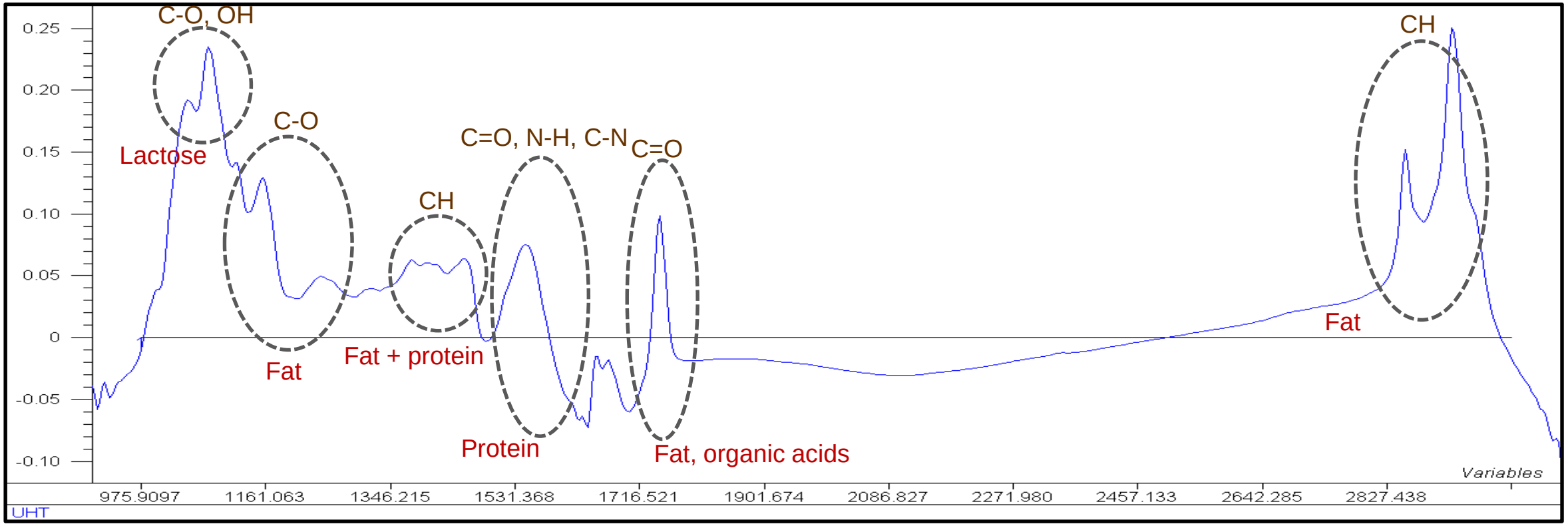
ICAR CONFERENCE & IDF/ISO ANALYTICAL WEEK 2019 PRAGUE CZECH REPUBLIC

New quality assurance challenges with recent mid-infrared models

Frédéric Dehareng & Clément Grelet

Prague, Thursday 20.06.2019
Technical session 7: Challenges in Creating Additional Value from Milk Analysis



- **Position of the peaks → Qualitative analysis**
- **Intensity of the peaks → Quantitative analysis**

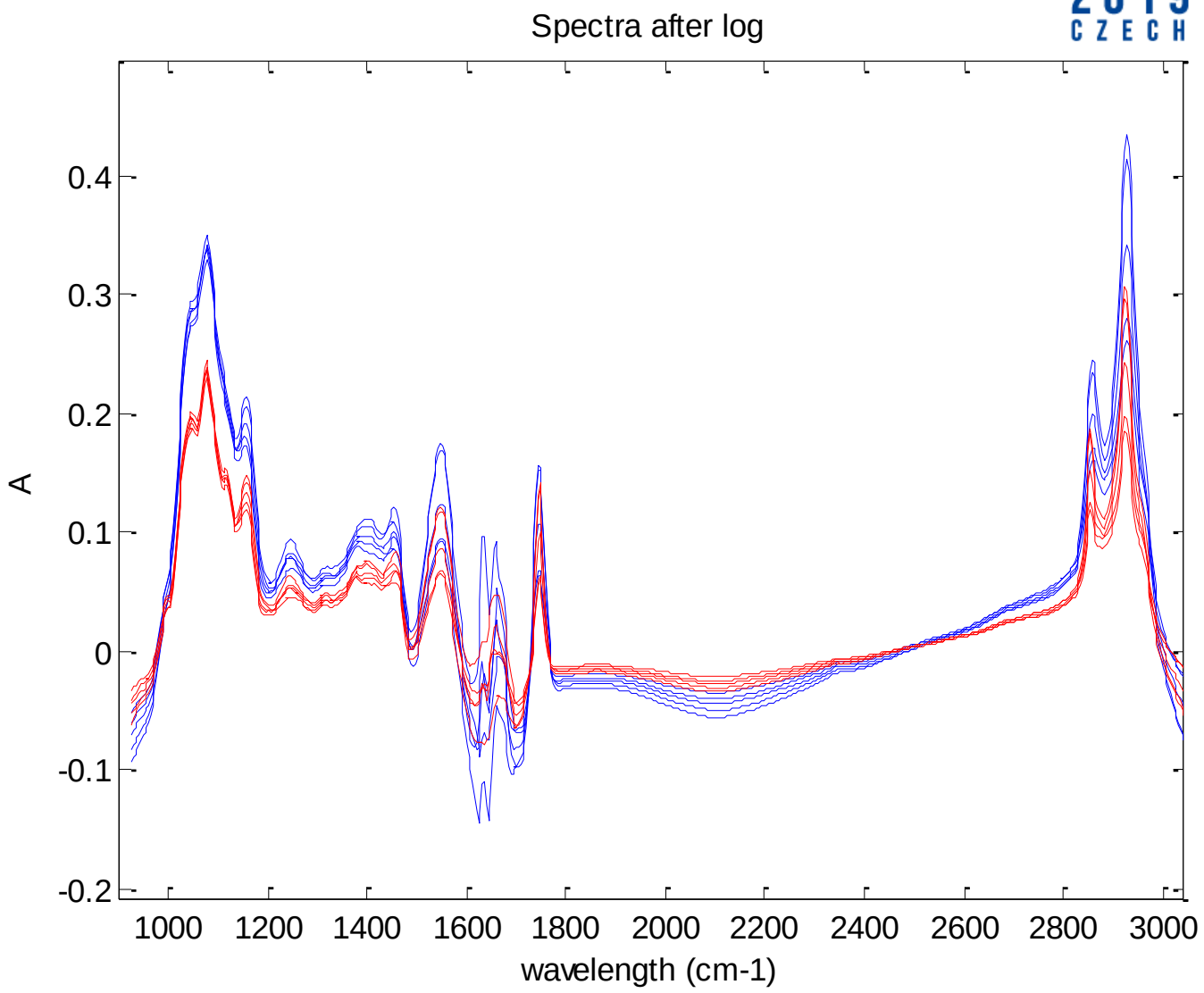
But…

Spectra of a same milk could differ:

- between different brands apparatus

Instruments are different

→ Common milks analyzed on 2 instruments **from 2 different brands**



But…

Spectra of a same milk could differ:

- between different brands apparatus
- between different models apparatus of the same brand
- between apparatus of the same model of the same brand

Moreover, even with the same instrument, the spectra could be different for the same milk. It's not stable in time !

-T°/humidity in the lab
-Piece replacement
-Maintenance operation
-Use/wear

Classical slope/bias correction

Milk component ⟷ **Milk MIR spectra**

Fat, Protein, Lactose, SNF

Casein Hewavitharana et al. 1997. Analyst 122:701–704.

