BUTYROMETRIC METHODS FOR MILK AND CREAM
- An international standardisation project -

Butyrometric methods are widely used in France and internationally for the determination of fat content in milk and dairy products. These methods are quick and do not require any solvents.

There are currently two international standards (quite old documents) on this subject:
- **ISO 488 for milk butyrometers,**
- **ISO 2446 for butyrometric method applicable to milk.**

Through the AFNOR commission V 04A, France has very carefully studied and reviewed these methods, over the past few years, integrating their adjustments to the corresponding reference methods.

- o In 1989, AFNOR reviewed the butyrometric method for cream (NF V 04-263) and the associated butyrometers (NF B 35-540) and adjusted the internal volume of the butyrometer (NF B 35-540) so it corresponded to the reference method ISO 2450.
- o In 2002, AFNOR also reviewed the Gerber method for milk and introduced a new criterion concerning the composition of the amylic alcohol used (isomeric ratio) in order to harmonise and adjust the butyrometric method to the reference method ISO 1211.

Thanks to this competence as well as the sustained, increasingly international use of these methods, the AFNOR V 04A standardisation commission and the IDF-France analytical commission proposed to the IDF and ISO to review the standards concerning the Gerber method. They will propose a supplementary document concerning the pipette used and the creation of two international standards concerning the butyrometric method for cream.

The first discussions at international level were held during the IDF analytical week in Montreal in 2010. There seems to be considerable interest in these methods and many countries backed this approach. At the time of the discussions, a question about the influence of the matrix on both these methods cropped up and it was decided, during the meeting of the SCAMC standing committee (major and minor components), to carry out investigations before the official proposition of the methods.

Many investigations have also been carried out at Actilait (Poligny) to reply to this demand and the results were presented during the IDF analytical week in Lyon from 25 to 29 May 2011.

1) "INFLUENCE OF THE MATRIX" TEST FOR MILK

For the method on milk, a specific test was carried out on 10 milk samples from different European countries (Germany, Belgium, Switzerland, France, Greece and Israel) preserved with Bronopol (0.02%). The analyses were performed in duplicate at Actilait's laboratory in Poligny according to the Gerber (NF V 04-210) and Röse-Gottlieb (ISO 1211) methods. The conversion g/L to g/Kg was achieved using the density.

The results obtained are described below (regression line and statistical parameters):

BUTYROMETRIC METHOD vs. ROSE-GOTTLIEB METHOD FOR MILK - (SPECIFIC TEST)

$y = 0,991x + 0,1094$
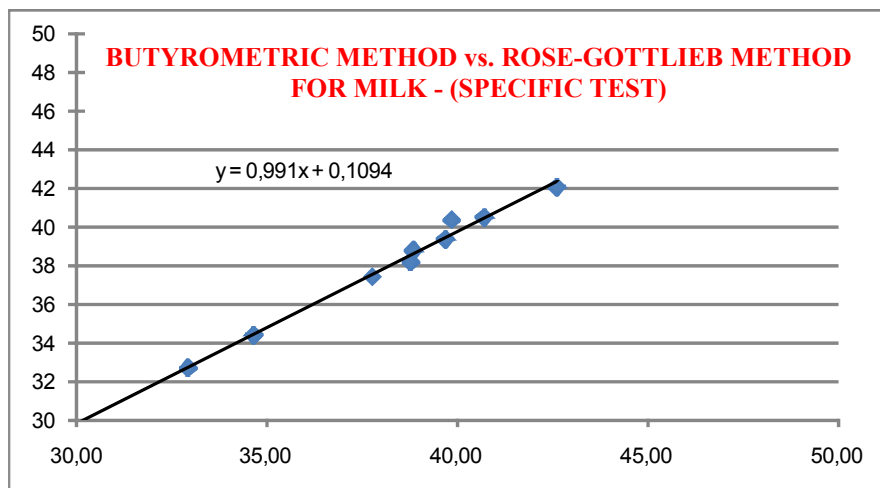
Mean deviation	0.23
Slope	0.991
Intercept	0.11
Residual SD	0.33

Unit: g/Kg of milk

A mean deviation of 0.23 g/Kg between the two methods can be observed and the regression slope is not significantly different from 1.00. The individual deviations observed between the two methods are about 0.5 g/Kg maximum, which is very close to the repeatability limits of the Gerber method (r = 0.5 g/L) and the reproducibility limits of the Röse-Gottlieb method (R= 0.53 g/Kg).

