ARTICLE

DETERMINATION OF FAT CONTENT IN CHEESE
Method comparison overview based on Cecalait® proficiency tests results

Two main principles are generally used for the determination of fat content in cheese:

- **The first principle involves extraction following acid attack: the extraction reference method, SBR (Schmid-Bondzynski-Ratzlaff), is standardised at international level: ISO 1735 / IDF 5.**
- **The second is acido-butyrometry, which involves two distinct methods: the Van Gulik (VG) method and the Heiss method. Both these methods are standardised at French level with (AFNOR) NF V 04-287: part 1 for the Van Gulik method and part 2 for the Heiss method. The Van Gulik method is more often applied to fromage frais, soft cheese and processed cheese. The Heiss method is principally applied to hard cheese and hard cooked cheese (as the higher dissolution temperature, 85°C, is more appropriate for this type of cheese). The butyrometers are the same for both these methods are also standardised at French level (NF B 35-530).**
  NB: The Van Gulik method is also standardised at international level (ISO 3433).

The relation between both these methods has been observed for many years and a difference between the acido-butyrometric methods and the extraction method has always been noted. The AFNOR V 04 milk and dairy products Commission has integrated this project into its work programme, firstly to evaluate the deviations with different types of cheese, and secondly to find solutions.

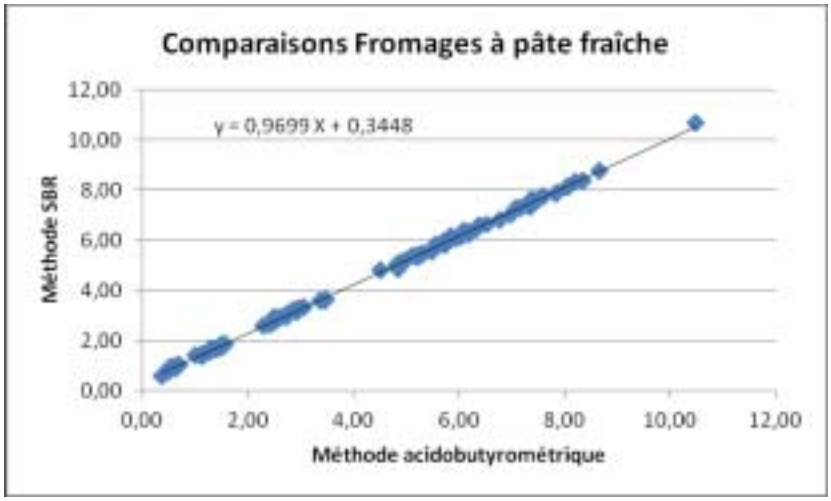
A first inventory was carried out on the basis of Cecalait® proficiency tests results from 2003 to 2011 on three types of cheese: fromage frais, soft cheese and hard cooked and non-cooked cheese. In practice, the laboratories use either the SBR extraction method or a butyrometric method (the method applied depends on the cheese analysed: mainly VG for fromages frais, Heiss for hard cheese and both methods for soft cheese). Nevertheless, within the context of proficiency tests, the Heiss method is being increasingly used by laboratories due to the more rapid dissolution of the cheese in the butyrometer and its greater reliability.

The statistical data from tests carried out for each group of methods (extraction and acido-butyrometric) between 2003 and 2011 were used for the comparisons described below. The Van Gulik and Heiss methods were grouped in an "acido-butyrometric" reference, even if the mean difference of each method in relation to the extraction method is slightly different (about 0.1 g/100 g). This approach was chosen firstly because the traceability of the method used is not always ensured, and secondly, differentiated action in the long run within the regulatory framework will be undoubtedly difficult considering the same butyrometer is used for both methods (and is partly responsible for the difference).

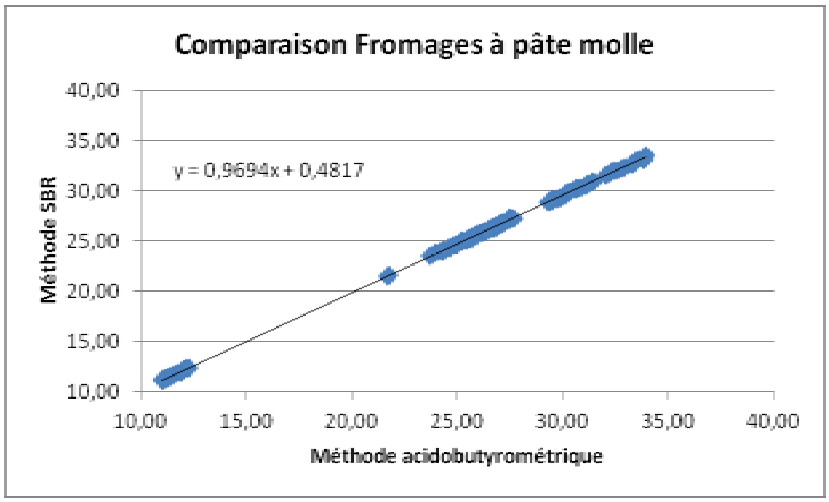
Firstly, comparisons of data were made per cheese type, then with all the cheeses on the basis of a simple linear regression with the SBR extraction method being taken as the reference (Y), and the acido-butyrometric methods (X) as the methods to be evaluated.

