



Harmonisation of Milk Analyser for Fatty Acid determination by FTMIR

An essential step prior to collective data use

O. Leray, M. Ferrand, H. Larroque, M. Brochard

This program is financially supported by ANR, Apis-Gene, FGE, FranceAgriMer, French Ministry of Agriculture, EU

www.phenofinlait.fr

phenofinlait@inst-elevage.asso.fr

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Summary of Phénofinlait

- Phenotyping dairy animals for fine milk composition (**Fatty Acids FA & Individual Protein IP**)
- **Fatty acid profiles** of individual milk (cow, sheep, goat) by **mid-infrared spectrophotometry** (FTMIR)
- Establishment of **calibration equation sets** (cow, sheep, goat)
- Application to about **800 000 milk spectra** of **20 000 females** of **3 species** collected on **14 months** from **13 FTMIR analysers** in **9 laboratories** and stored in a **spectral data base**.
- Parallel **survey of feeding, parentage, DNA mapping, etc**, to establish suitable relations **for further data analysis**.
- Establishment of **diagnosis tools and levers** for farmers to monitor and **orient fine composition of milk**.



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Unique national calibration => Evaluation of the reproducibility of FTMIR method

❖ Interlaboratory study (October 2008):

- 8 laboratories
- 12 Milkoscan FT6000 (Foss, DK)
- 15 individual bovine milk samples
- 13 calibration samples (mid-range MIR RM set, Actilait)
- Prior spectrum standardisation
- Analysis in triplicates in spectrum extraction mode
- FA predictions from spectrum data using Phénofinlait equations



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Outcomes

- Heterogeneous distribution of biases between analysers
- High relative biases and bias ranges resulting in high reproducibility values R depending on FA equation
- Biases with natural individual samples and biases with calibration samples are tightly correlated hence calibration is possible.

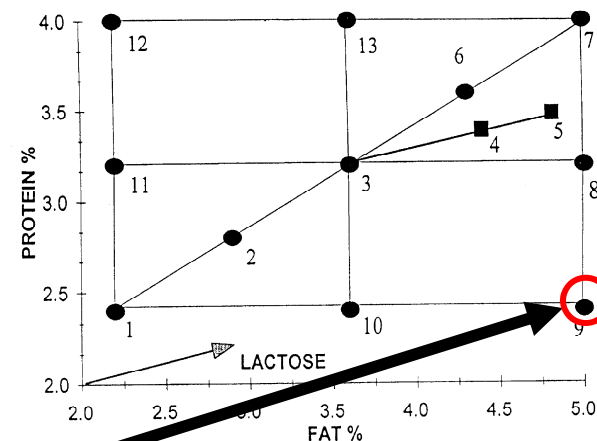
Improvement of the method => **Evaluation of correction modes**

Predictions :

- Linear regression thr. 13 calibration samples

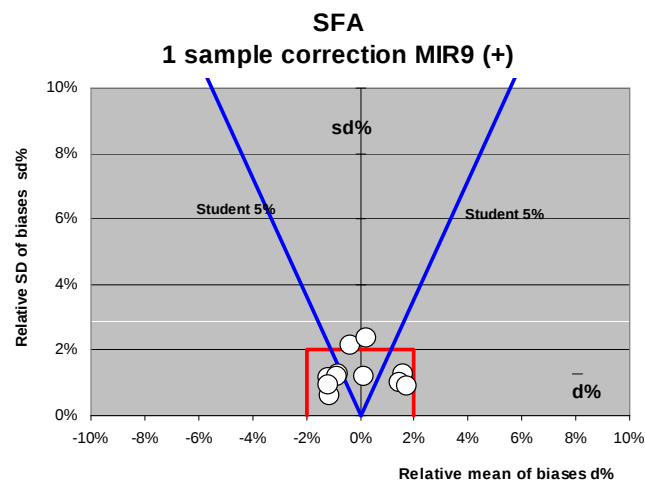
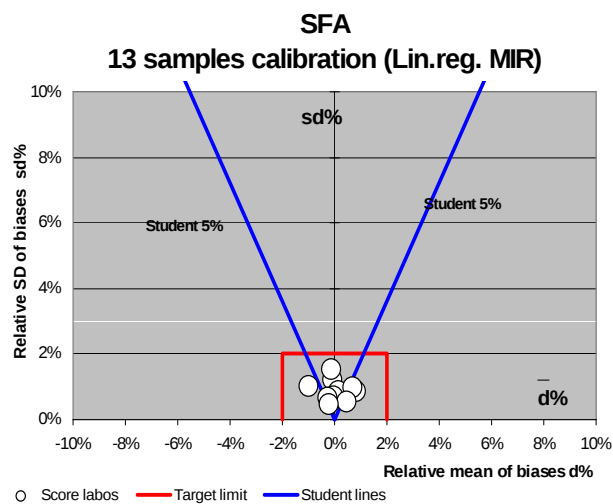
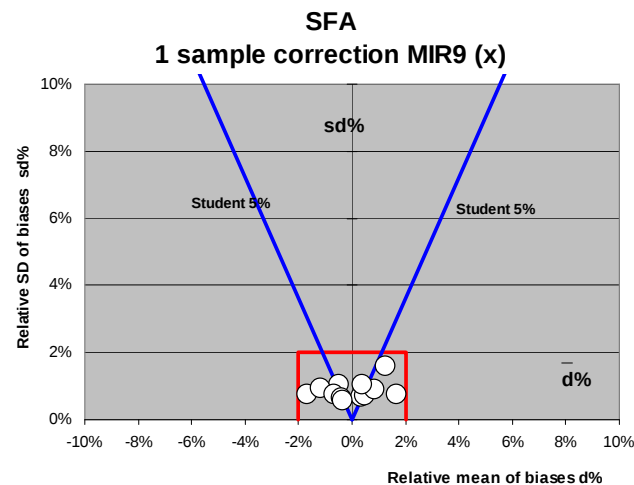
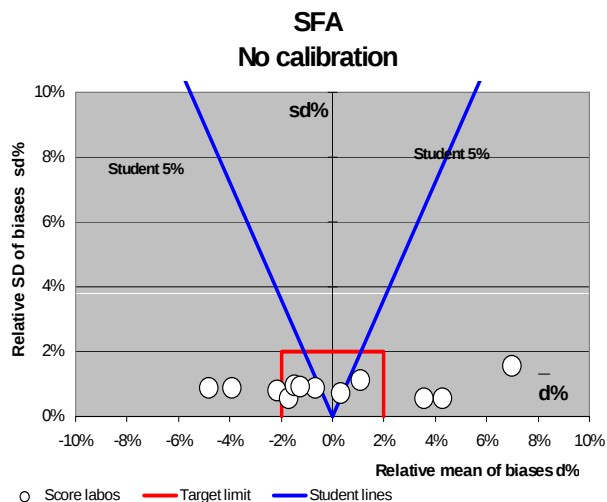
Predictions and absorbances :