2) USE OF PROFICIENCY TEST DATA FOR THE METHOD WITH MILK AND CREAM

For many years, Actilait has been proposing Cecalait® proficiency tests (PT) for the determination of fat in milk and cream using the acidobutyrometric and Röse-Gottlieb methods on the same set of samples. Four PT are scheduled each year, in March, June, September and December for milk, and in February, May, September and November for cream.

The cows' feed varies significantly during these 4 periods (in the region where the milk and cream used for the samples is produced), thus the fat composition of the basic products (milk and cream) is different. It also seemed interesting to verify if the relationship between the two methods is influenced by the different periods of sample preparation.

The data compared below come from the results of proficiency tests from 2003 to 2010 (4 tests per year). The butyrometric values correspond to the robust mean (according to ISO 13528) of around 100 lab results (mainly French) using the standard NF V 04-210 for milk and the standard NF V 04-263 for cream. The Röse-Gottlieb values correspond to the robust mean (ISO 13528) of around 40 French and foreign lab results using the ISO 1211 method for milk and the ISO 2450 method for cream. A comparison was made between 32 different PT on 10 samples, and an additional study was performed in the range of 3-5 % fat in milk (best agreement between the two methods) to observe the traceability of this alternative method.
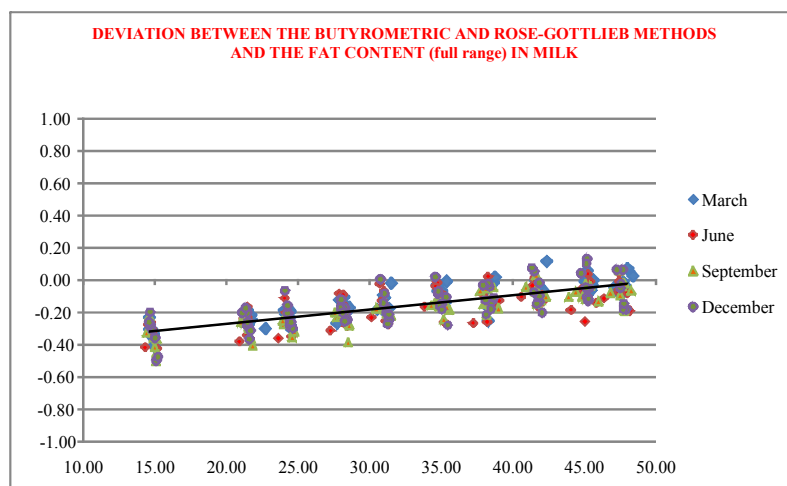
The results and comparison of 4 different periods using the butyrometric and extraction methods for milk and cream are presented below:
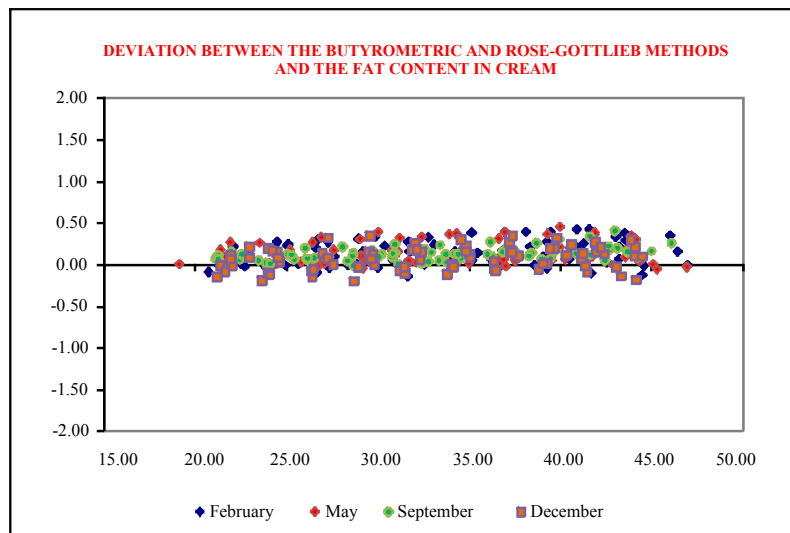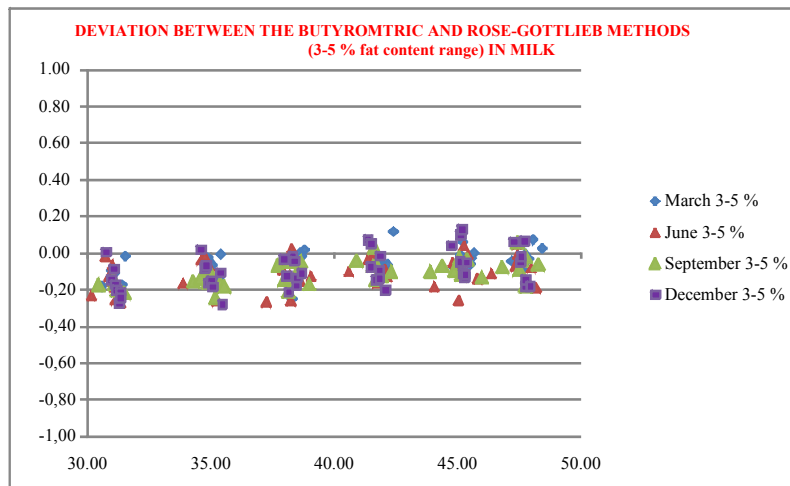
	Deviation Buty / RG	Slope	Bias	Sy,x
March	-0.13	0.991	0.444	0.057
June	-0.17	0.992	0.440	0.08
September	-0.19	0.991	0.493	0.061
December	-0.16	0.991	0.446	0.085
All PT	-0.16	0.991	0.446	0.074