Urea Hansen, P. W. 1998. Milchwissenschaft 53:251–255

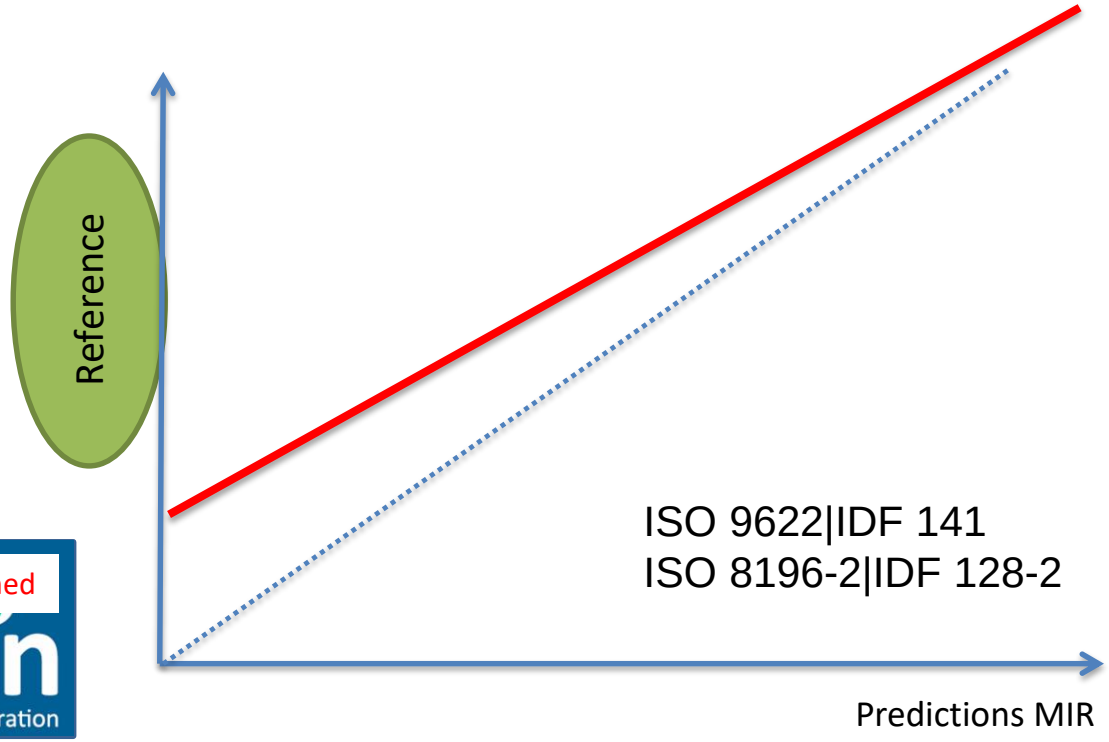
Fatty acids Soyeurt et al. 2006 J. Dairy Sci. 89: 3690–3695

New applications of MIR spectrometry: QA practices with new parameters in raw milk analysis

Will be published

Bulletin of the International Dairy Federation

Slope and bias correction based on standard samples with known reference values



Classical slope/bias correction

Milk component

Fat, Protein, Lactose, SNF

Casein Hewavitharana et al. 1997.

Urea Hansen, P. W. 1998.

Fatty acids Soyeurt et al. 2006

Milk MIR spectra

Need for another solution

Phenotype

Energy Balance Dale et al., 2019

Methane Vanlierde et al., 2015

Nitrogen used efficiency Grelet et al. 2019

Blood component

Blood BHB, NEFA, IGF-I, Glucose Grelet et al.,2016

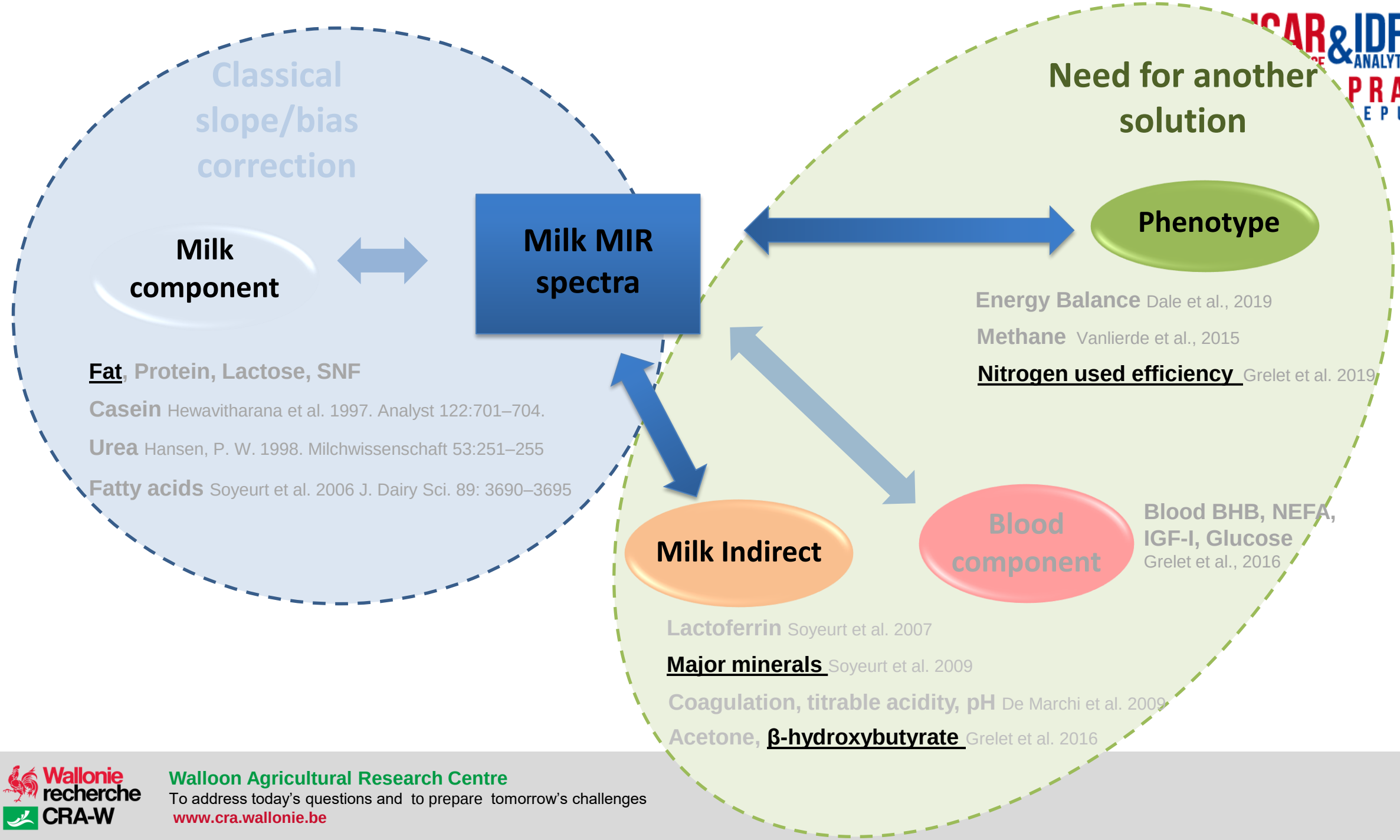
Milk Indirect

Lactoferrin Soyeurt et al. 2007

Major minerals Soyeurt et al. 2009

Coagulation, titrable acidity, pH De Marchi et al. 2009

Acetone, β-hydroxybutyrate Grelet et al. 2016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Blood component
Blood BHB, NEFA, IGF-I, Glucose
Grelet et al., 2016

Tested solution → Standardization of the MIR spectra

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J. Dairy Sci. 98:2150–2160
http://dx.doi.org/10.3168/jds.2014-8764

Standardization of milk mid-infrared spectra from a European dairy network

C. Grelet,[1] J. A. Fernández Pierna,[1] P. Dardenne, V. Baeten, and F. Dehareng[2]
Walloon Agricultural Research Center, Valorisation of Agricultural Products Department, 24 Chaussée de Namur, 5030 Gembloux, Belgium

J. Dairy Sci. 100:7910–7921
https://doi.org/10.3168/jds.2017-12720

Standardization of milk mid-infrared spectrometers for the transfer and use of multiple models

C. Grelet,* J. A. Fernández Pierna,* P. Dardenne,* H. Soyeurt,† A. Vanlierde,* F. Colinet,† C. Bastin,‡ N. Gengler,† V. Baeten,* and F. Dehareng*[1]
*Valorization of Agricultural Products Department, Walloon Agricultural Research Center, 5030 Gembloux, Belgium
†Agriculture, Bio-Engineering, and Chemistry Department, University of Liège, Gembloux Agro-Bio Tech, 5030 Gembloux, Belgium
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QUALITY ASSURANCE TOOLS FOR MID INFRARED SPECTROMETRY IN DAIRY LABORATORIES – PART 1

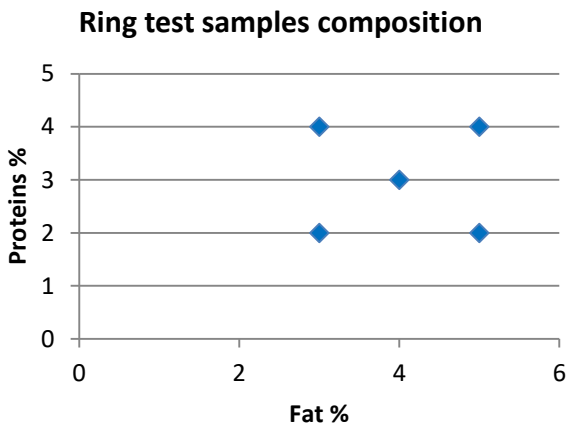
Bulletin 490 2017 of the International Dairy Federation