- Multiplicative correction thr. 1 sample (x)
- Additive correction thr. 1 sample (+)

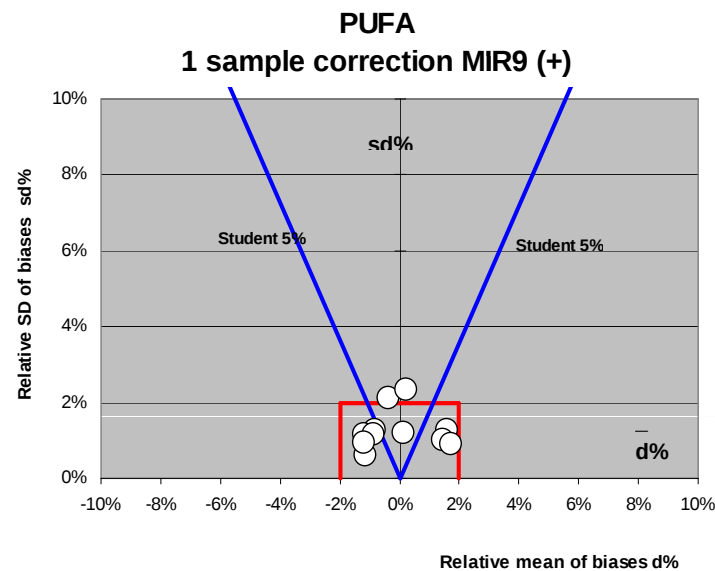
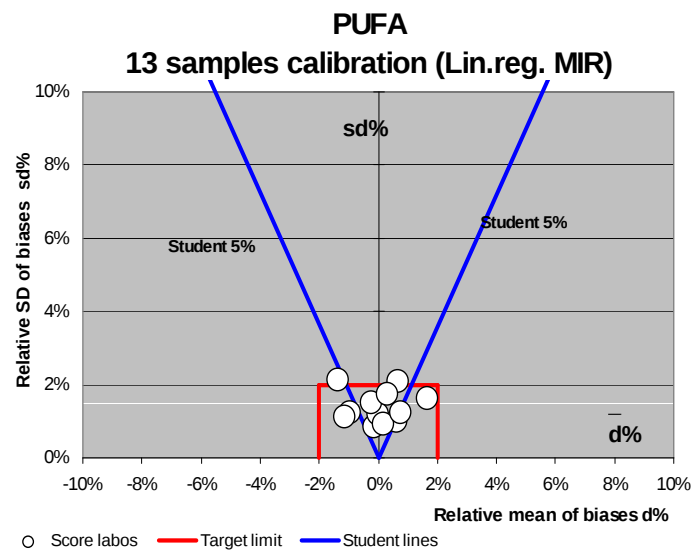
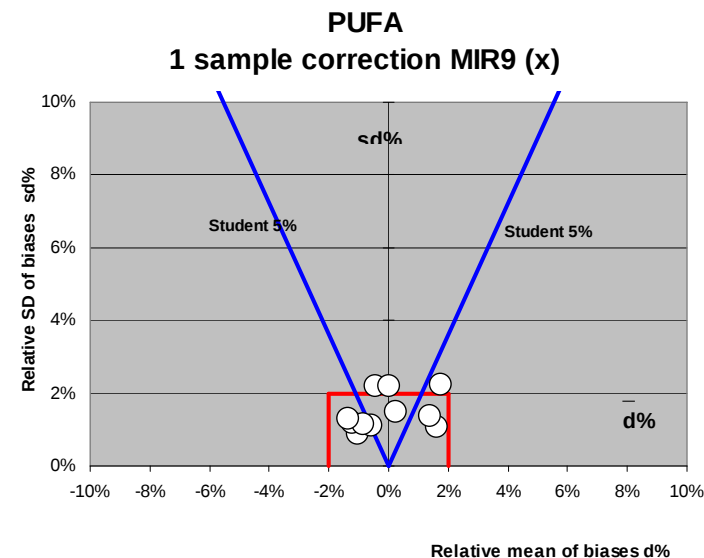
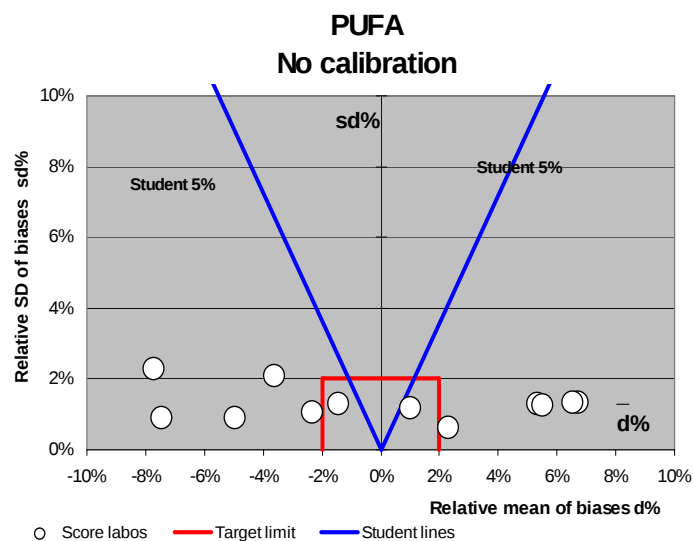