Relationship between the butyrometric Gerber and Röse-Gottlieb methods (in g/100 g) –Milk

	Deviation Buty / RG	Slope	Bias	Sy,x
February	0.12	0.995	0.05	0.15
May	0.12	0.996	-0.01	0.13
September	0.13	0.994	0.07	0.07
November	0.08	0.995	0.09	0.14
All PT	0.11	0.995	0.05	0.13

Relationship between the butyrometric Gerber and Röse-Gottlieb methods (in g/100 g) –Cream

DEVIATION BETWEEN THE BUTYROMTRIC AND ROSE-GOTTLIEB METHODS (3-5 % fat content range) IN MILK

DEVIATION BETWEEN THE BUTYROMETRIC AND ROSE-GOTTLIEB METHODS AND THE FAT CONTENT IN CREAM

For milk a mean deviation of –0.16 g/L can be observed over the entire measurement range (15 to 50 g/Kg) of the Gerber method in comparison with the Röse-Gottlieb method. Concerning the 30 to 50 g/Kg range, which corresponds to the range targeted for optimum coupling when the method was reviewed in 2002, the mean deviation between the two methods is 0.10 g/Kg. These mean deviations and the parameters of the linear regression (slope, intercept and residual standard deviation) are equivalent for all the periods observed.

For cream, a mean deviation of +0.11 g/100 g can be observed over the entire measurement range of the method between the butyrometric and the Röse-Gottlieb methods. This mean deviation and the parameters of the linear regression (slope, intercept and residual standard deviation) are equivalent for all the periods observed.

GENERAL CONCLUSION

On the basis of the specific test on milk and the proficiency test data, it can be noted that the relationship between the two methods is very stable over the entire year for milk and cream. No significant difference was measured between the 4 periods tested. We can thus conclude that the fat composition does not have an influence on the butyrometric method for milk and cream. Moreover, a good fit between the butyrometric method and the extraction method was observed for both matrixes: milk and cream.

These results were presented to the butyrometric methods work group during the IDF analytical week in Lyon. The work group confirmed these conclusions and validated the official dispatch of these new drafts, firstly to the national IDF committees for consultation and to be added to the work programme in the event of a positive vote. If the draft is accepted at this level, the same procedure will be carried out by ISO for dispatch to member countries.

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