

RESULTS OF THE VALIDATION TESTS FOR THE NF V 04-287 A/B METHOD

The results observed between the acido-butyrometric method (NF V 04-287) and the reference method by extraction (ISO 1735) differ for the determination of fat content. So, a "correction" equation was calculated on the basis of proficiency tests organised by ACTALIA Cecalait from 2003 to 2013.

To validate this equation, a validation test was conducted on cheeses, which were not represented in the samples set used for the initial calculation of the correction. These tests were carried out at the end of 2015 and at the beginning of 2016 (processed cheese). A comparative analysis of 30 samples (8 fresh cheese samples, 8 soft cheese samples, 8 hard cheese samples and 6 processed cheese samples) was performed by many laboratories using the butyrometric (NF V 04-287 A or B) and the extraction (ISO 1735) methods.

The results obtained by the butyrometric method were then transformed using the initially calculated equation ($Y = 0.974 X + 0.345$) and compared to the results obtained by the extraction method

These test were performed within the context of the CNIEL analytical commission and AFNOR V04 "milk and dairy products" commission.

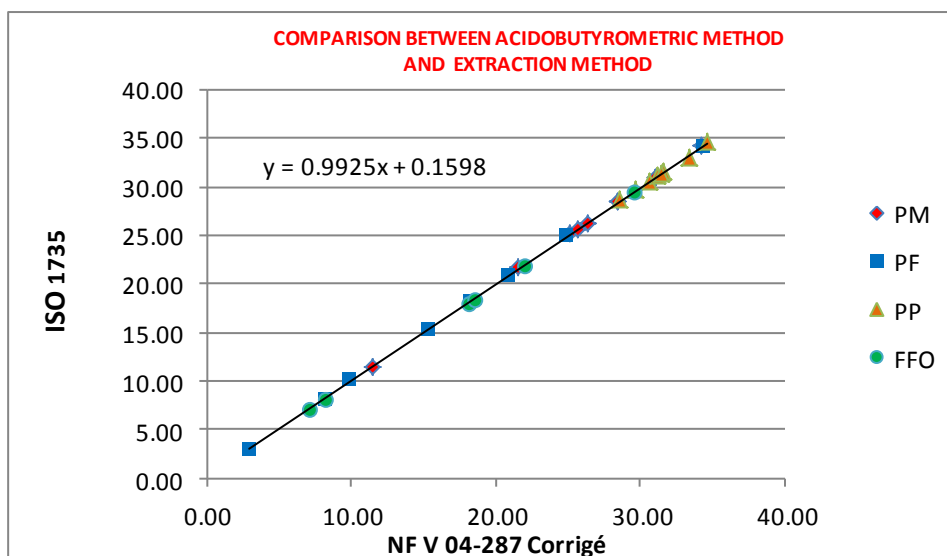
Number of participating laboratories

The table below presents the number of the participating laboratories in the tests according to the method used:

| Type of cheese | Number of laboratories ISO 1735 | Number of laboratories NF V 04-287 |
|----------------------|------------------------------------|---------------------------------------|
| Soft cheese PM | 6 | 8 |
| Fresh cheese PF | 5 | 5 |
| Herd cheese PP | 4 | 9 |
| Processed cheese FFO | 4 | 8 |

Final results

The figure below presents the relation between the acido-butyrometric method after correction and the extraction method on 30 cheese samples:



The table below presents the results of the comparison between the both methods on 30 cheese samples (before and after correction):

| | ISO 1735 | NF V 04 287 | NF V 04 287 Corr | Ecart |
|----------------|--------------|--------------|------------------|-------------|
| PM 1 | 34,28 | 34,87 | 34,31 | 0,03 |
| PM 2 | 30,97 | 31,49 | 31,01 | 0,05 |
| PM 3 | 25,15 | 25,40 | 25,09 | -0,06 |
| PM 4 | 25,70 | 26,10 | 25,77 | 0,07 |
| PM 5 | 26,23 | 26,72 | 26,37 | 0,14 |
| PM 6 | 28,52 | 28,92 | 28,51 | -0,01 |
| PM 7 | 11,47 | 11,39 | 11,44 | -0,03 |
| PM 8 | 21,64 | 21,81 | 21,59 | -0,05 |
| PF1 | 8,18 | 8,04 | 8,17 | -0,01 |
| PF2 | 3,01 | 2,64 | 2,92 | -0,10 |
| PF3 | 34,26 | 34,92 | 34,36 | 0,10 |
| PF4 | 24,91 | 25,15 | 24,85 | -0,07 |
| PF5 | 20,93 | 21,12 | 20,92 | -0,01 |
| PF6 | 18,31 | 18,37 | 18,24 | -0,07 |
| PF7 | 10,16 | 9,78 | 9,87 | -0,30 |
| PF8 | 15,45 | 15,34 | 15,29 | -0,16 |
| PP1 | 28,75 | 29,05 | 28,64 | -0,10 |
| PP2 | 30,46 | 31,10 | 30,63 | 0,18 |
| PP3 | 33,07 | 33,94 | 33,40 | 0,33 |
| PP4 | 29,67 | 30,15 | 29,72 | 0,04 |
| PP5 | 34,55 | 35,19 | 34,62 | 0,07 |
| PP6 | 31,06 | 31,65 | 31,17 | 0,11 |
| PP7 | 31,58 | 32,18 | 31,69 | 0,11 |
| PP8 | 31,34 | 31,98 | 31,49 | 0,15 |
| FFO1 | 29,58 | 30,05 | 29,62 | 0,04 |
| FFO2 | 18,07 | 18,17 | 18,04 | -0,02 |
| FFO3 | 7,15 | 6,95 | 7,12 | -0,03 |
| FFO4 | 21,98 | 22,20 | 21,97 | -0,01 |
| FFO5 | 8,21 | 8,07 | 8,21 | 0,00 |
| FFO6 | 18,46 | 18,61 | 18,47 | 0,01 |
| Moyenne | 23,10 | 23,38 | 23,12 | 0,01 |
| Différence | | 0,27 | 0,01 | |
| Sy,x | | 0,091 | 0,091 | |
| Min | 3,01 | | | -0,30 |
| Max | 34,55 | | | 0,33 |

Conclusion:

Firstly, these tests confirmed the deviations previously observed between the both methods (mean bias of about 0.27 g/100 g for all cheeses and all contents). Apply the correction equation (calculated on the basis of the proficiency tests results) to the results obtained by the acido-butyrometric method allows to obtain fat contents equivalent to those obtained by the extraction method. Indeed, the study of the simple linear regression between the both sets of data (corrected acido-butyrometric method and extraction method) allows to obtain a mean deviation between the both methods equal to +0.01 g/100 g and a regression slope close to 1.00 (0.9925).

We can then conclude that the equation calculated to adjust the acido-butyrometric method is efficacy for the extraction method in fresh, soft, hard and processed cheeses.