Cecalait MIR RM, O Leray 1998

Correction of predictions – Saturated Fatty Acids



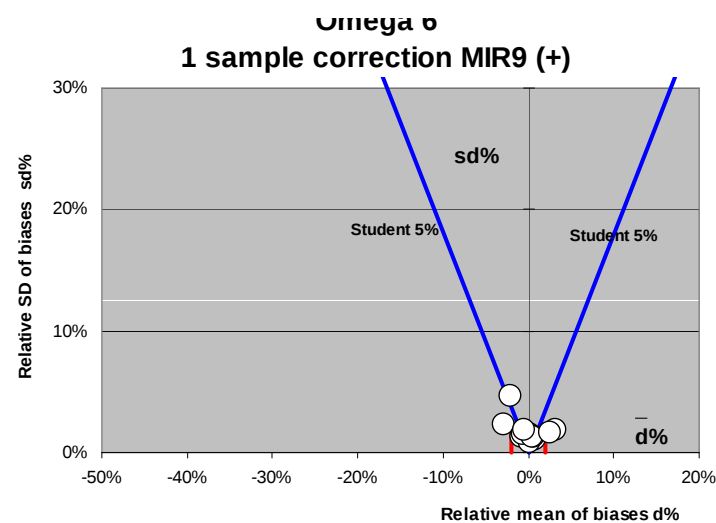
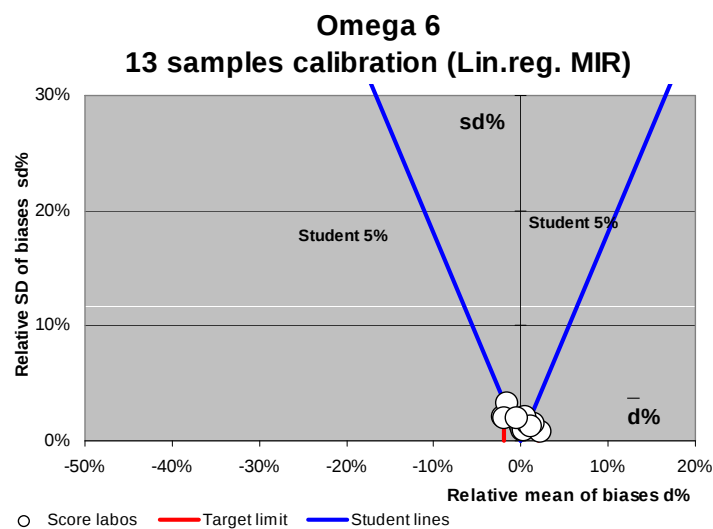
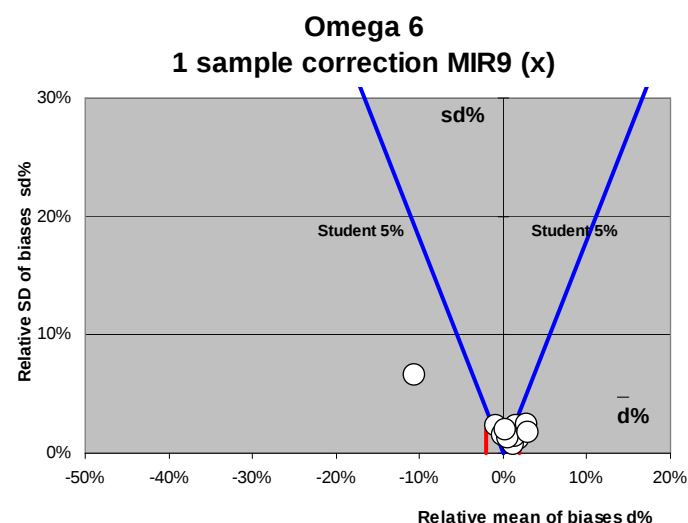
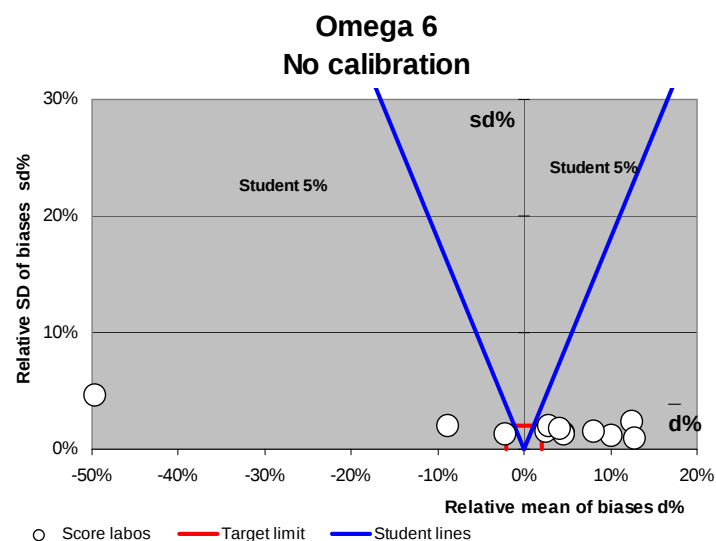
Correction of predictions – Polyunsaturated Fatty Acids