- **<u>Fromages frais</u>**

The regression equation Y = 0.9666 X + 0.3448 was obtained between the methods (see graph below) with a mean difference of –0.22 g of fat/100 g for this type of cheese (Extraction: 4.24 g/100 g – Acido-butyrometry: 4.02 g/100 g).

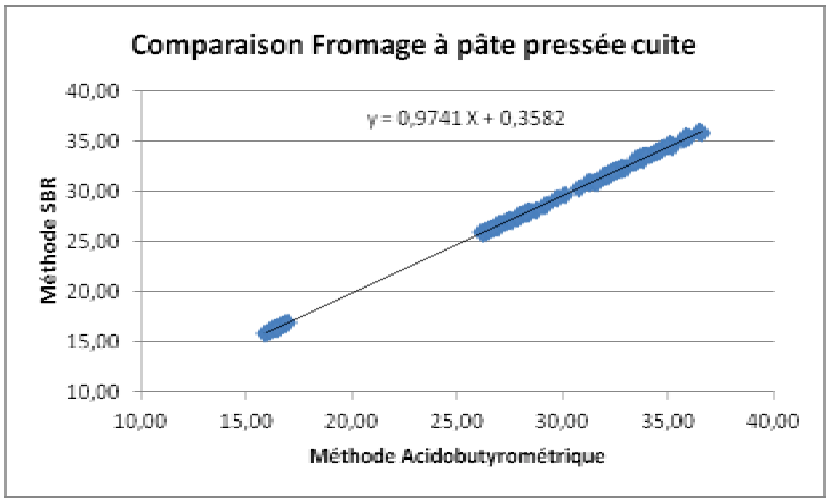


- **Soft cheese**



The regression equation Y = 0.9694 X + 0.4817 was obtained between the methods (see graph above) with a mean difference of +0.30 g of fat/100 g for this type of cheese (Extraction: 25.23 g/100 g – Acido-butyrometry: 25.53 g/100 g)