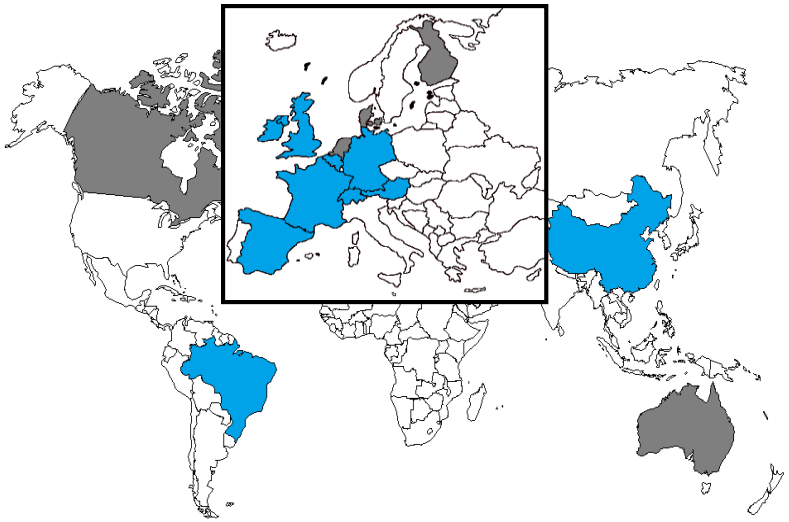
Standardization of FT-MIR instruments for milk analysis

EMR

www.milkrecording.eu

In practice





41 Labs

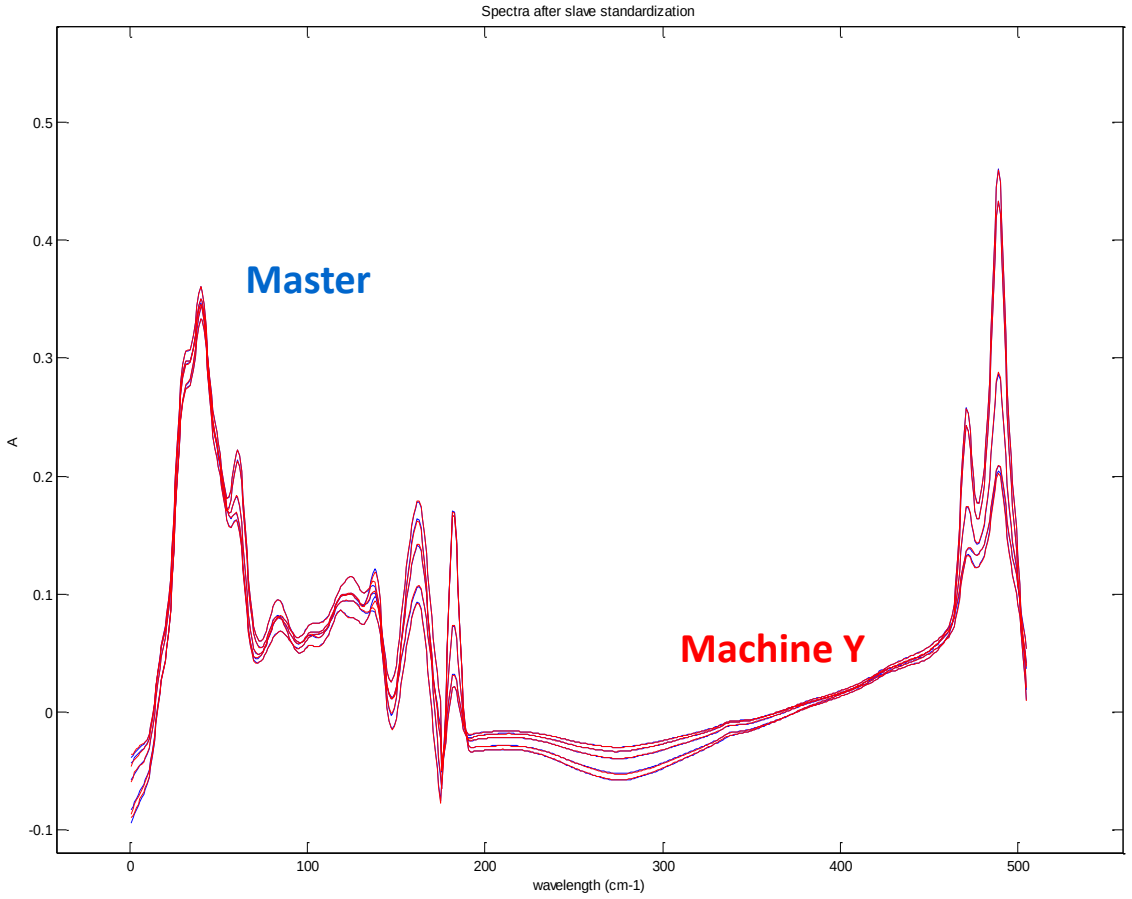
103 Apparatus

- Standardized instruments for creation of equations
- Standardized instruments for use of equations