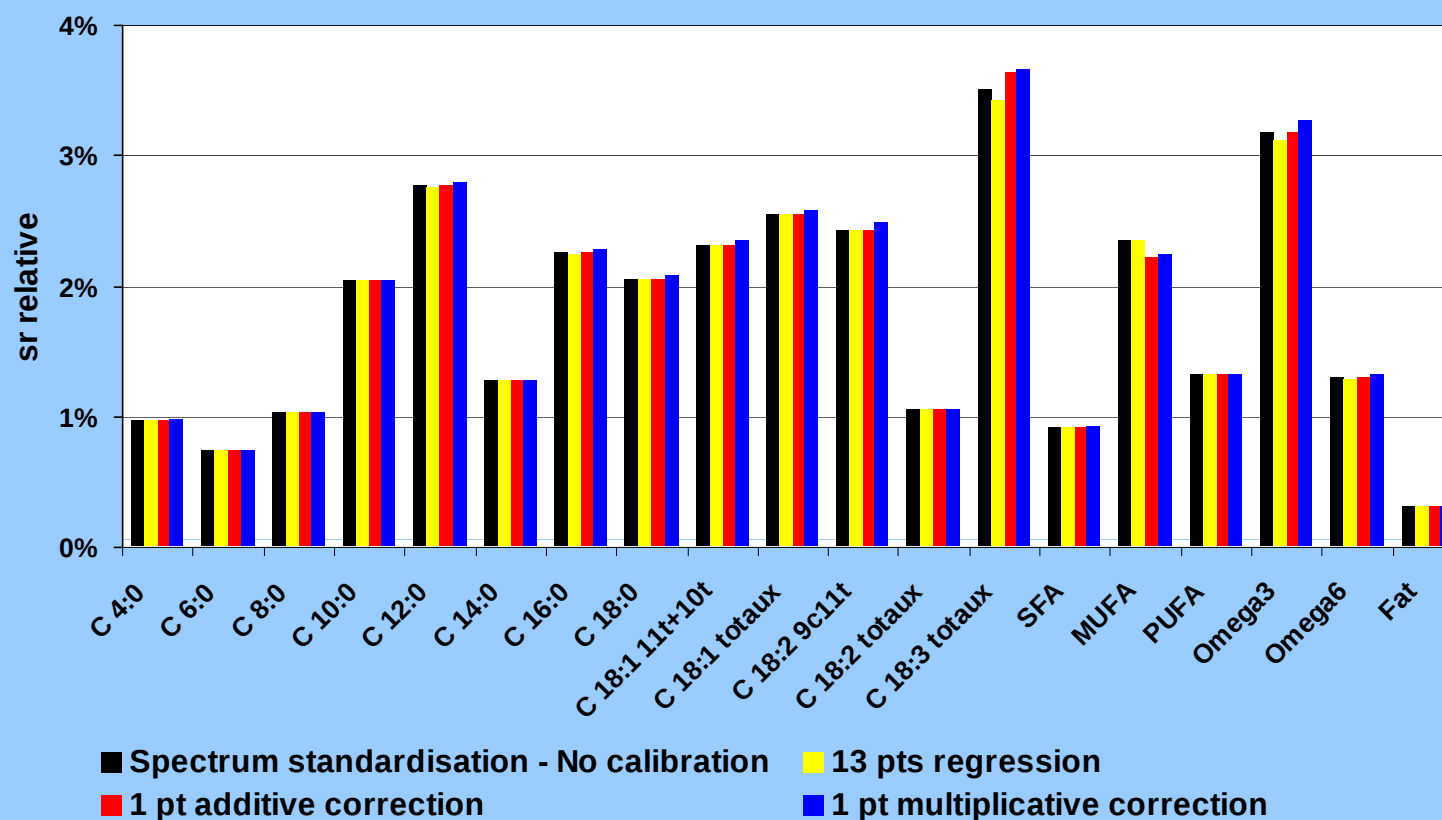
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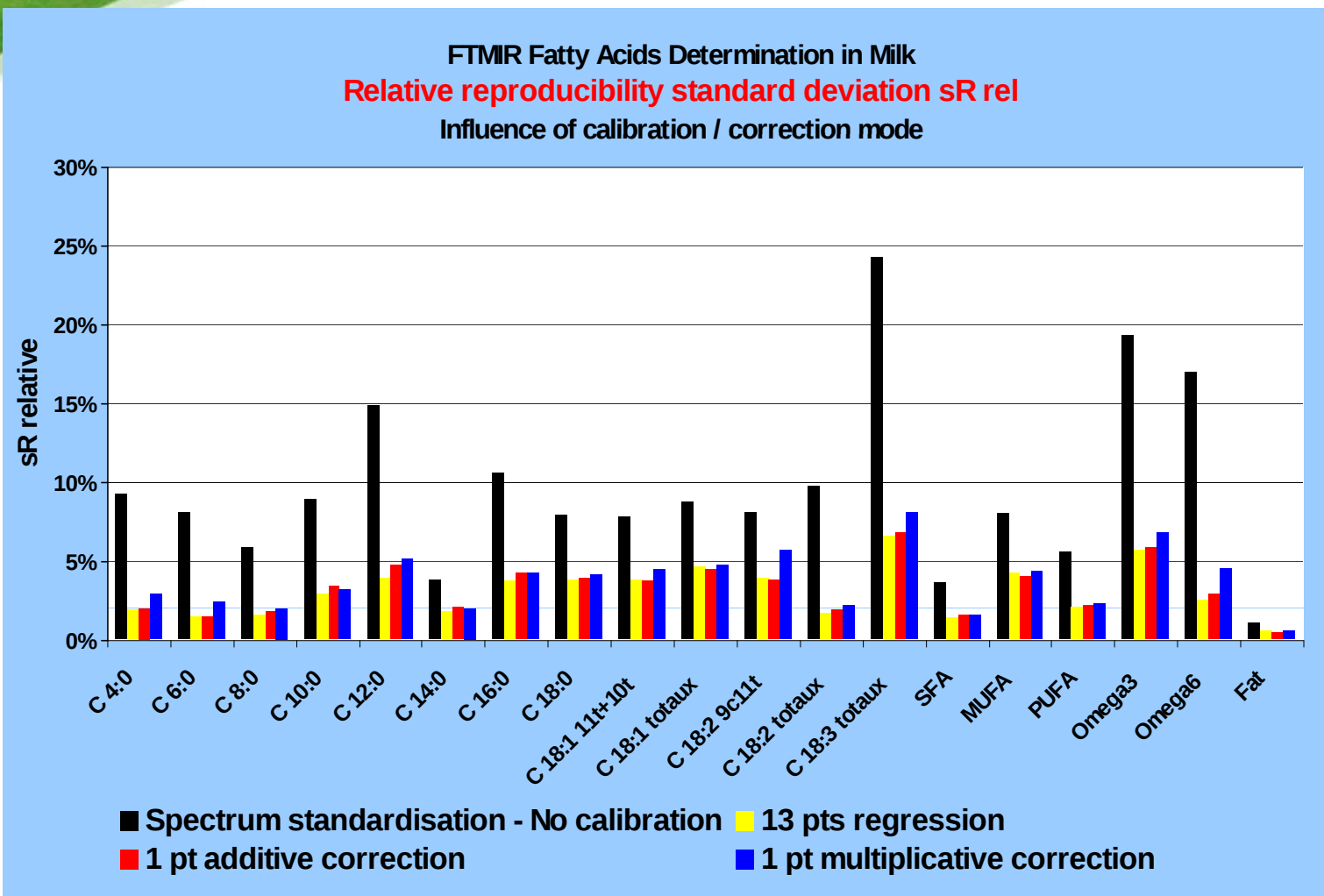
Correction of predictions – Omega 6

