

New tools for the dairy sector based on MIR and NIR spectroscopy



Grelet C.¹, Dehareng F.¹, Vanlierde A.¹, Nguyen H.N.¹, Colinet F.² & Dardenne P.¹

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1. Classical use of IR in dairy sector
2. New tools to assess milk quality
3. New tools for dairy farmers
4. International networks
5. Lab to the sample



1. Classical use of IR in dairy sector



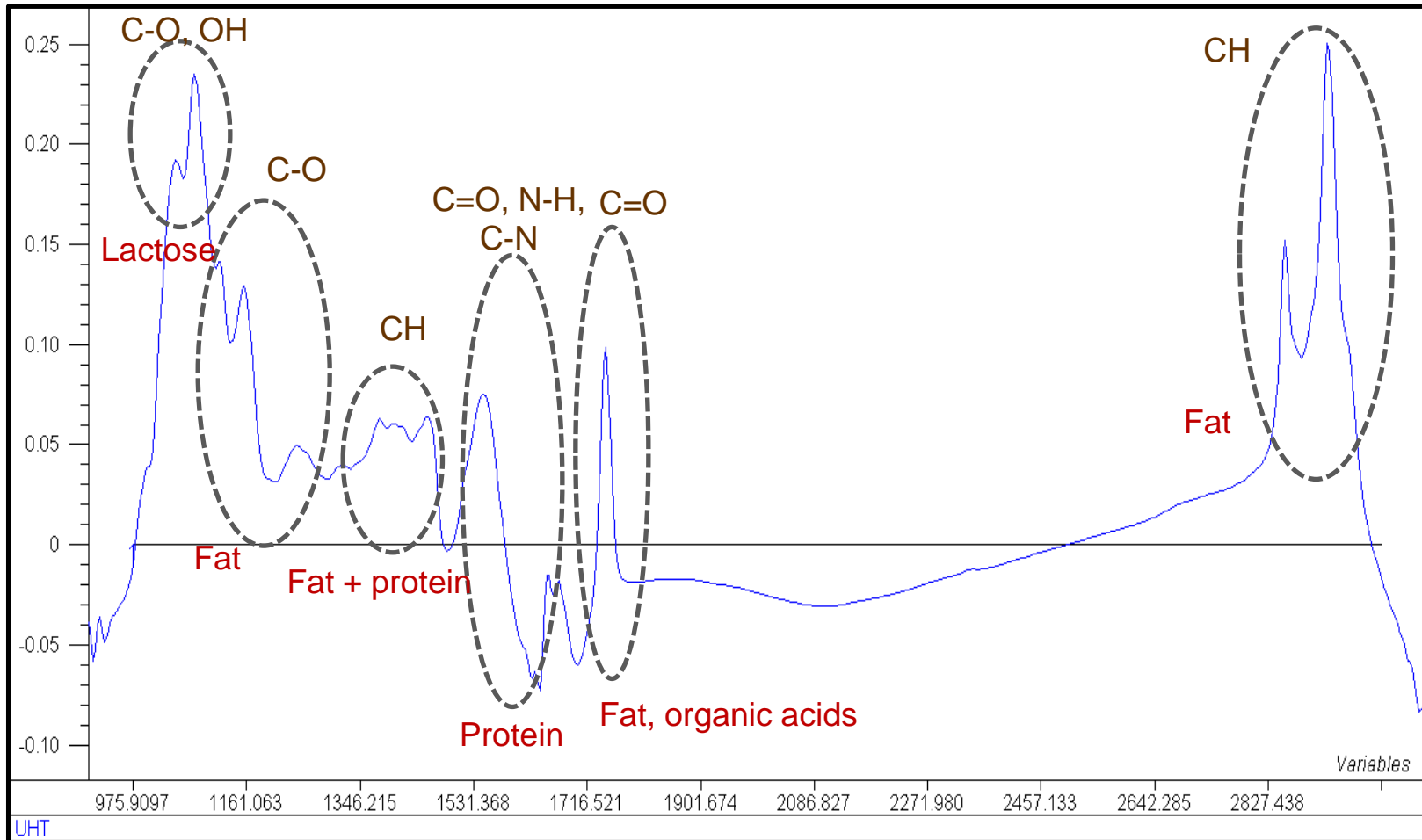
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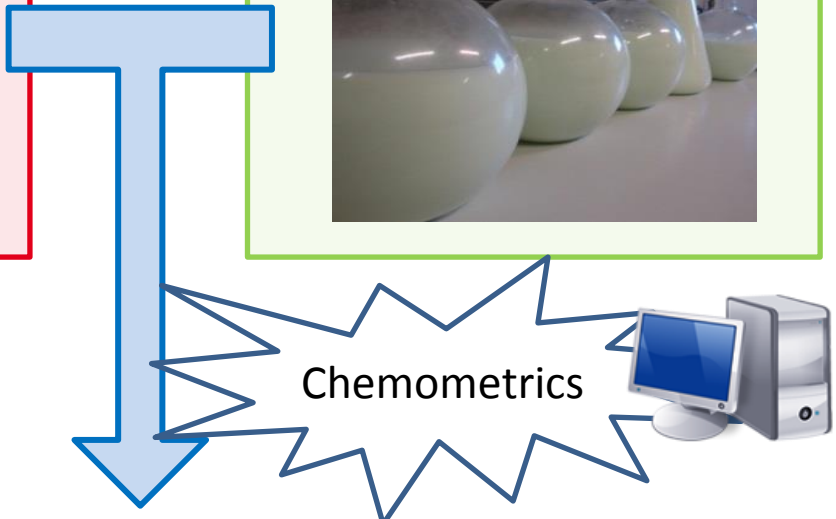
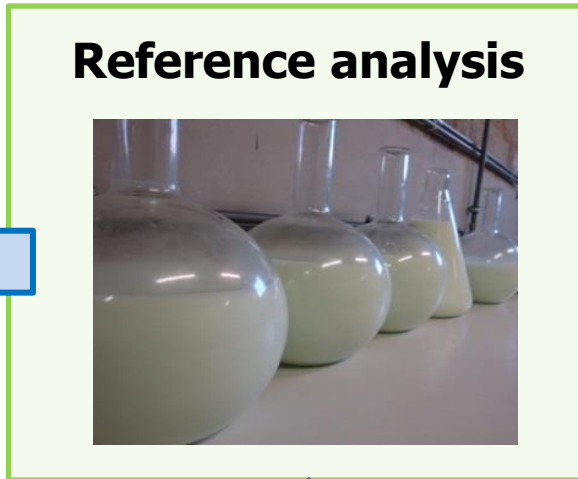
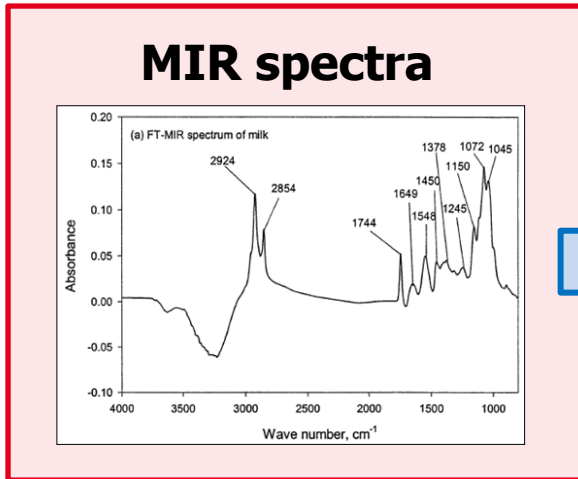
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MIR technology