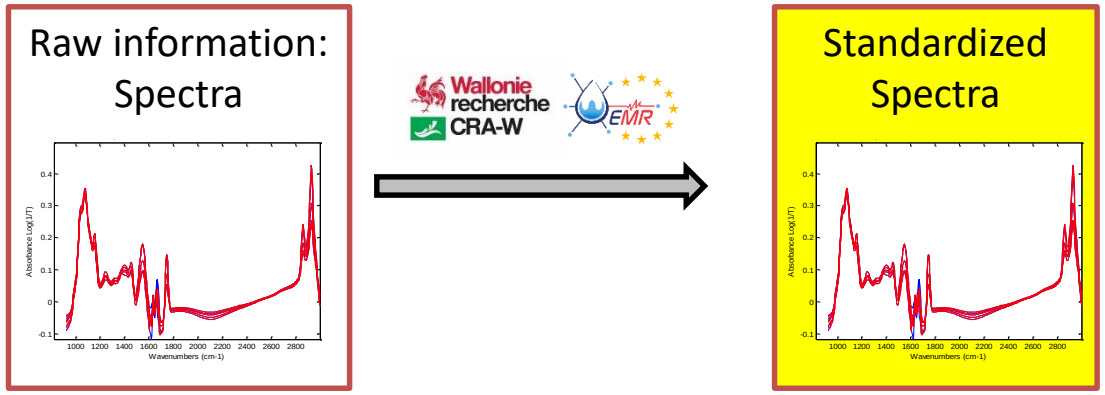
Instruments are different

→ 5 common milks samples analyzed on 2 different instruments

PDS Standardization



To test the standardization

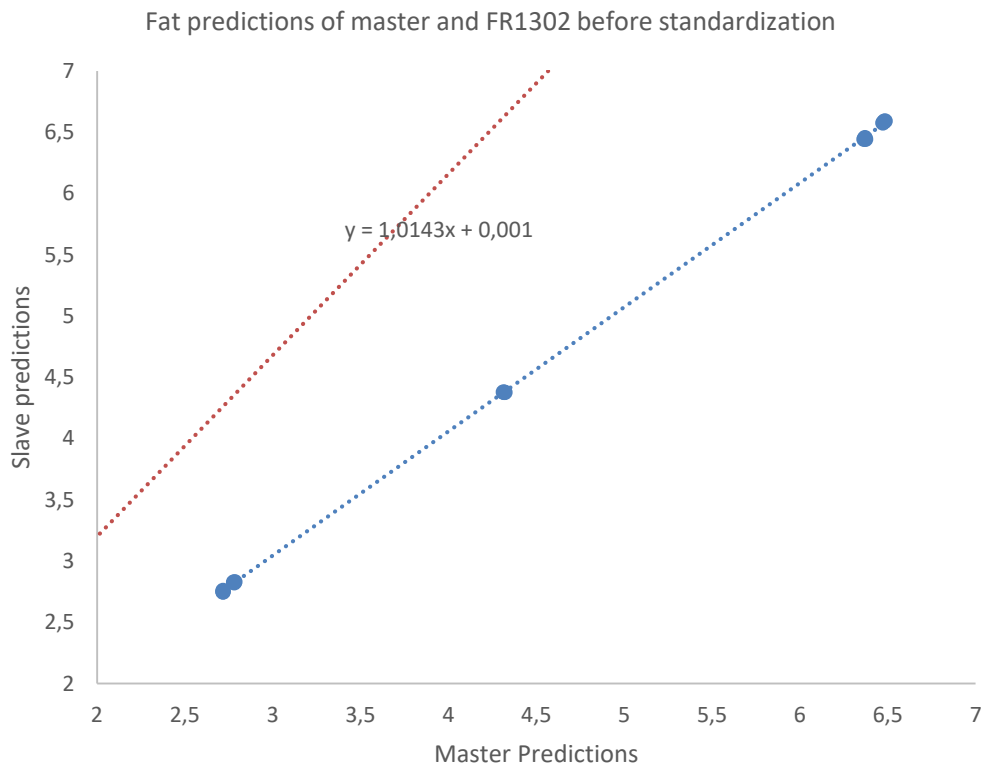


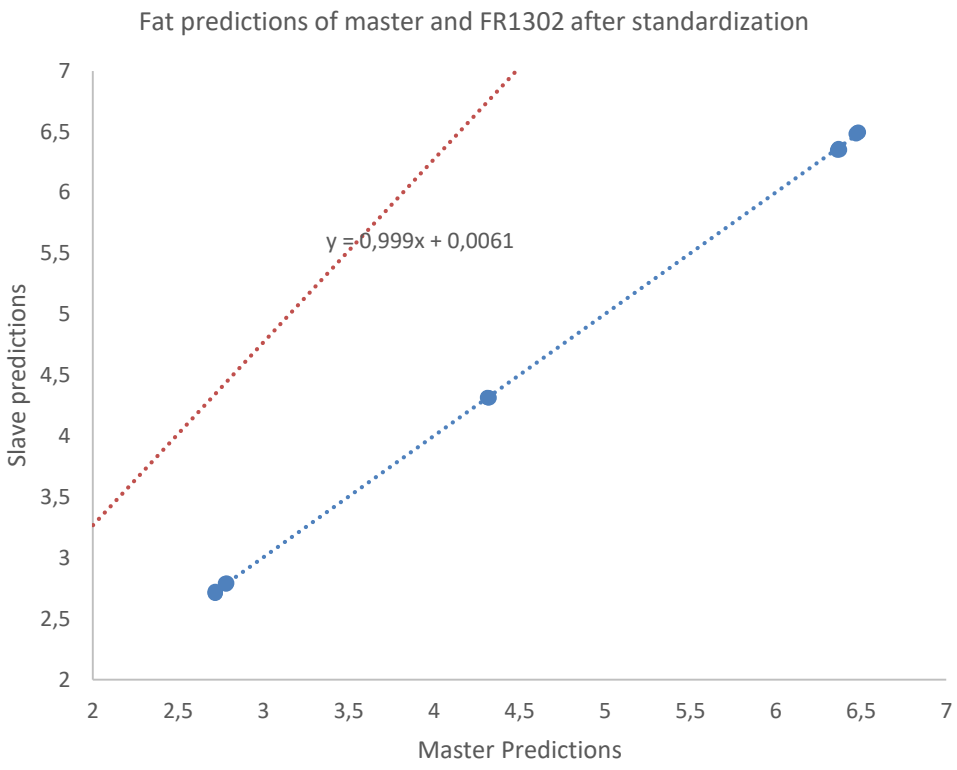
- 6 FT+ apparatus in France
- 7 FT6000 apparatus in Switzerland

Prediction models used:

- Total Fat
- BHB milk
- Milk Phosphorous
- Nitrogen use efficiency

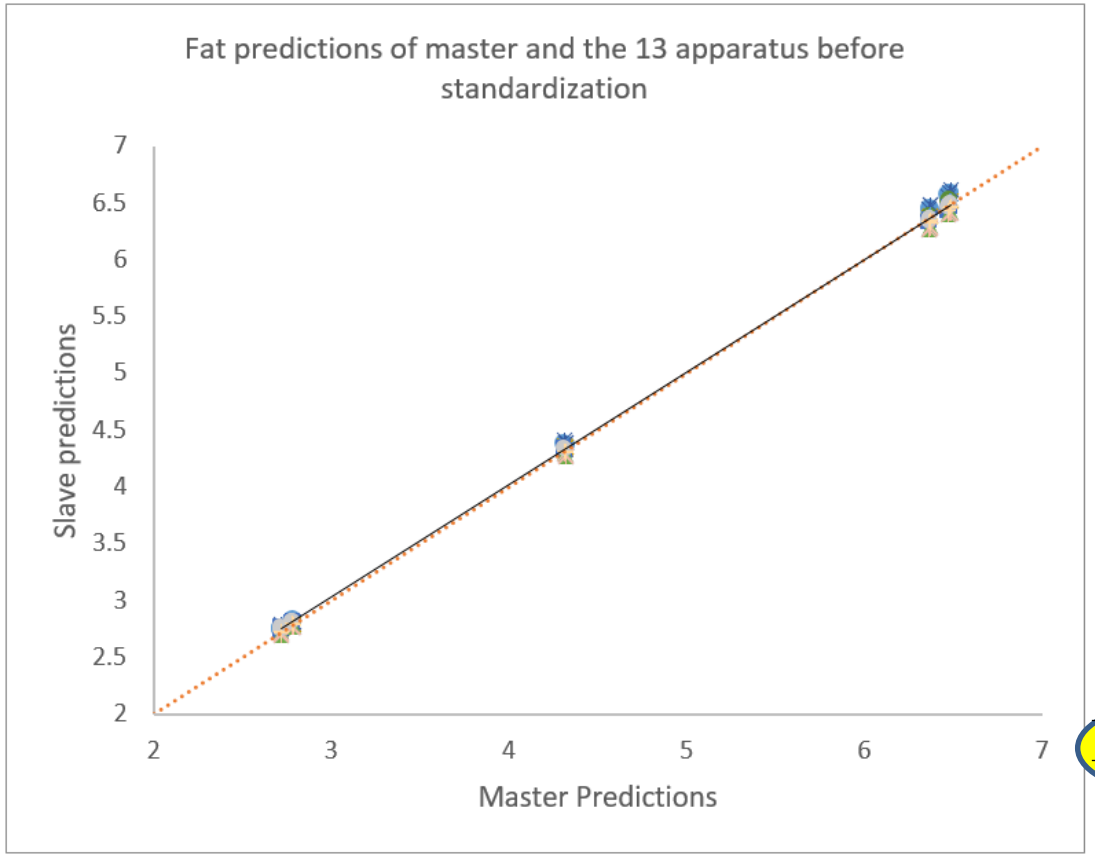
Results: Total Fat predictions





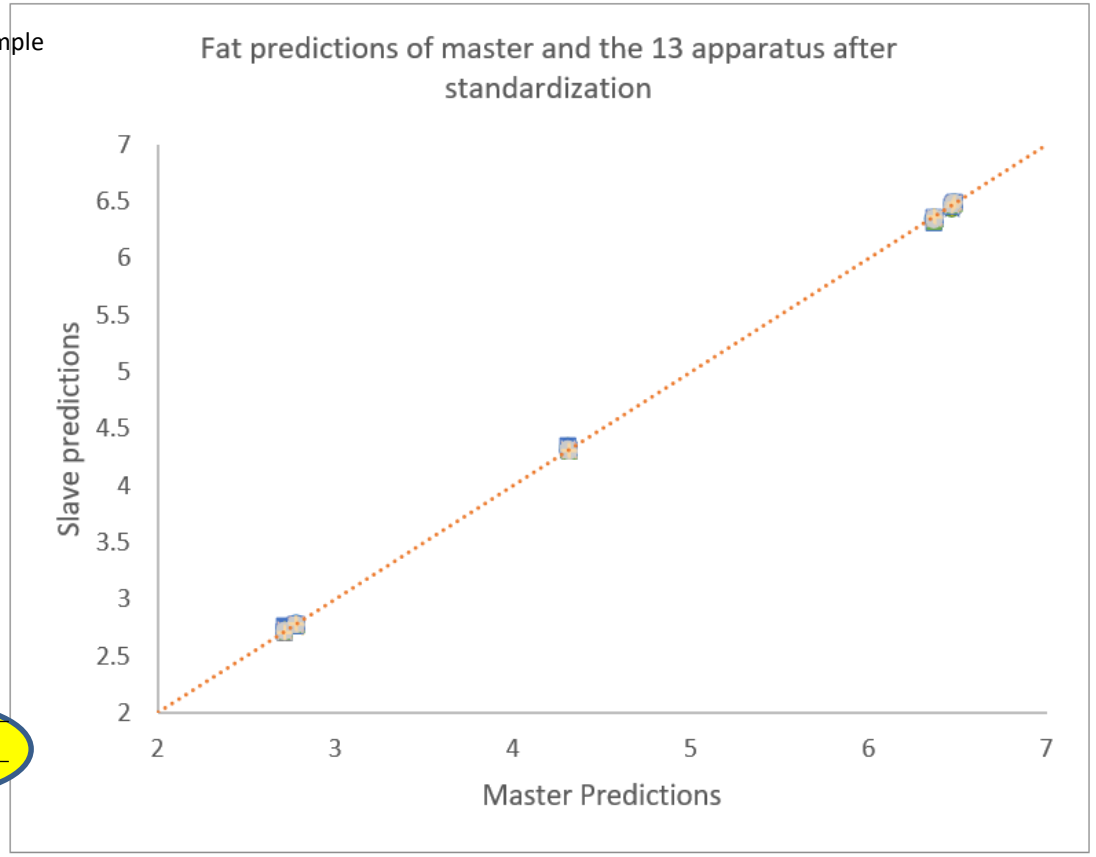
RMSE : 0.071 → RMSE : 0.010

Results: Total Fat predictions



R for each sample

Before STD
0.016
0.016
0.017
0.052
0.052
0.051
0.025
0.018
0.019
0.054
0.055
0.057
0.035
0.032
0.034
0.036