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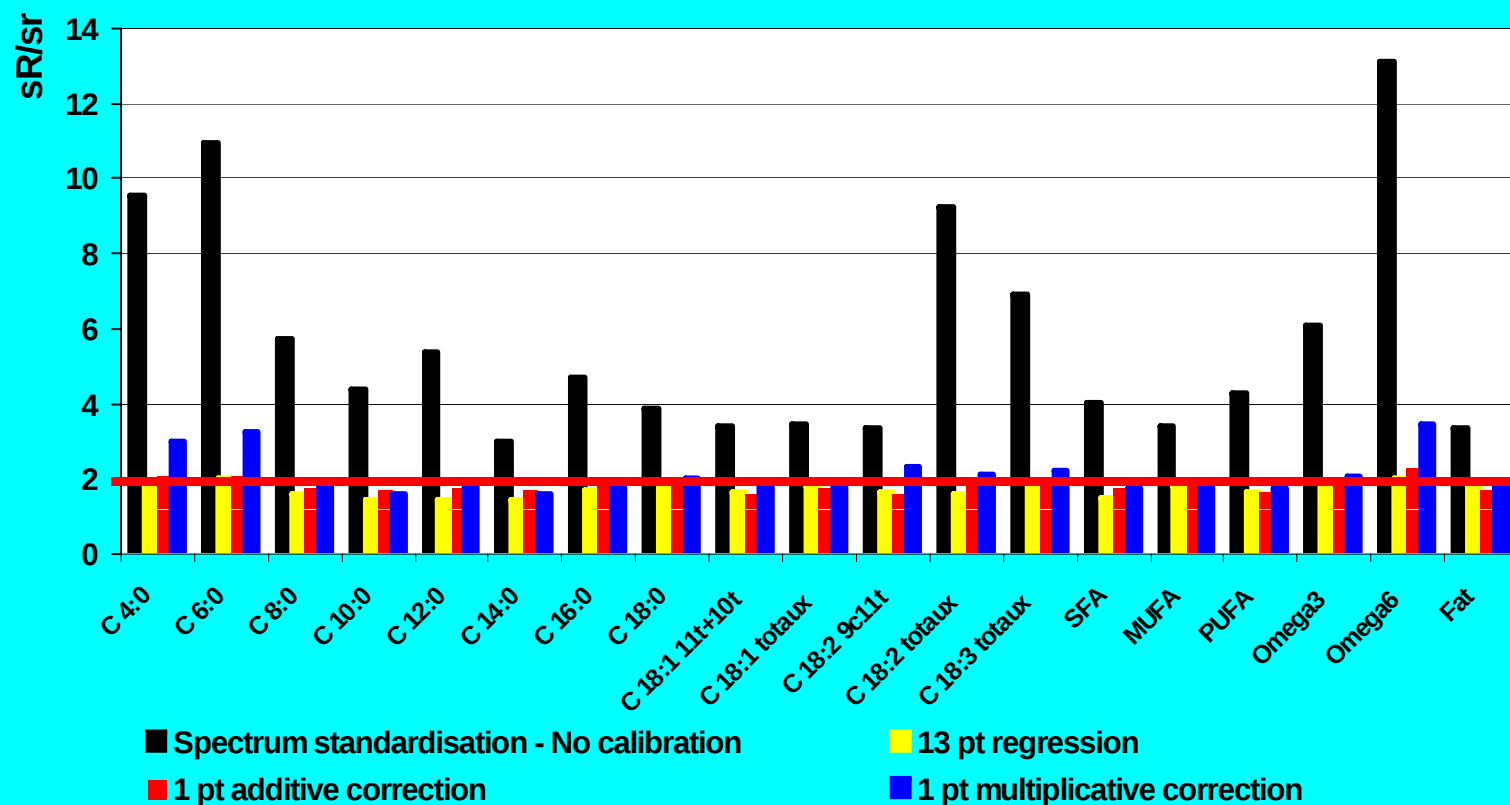
FTMIR Fatty Acids Determination in Milk
Relative repetability standard deviation sr rel
Influence of calibration / correction mode





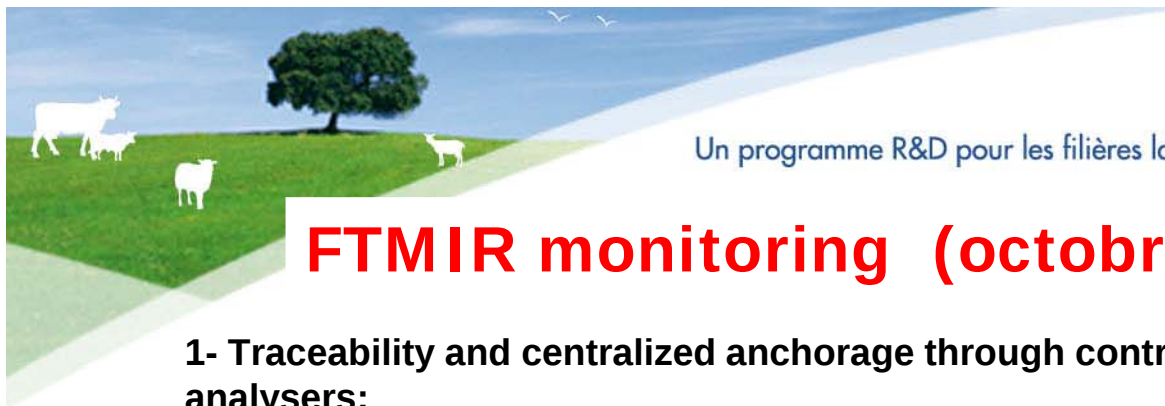


FTMIR Fatty Acids Determination in Milk
- Precision ratio R/r depending on the calibration / correction mode -