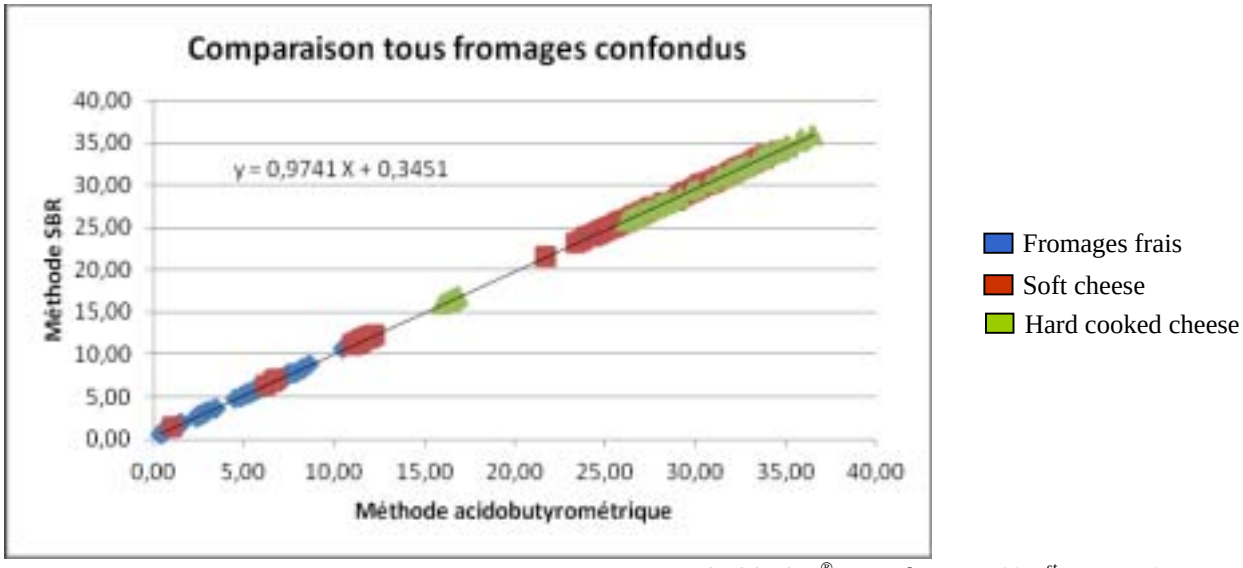
- **Hard cooked cheese**



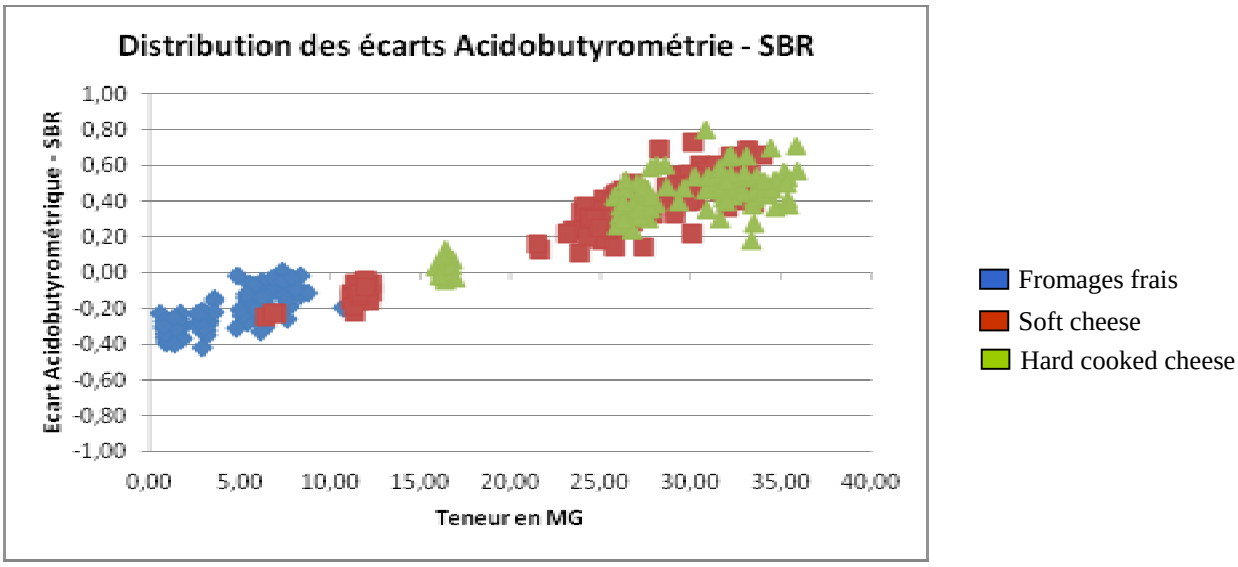
The regression equation Y = 0.9741 X + 0.3582 was obtained between the methods (see graph above) with a mean difference of +0.39 g of fat/100 g for this type of cheese (Extraction: 28.56 g/100 g – Acido-butyrometry: 28.95 g/100 g)