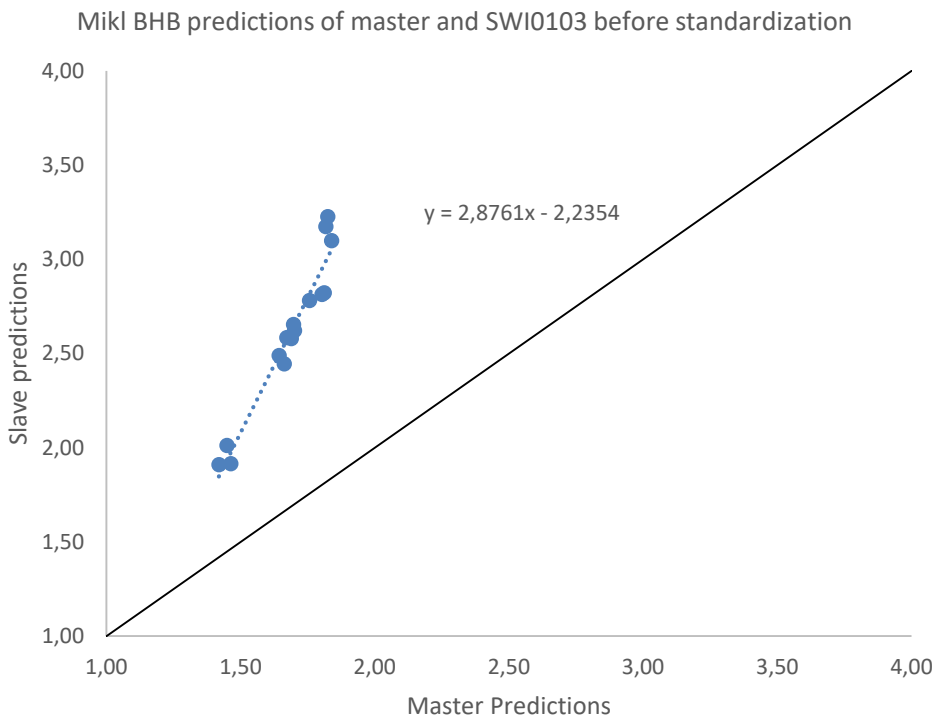


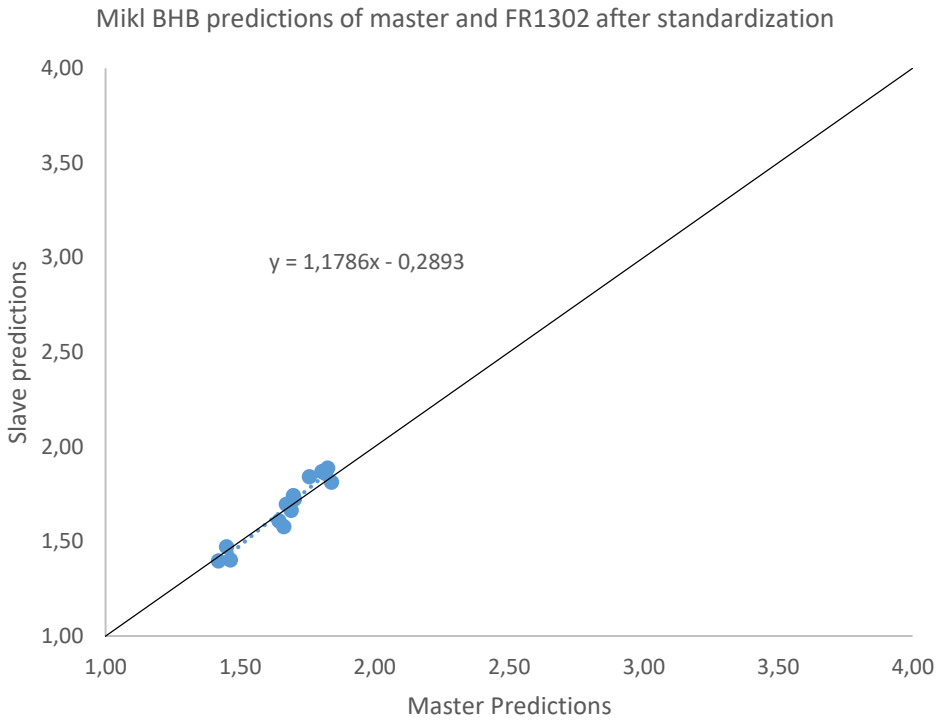
R for each sample

After STD
0.011
0.007
0.007
0.015
0.009
0.006
0.015
0.007
0.009
0.013
0.009
0.008
0.010
0.006
0.005
0.009

		Calibration							Cross validation with 4 subsets		
Total Fat	Unit	# terms	#N	#WL	Mean	SD	SEC	R^2c	SECV	R^2cv	RPDcv
	g/100ml	7	1792	212	3.912	0.971	0.007	1.00	0.007	1.00	132.99