Repeatability and reproducibility of FTMIR analysers

Fatty acids	Mean level g/kg	Spectrum standardisation				Centralised calibration			
		r	r rel	R	R rel	r	r rel	R	R rel
C 4:0	1,84	0,05	2,7%	0,48	26,1%	0,05	2,7%	0,10	5,3%
C 6:0	1,05	0,02	2,1%	0,24	22,7%	0,02	2,1%	0,04	4,2%
C 8:0	0,59	0,02	2,9%	0,10	16,7%	0,02	2,9%	0,03	4,6%
C 10:0	1,23	0,07	5,7%	0,31	25,1%	0,07	5,7%	0,10	8,3%
C 12:0	1,36	0,11	7,8%	0,57	41,8%	0,11	7,7%	0,15	11,0%
C 14:0	4,57	0,16	3,6%	0,50	10,8%	0,16	3,6%	0,24	5,2%
C 16:0	13,47	0,85	6,3%	4,01	29,8%	0,85	6,3%	1,43	10,6%
C 18:0	4,37	0,25	5,8%	0,98	22,3%	0,25	5,8%	0,47	10,8%
C 18:1 11t+10t	0,74	0,05	6,5%	0,16	22,0%	0,05	6,5%	0,08	10,8%
C 18:1 total	11,20	0,80	7,1%	2,76	24,6%	0,80	7,2%	1,47	13,1%
C 18:2 9c11t	0,28	0,02	6,8%	0,06	22,8%	0,02	6,8%	0,03	11,2%
C 18:2 total	0,89	0,03	3,0%	0,24	27,5%	0,03	3,0%	0,04	4,8%
C 18:3 total	0,29	0,03	9,8%	0,19	68,0%	0,03	9,6%	0,05	18,6%
SFA	30,55	0,79	2,6%	3,17	10,4%	0,79	2,6%	1,19	3,9%
MUFA	12,74	0,84	6,6%	2,87	22,5%	0,84	6,6%	1,53	12,0%
PUFA	1,29	0,05	3,7%	0,21	15,8%	0,05	3,7%	0,08	6,1%
Omega3	0,36	0,03	8,9%	0,20	54,2%	0,03	8,8%	0,06	16,1%
Omega6	0,85	0,03	3,6%	0,41	47,8%	0,03	3,6%	0,06	7,3%
Fat	45,03	0,41	0,9%	1,38	3,1%	0,41	0,9%	0,73	1,6%



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FTMIR monitoring (octobre 2009 -> Déc.2010)

1- Traceability and centralized anchorage through control milk sample common for the analysers:

-> LC = Control milk -80°C

⇒ to determine correction

-> LT = Liquid milk with bronopol at 4°C

⇒ to check and secure correction

2- Afterward standardization of predictions by **additive correction** from the national database:

Mois	12/09		01/10		02/10		03/10		04/10		05/10		06/10		07/10		08/10		09/10		10/10		11/10		12/10	
Appareil	1q	2q	1q	2q	1q	2q	1q	2q	1q	2q	1q	2q	1q	2q	1q	2q	1q	2q	1q	2q	1q	2q	1q	2q	1q	2q
1	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT
2	LC	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT
3	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT	LT
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Master
analyser

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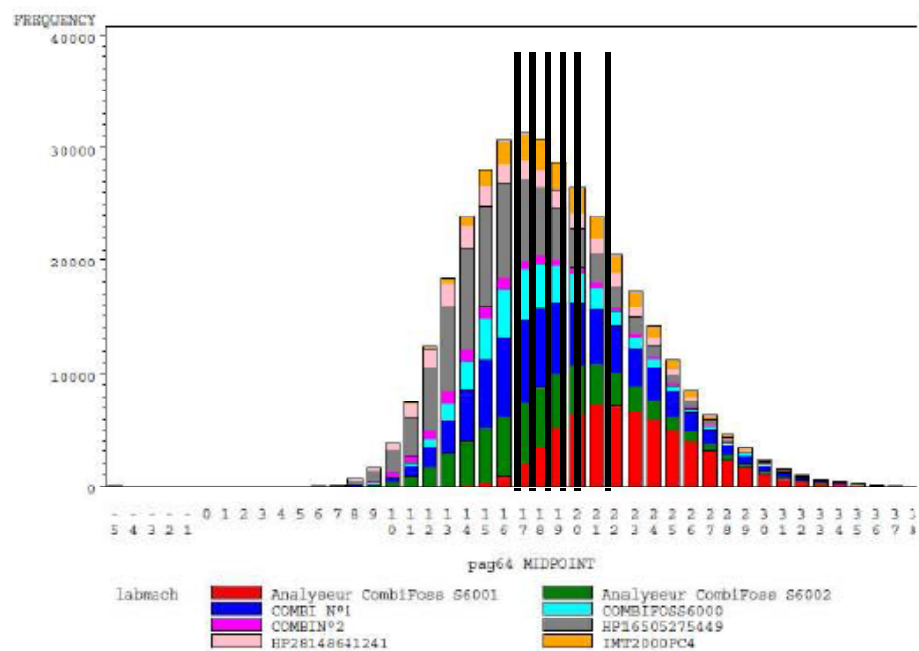
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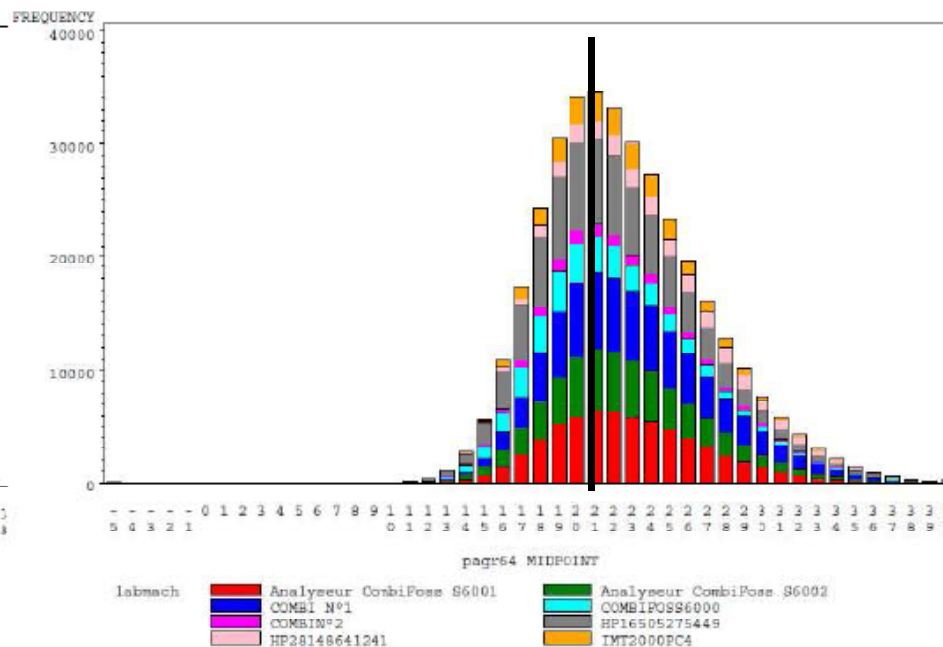
*Effect of data correction on a 14 month-long
population of results through a control milk network
8 FT MIR analysers*