- **All the cheeses**

  - *Total regression*



- *Diagram of residuals*

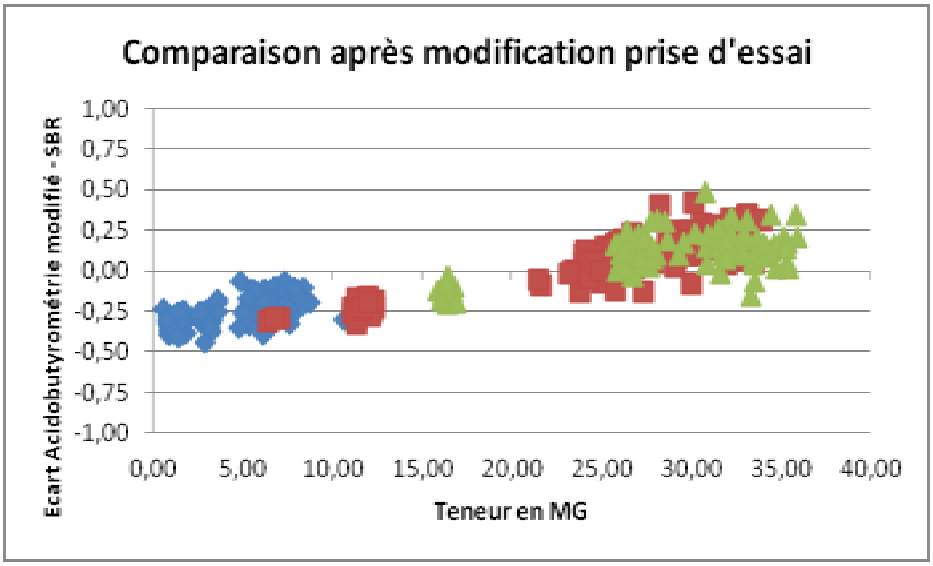


When all the various cheese types were integrated in the regression, an equation between the methods Y = 0.9741 X + 0.3451 was obtained with a mean deviation of +0.18 g / 100 g. Deviations from –0.40 g / 100 g to +0.80 g / 100 g of cheese were observed which are closely linked to the fat concentration of the product.

If we look at each regression equation obtained independently for the three types of cheese, the regression slopes are very close, almost equivalent to the regression slope obtained with the data from all the cheeses (even if the mean differences are different). It can be concluded that the difference observed between both methods is linked entirely to the fat content of the product and is independent of the cheese type (no specific difference with the type of cheese was observed).

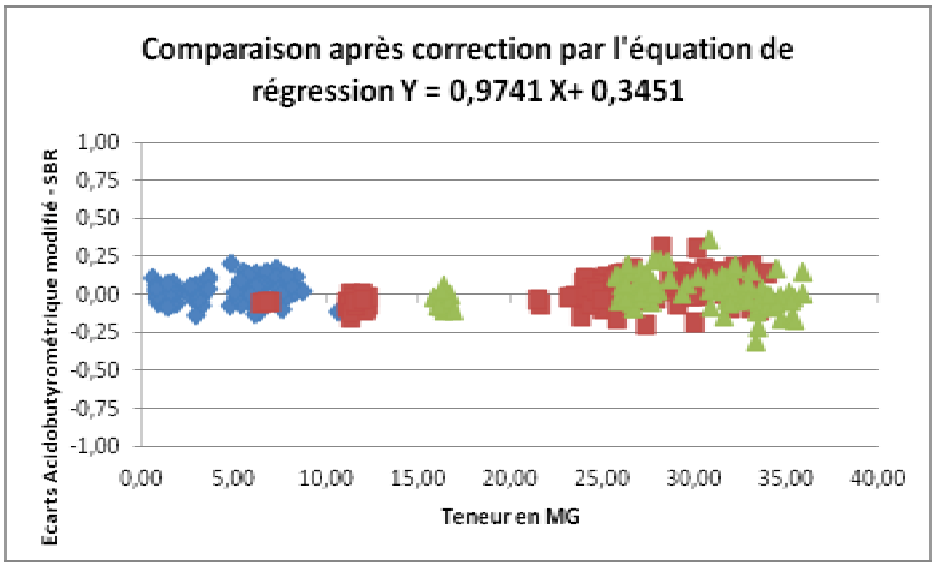
On the basis of these results, two working hypotheses have been examined to propose a solution to link the acido-butyrometric methods to the extraction method:

- To adopt a different test sample of 3 g, calculated according to the mean difference observed between the methods to obtain a mean difference of zero (2.972 g rounded down to 2.970 g for this study). A corrective factor was then applied to the results obtained using the acido-butyrometric method and we observed the deviations between the methods. These data are presented in the graph below:



Although after correction of the results an improvement and a decrease in the deviations between the methods was observed (about -0.40 to +0.40 g /100 g), this type of modification to the operating procedure does not enable the difference observed to be resolved over the entire fat content range within the field of application of the method tested.

- To apply a correction equation on all the cheeses and on the entire fat content range (0 to 40 g / 100 g).



Applying a correction equation ensures the traceability between the methods in all fields of application of the method. No systematic difference specific to cheese types tested was observed. The accuracy estimate (calculated by 2.Sy,x: residual standard deviation of regression) of the acido-butyrometric method in relation to the SBR reference method is ± 0.18 g / 100 g.

CONCLUSION

Both these working ideas offer advantages and disadvantages:

- **With both these solutions, it is not necessary to modify the graduations of the butyrometers and thus they do not need to be changed in every laboratory.**
- **A modified test sample can only be applied to a very small fat content range. Then, the slope error of the acido-butyrometric method cannot be corrected (specific test sample per low range of fat content according to the product).**
- **Applying an equation (which is not easy in practice) ensures the link between the methods over the entire fat content range for all the cheeses. This type of correction is already applied for the determination of the fat content in skimmed milk.**

The results obtained were presented and both these working ideas examined at the AFNOR Commission in March 2012. Discussions are continuing in order to ensure the correlation between both methods in 2012.