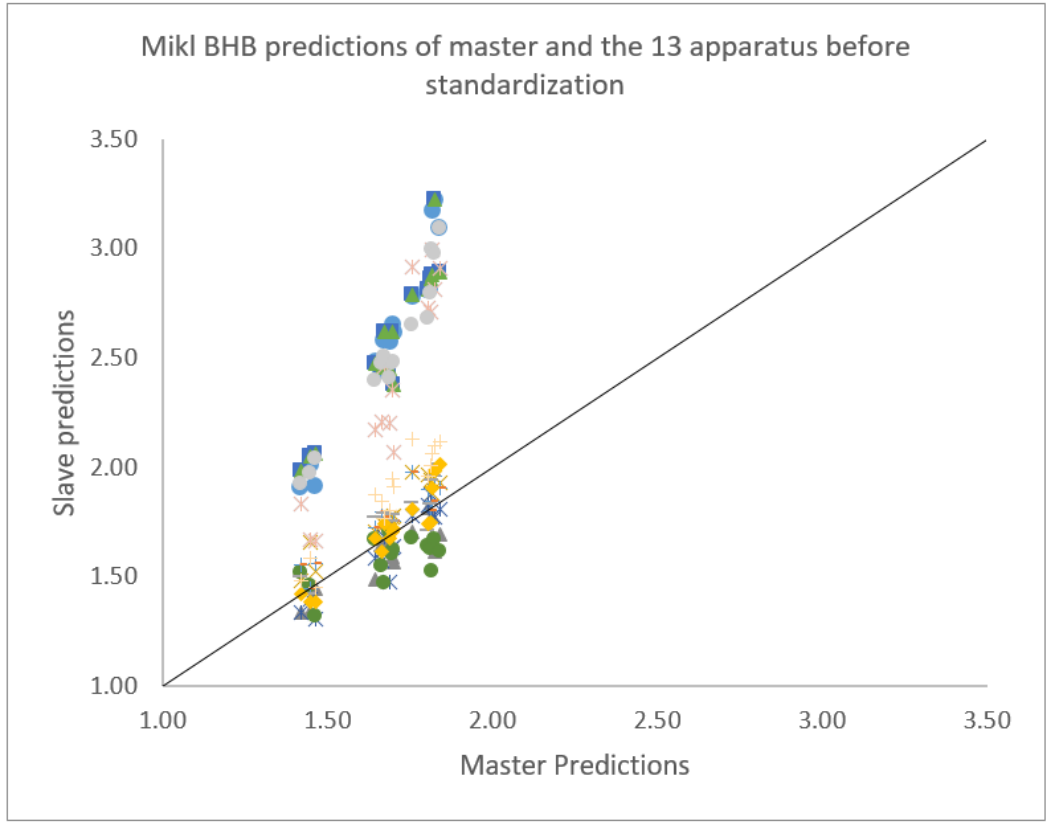
Results: BHB milk





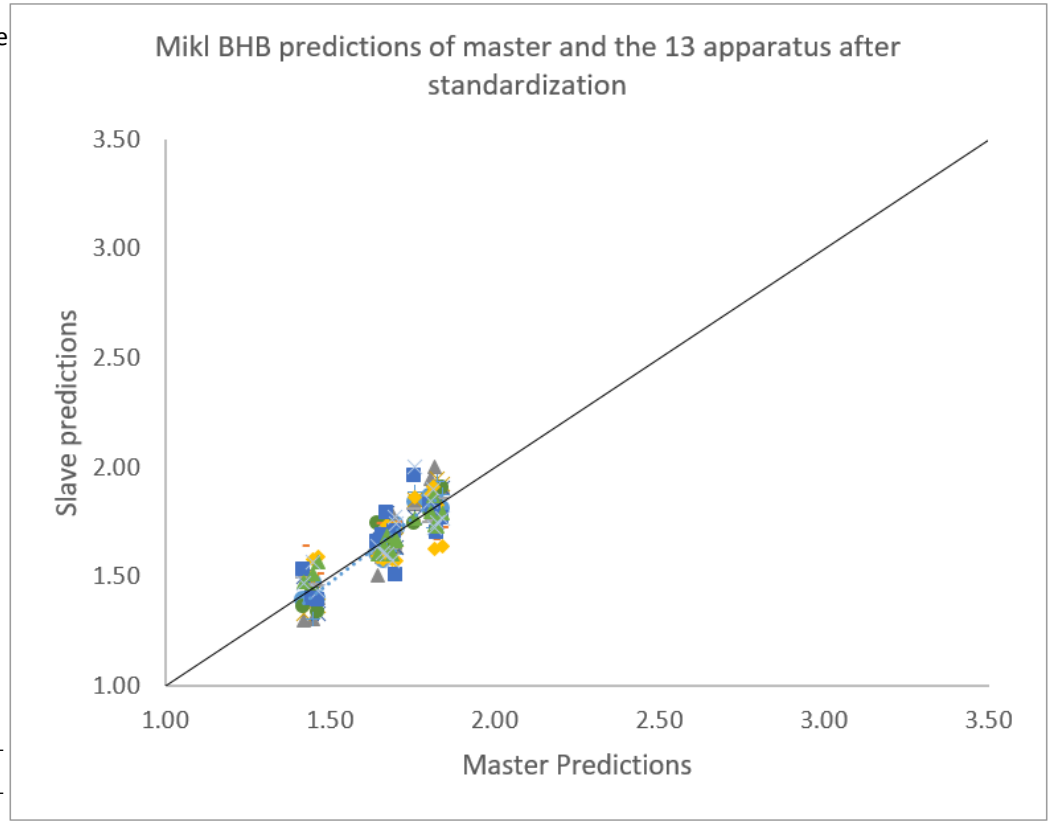
RMSE : 0.963 → RMSE : 0.050

Results: BHB milk



R for each sample

Before STD
0.253
0.228
0.269
0.460
0.466
0.482
0.355
0.366
0.366
0.583
0.599
0.556
0.409
0.412
0.357
0.411

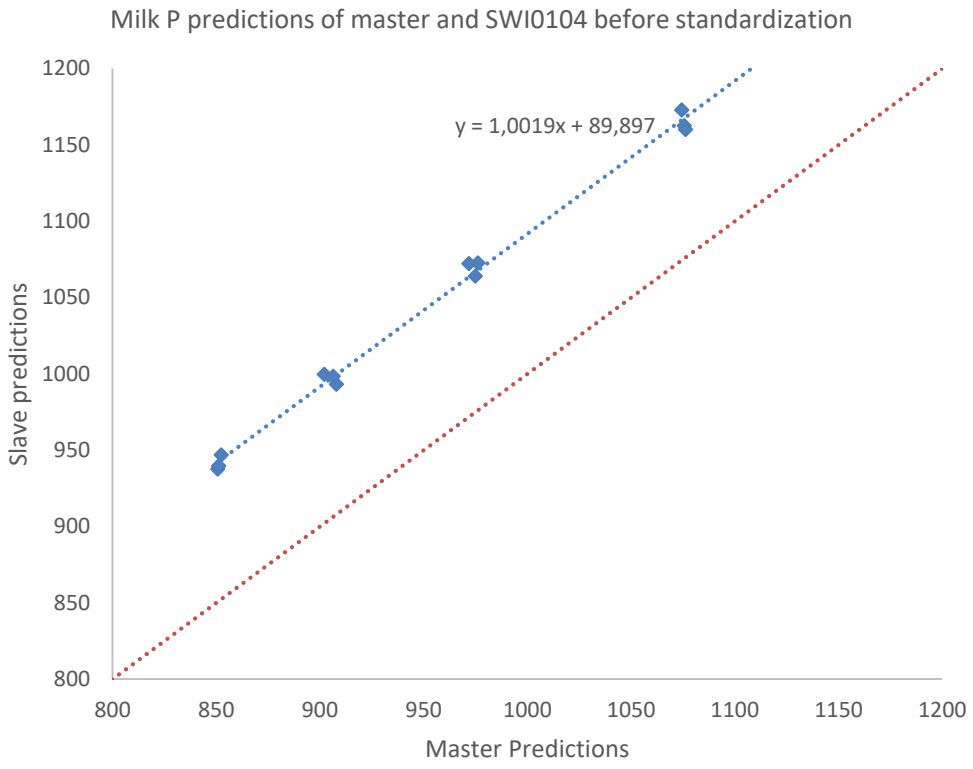


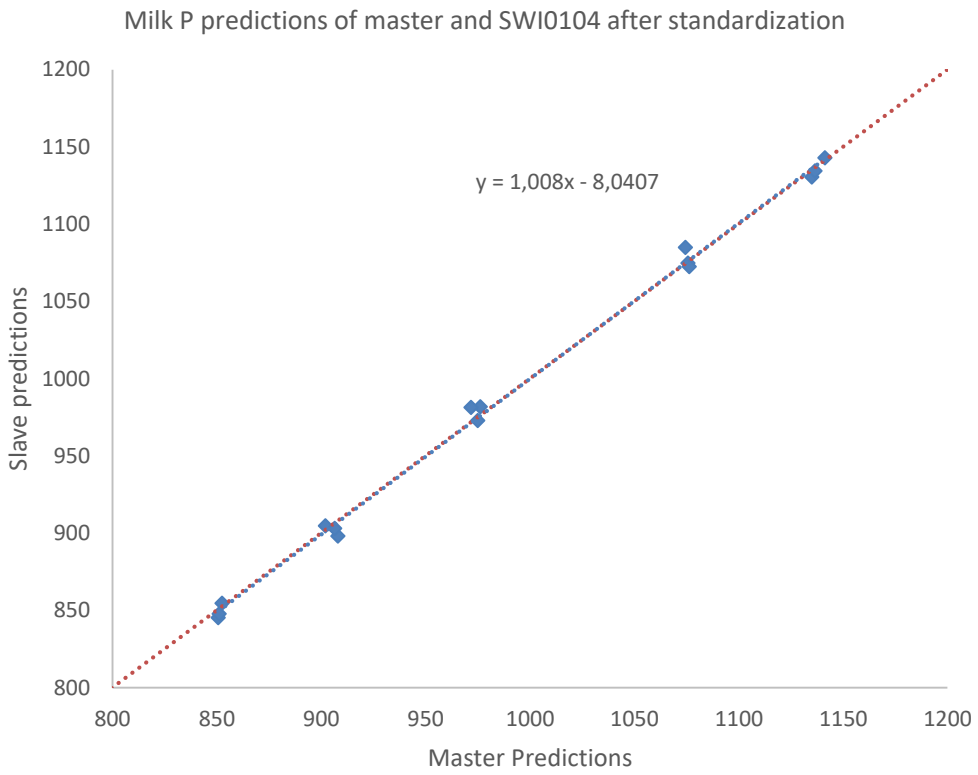
R for each sample

After STD
0.088
0.096
0.083
0.075
0.079
0.058
0.071
0.053
0.066
0.102
0.085
0.085
0.043
0.069
0.070
0.075