Bovine milk – C18:1 %

Raw data



Corrected data

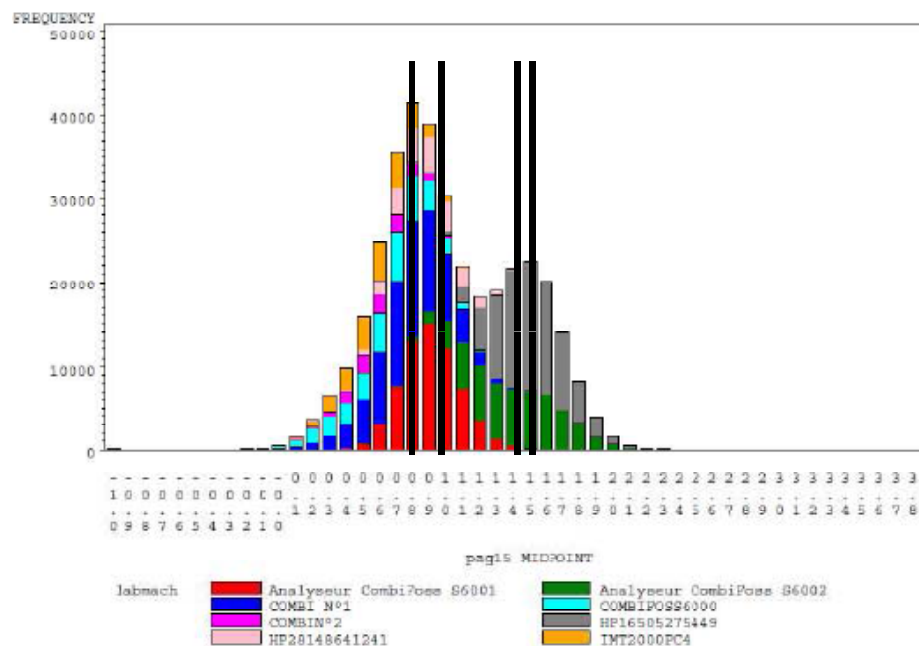




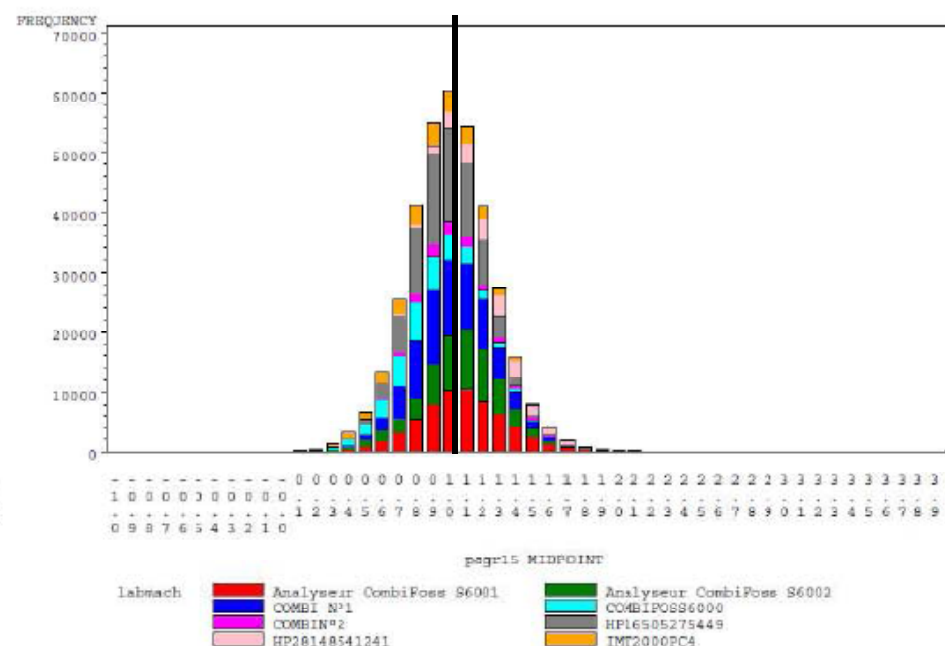
*Effect of data correction on a 14 month-long population of results through a control milk network
8 FTMIR analysers*

Bovine milk – Omega3 %

Raw data



Corrected data







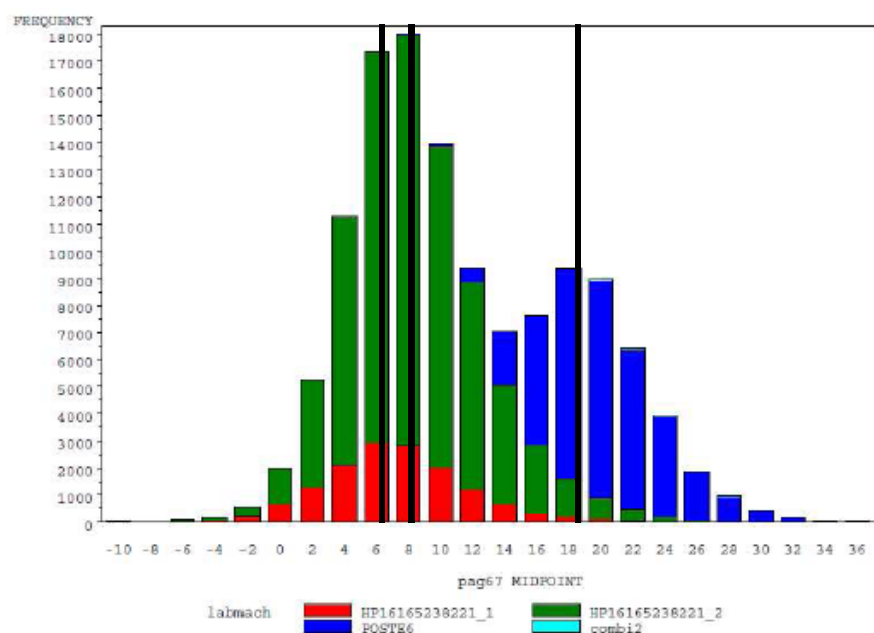
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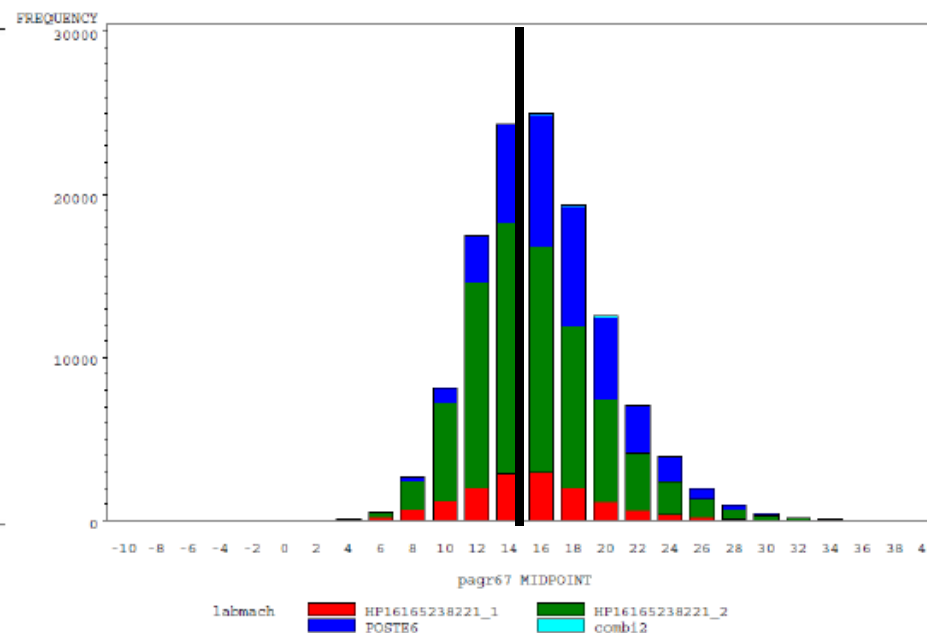
*Effect of data correction on a 14 month-long population of results through a control milk network
4 FT MIR analysers*

