiency tests. A definition of the term "successful participation" would be needed.

Secondly, the data presented by a dairy capable of producing reliable control data must in fact be the data obtained, when controlling the production process and not data "produced for the control authority". This is probably the most difficult element of the new approach.

It is not possible to present a final solution. However, some strategies can be outlined:

- It should be possible to allocate control data to final products stored in the dairy or found in a supermarket. Samples from these products can be analysed and the results compared with the relevant dairy control results.
- The determination of the process standard deviation is supervised by the control authority (sampling and analysis in the presence of an inspector).
- The difference between two composite samples, each taken randomly, is used to control the reported process standard deviation.
- Information on process standard deviation figures obtained in different dairies is collected by the control authority. Dairies reporting "suspect" figures can thus be identified. Special control measures may be envisaged in these cases.

5. Control of the "added water" content of drinking milk

When drinking milk is produced, a small amount of water is unavoidably added during processing. This is due to the fact that the processing equipment is not completely dry. However, a distinction has to be made between "unavoidable" water addition and water addition resulting from lack of care or from fraudulent activities. An adequate control has therefore to be established.

It has already been mentioned that a control should be based on a comparison of the composition of raw milk and the composition of drinking milk from this raw milk. Several parameters can be used for that purpose, for example, the

- freezing point
- protein content
- lactose content.

A number of different aspects has to be considered, when selecting the appropriate control procedure:

- natural variation of the analyte
- precision of the control method
- effect of processing on the results obtained
- manipulation possibilities

An important practical problem is the determination of the average composition of the raw milk used for drinking milk production. A combination of autocontrol data obtained by the dairy and official control data obtained at the farm level appears to be the appropriate way of tackling this problem.

The control principle is at present being examined with respect to the establishment of a future strategy for the control of the protein content of milk powder.

It may appear that the conventional approach (comparison with a limit for the freezing point) is much easier and therefore preferable. However, a comprehensive discussion of the problem leads to the conclusion that the procedure outlined above is much more appropriate. We have to be aware that certain analytical problems cannot be tackled in the conventional way by comparing a result with a limit established by legislation.

6. Conclusion

6.1. Control of the adjusted water content

Compared to the present system, the new approach for the determination of the water content of dairy products offers major advantages:

- limits for the water content established by legislation are clearly defined
- there is comprehensive information on product composition
- due to the broad data basis, adequate decisions on compliance/non-compliance can be made
- the new approach does not lead to an increased work load for the producer and the control authority

The latter statement requires some explanations. We are considering industrial production. It can therefore be assumed that statistical process control is already established by the dairies (if not, it can easily be done).

The new element is the data transfer from the dairy to the control authority. Again this is not a time-consuming activity.

From the point of view of the control authority, activities will change to a certain extent. Apart from an initial phase, where inter-laboratory comparisons may have to be organized (if proficiency test data are not available), the analytical control may be at the same level or even less intensive. The aim is different: the control authority would no longer perform compliance testing, but verify the reliability of the data presented. This verification includes analytical and further activities, such as those described in the previous section. The new system is transparent for both sides, the producer and the control authority. In addition, it offers a solid basis for decisions on compliance.

6.2. Added water in drinking milk

An adequate control of drinking milk for added water requires a new approach:

- "Unavoidable" water addition has to be defined for each type of drinking milk
- Natural variation of raw milk composition has to be taken into consideration
- Decisions on acceptability are to be based on statistical procedures.

This new approach requires a new way of thinking, but offers a considerable advantage: it is fair for the producer and the consumer; at the same time it reduces the risk of fraud.

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