		Calibration								Validation		
BHB milk	Unit	# terms	#N	#WL	Mean	SD	RMSEc	R^2c	RPDc	#N	Mean	RMSEv
	Log(mmol/L)	9	419	212	0.225	0.171	0.086	0.75	1.97	124	0.156	0.065

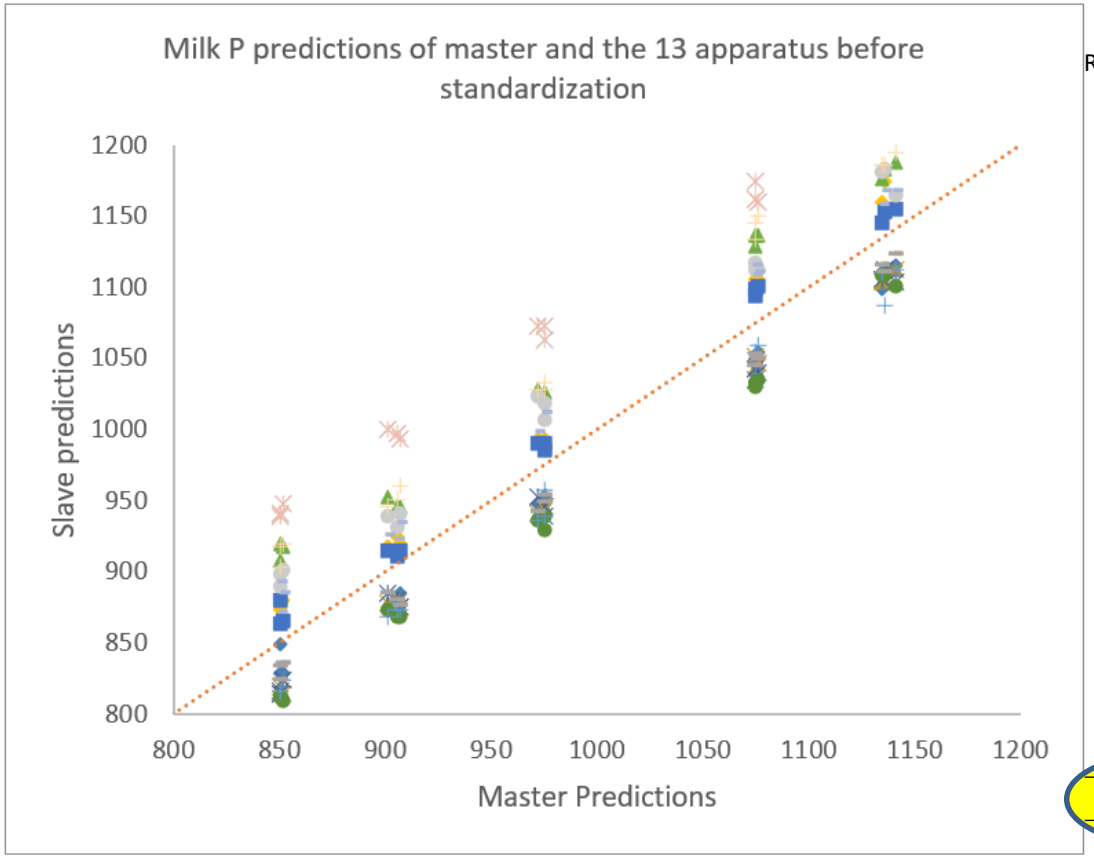
Results: Milk Phosphorous





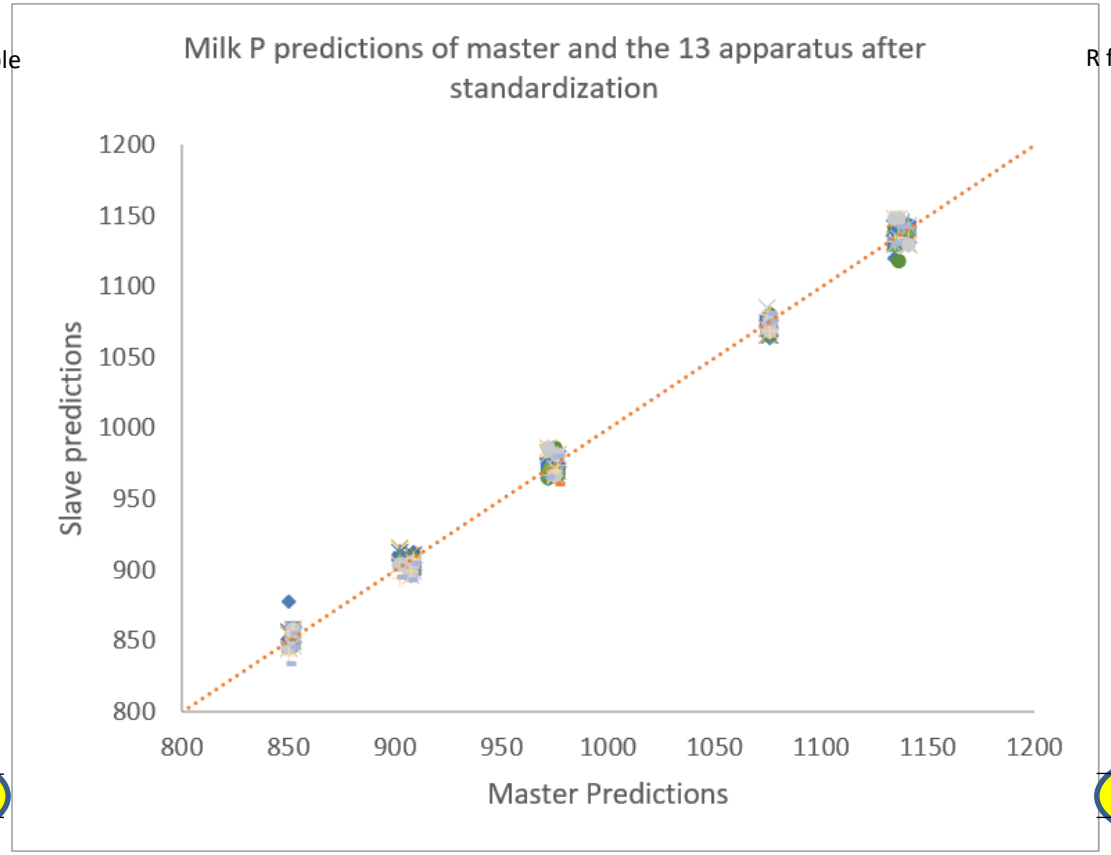
RMSE : 91.891 → RMSE : 5.430

Results: Milk Phosphorous



R for each sample

Before STD
41.177
46.135
46.834
39.785
40.239
38.811
44.527
46.751
43.949
40.210
41.807
43.208
45.186
44.427
40.900
42.930

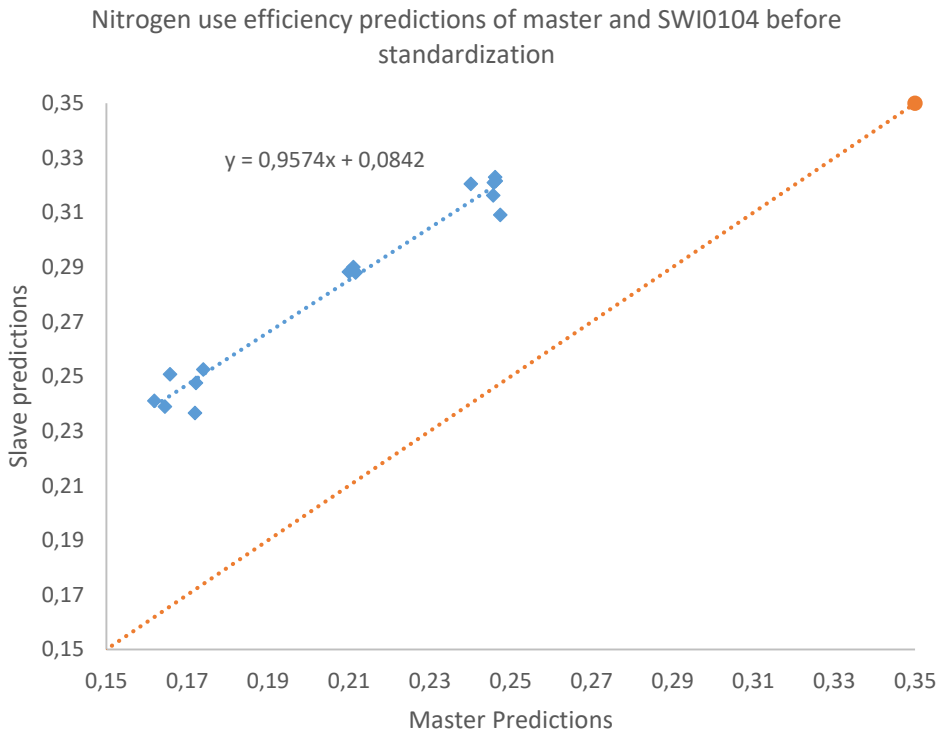


R for each sample

After STD
10.324
4.325
4.911
6.178
4.685
5.736
6.576
5.274
4.755
7.100
5.031
7.767
5.508
6.830
5.810
6.054