Ovine milk – C18:1 %

Raw data



Corrected data



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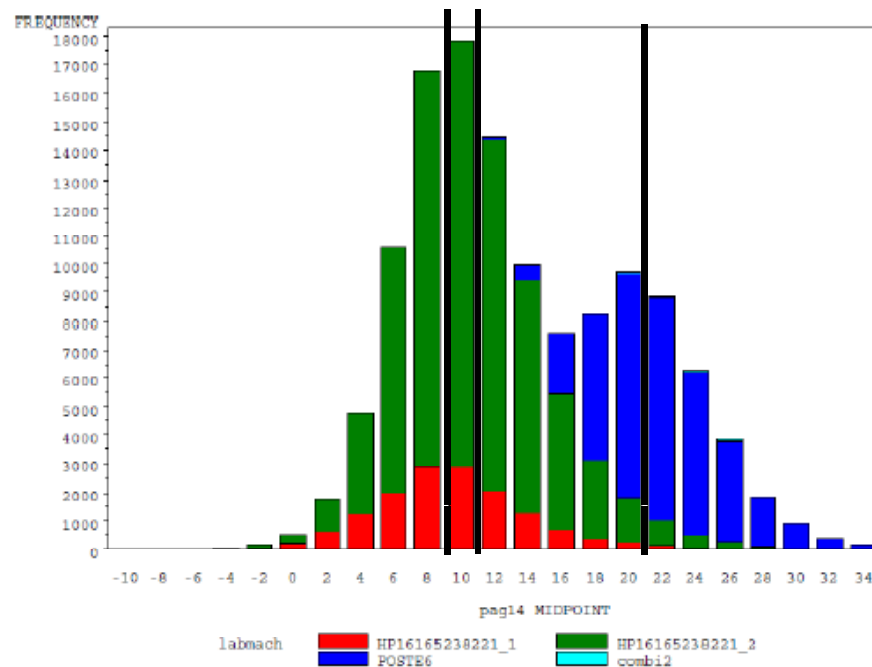
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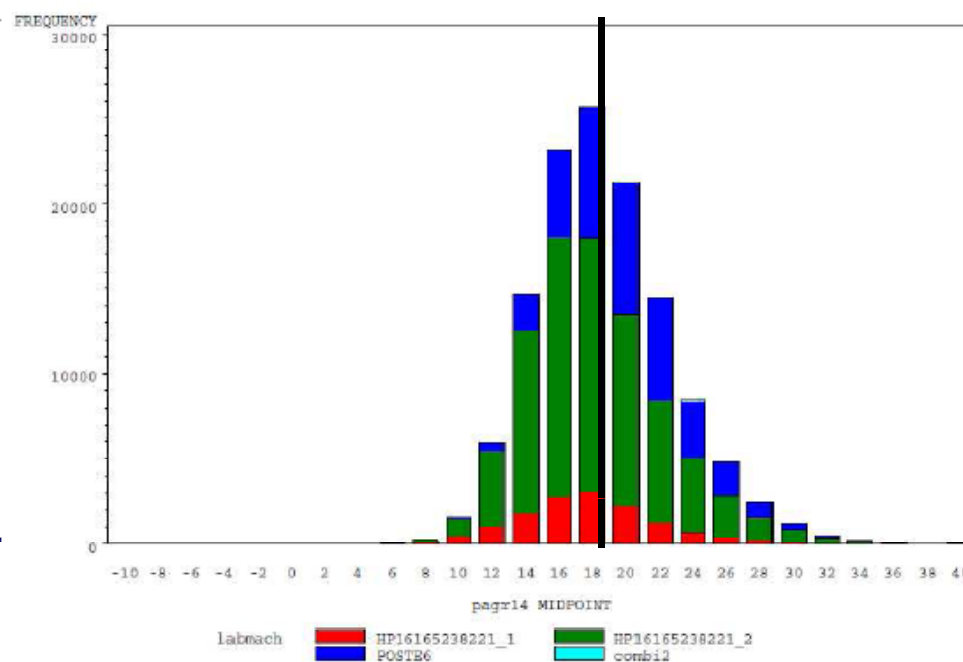
*Effect of data correction on a 14 month-long
population of results through a control milk network
4 FTMIR analysers*

Ovine milk – MUFA %

Raw data



Corrected data





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Conclusion

- Spectrum standardization as applied did not suffice to harmonise results between instruments
- Efficient result standardization is achieved through centralised calibration thus providing good analytical characteristics r , R and R/r
- Standardisation can be achieved either at the level of predicted FA content values or at the level of absorbances
- Building a multi lab/instrument spectrum data base for multi purpose implies either collecting well standardised spectral data or include the means for afterward correction to minimise possible strong background noise and develop more efficient diagnosis tools.

Acknowledgment is given

to the companies Foss and Bentley which provided standard spectrum export facilities to laboratories,

to the partners of the programme, laboratories and DHI organisations, which provided data,

to the members of the scientific committee of Phénofinlait who advised for and managed that work.

Thank you for your attention

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