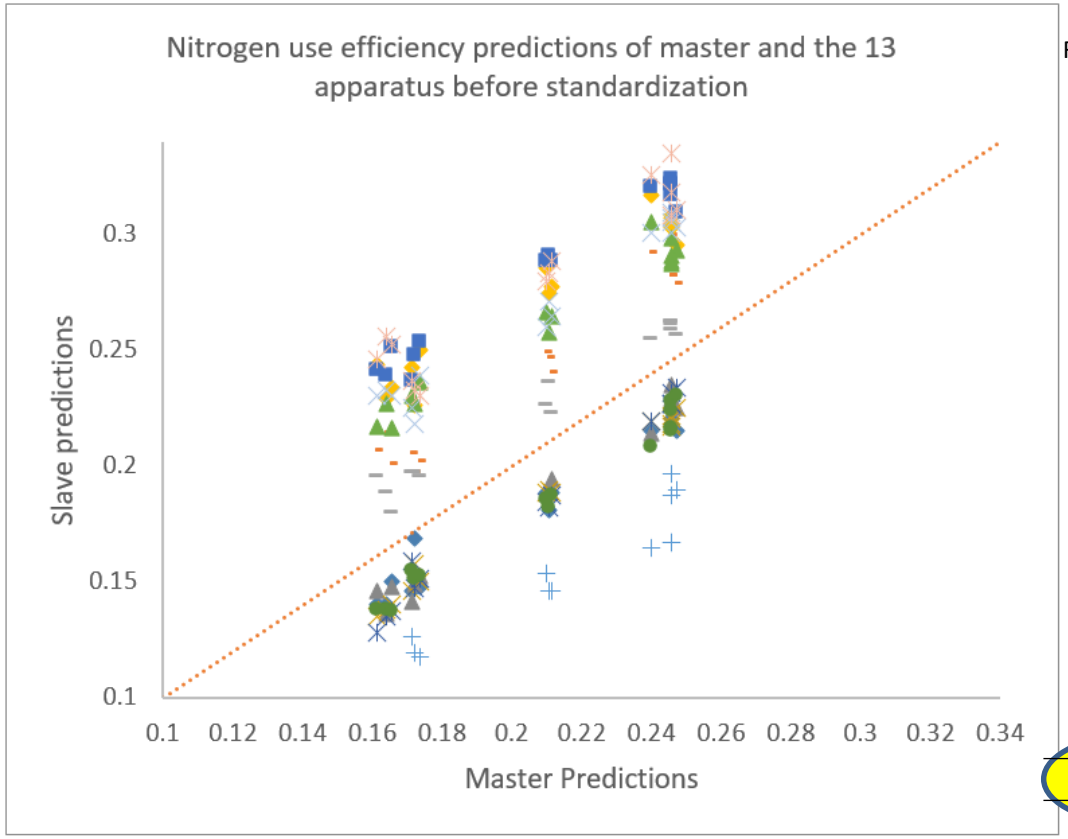
		Calibration									Cross validation with 4 subsets		
P	Unit	# terms	N	#WL	Mean	SD	Min	Max	SEC	R^2c	SECV	R^2cv	RPDcv
	mg/kg	10	1083	212	998.1	116.8	509	1447	57.4	0.76	58.71	0.75	1.99

Results: Nitrogen Use Efficiency (NUE)



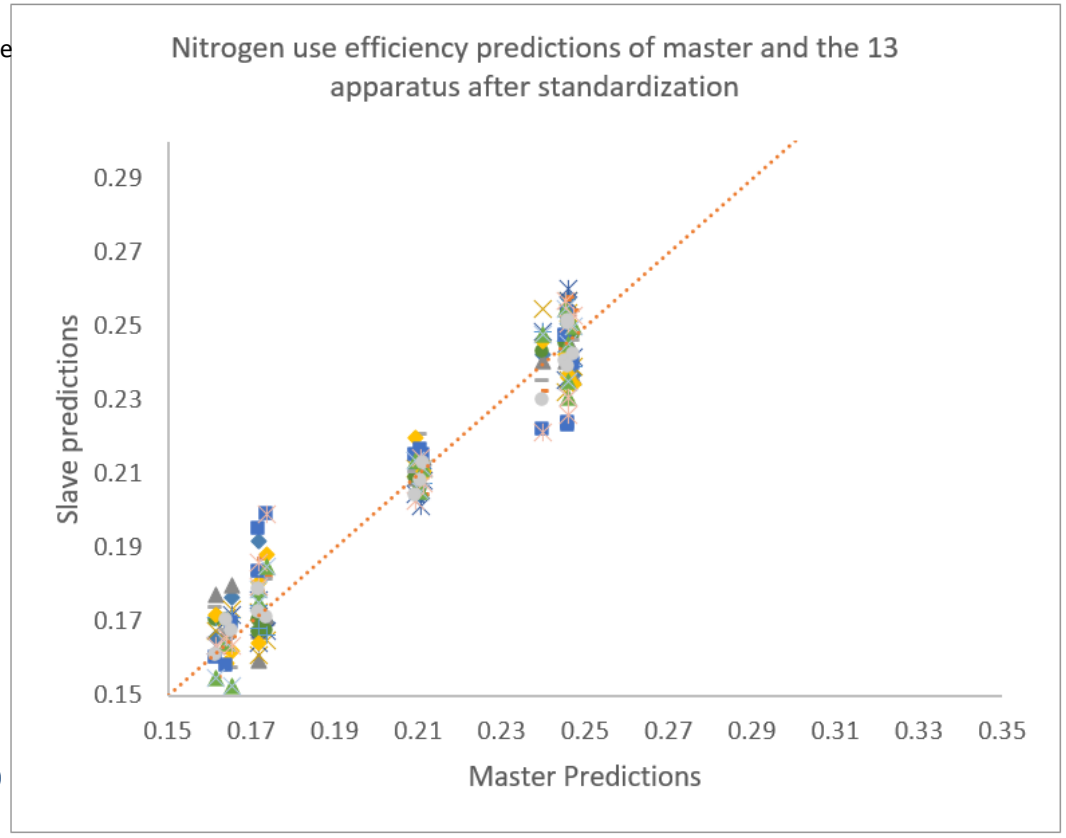
RMSE : 0.076 → RMSE : 0.014

Results: Nitrogen Use Efficiency (NUE)



R for each sample

Before STD
0.041
0.047
0.044
0.053
0.054
0.052
0.043
0.045
0.041
0.051
0.053
0.054
0.049
0.047
0.047
0.048



R for each sample

After STD
0.010
0.012
0.009
0.007
0.005
0.009
0.008
0.007
0.006
0.012
0.012
0.011
0.005
0.003
0.005
0.008

		Calibration								Cross-validation with 4 subset			External-cows-validation (25% cows out)		
NUE	N in milk/N Intake, daily ratio	# terms	N	min	max	Mean	SD	SEC	R^2c	SECV	R^2cv	RPDcv	SEP	R^2p	RPDp
		12	1033	0.10	0.82	0.37	0.10	0.05	0.73	0.06	0.71	1.87	0.05	0.69	1.91

Conclusion

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Electronic posters

S07(T)-PP-01 **„MastiMIR“ - A mastitis early warning system based on MIR spectra**
Laura Monica Dale

S07(T)-PP-02 **Prediction of evaluated energy balance (NEL and ME) in dairy cows by milk mid-infrared (MIR) spectra**
Laura Monica Dale

S07(T)-PP-03 **„KetoMIR2“ - Modelling of ketosis risk using vets diagnosis and MIR spectra for dairy cows in early lactation**
Laura Monica Dale

S07(T)-PP-05 **Large scale dataset to improve and validate the prediction of lactoferrin content using milk mid-infrared spectrometry**
Hélène Soyeurt

S07(T)-PP-06 **A first approach to predict nitrogen efficiency of dairy cows through milk FT-MIR spectra**
Clément Grelet

Important if you want to create historical spectral database !

Thank to all our partners

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