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

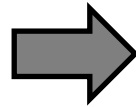
MIR predictions



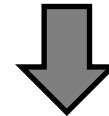
$$\begin{aligned}
 \text{Fat (g/100ml)} = & 0.0985 + 4.6191 *X_1 + 1.1659*X_2 + 1.4827*X_3 + 4.1684*X_4 + 7.3294*X_5 \\
 & + 9.8991*X_6 + 11.183*X_7 + 8.0711*X_8 + 2.1599*X_9 - 0.4619*X_{10} - 1.7876*X_{11} - 2.5708*X_{12} - \\
 & 2.8941*X_{13} - 2.9217*X_{14} - 2.7392*X_{15} - 2.2543*X_{16} - 1.2677*X_{17} + 0.0676*X_{18} + 1.0762*X_{19} \\
 & + 1.3228*X_{20} + 1.0241*X_{21} + 0.536*X_{22} + 0.0177*X_{23} - 0.5265*X_{24} - 1.1445*X_{25} - 1.8178*X_{26} + \\
 & 2.212*X_{27} - 2.0766*X_{28} + 8.3083*X_{29} - 3.703*X_{30} + 1.1999*X_{31} + 0.5698*X_{32} - 0.1674*X_{33} \\
 & + 0.246*X_{34} + 0.666*X_{35} + 1.2938*X_{36} + 2.0946*X_{37} - 0.0689*X_{38} - 1.4774*X_{39} - 1.7984*X_{40} - \\
 & 2.0553*X_{41} - 2.9338*X_{42} - 4.644*X_{43} - 6.764*X_{44} - 8.1475*X_{45} - 5.6904*X_{46} + 2.6657*X_{47} \\
 & + 10.9883*X_{48} + 14.4346*X_{49} + 13.8878*X_{50} + 10.2135*X_{51} + 4.8464*X_{52} - 1.2081*X_{53} - 7.4854*X_{54} - \\
 & 11.6799*X_{55} - 12.6849*X_{56} - 10.7724*X_{57} - 4.8936*X_{58} + 0.4425*X_{59} \\
 & + 3.583*X_{60} + \dots + 2.9636*X_{n-1} + 6.4566*X_n
 \end{aligned}$$

Classical use of MIR spectra

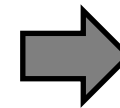
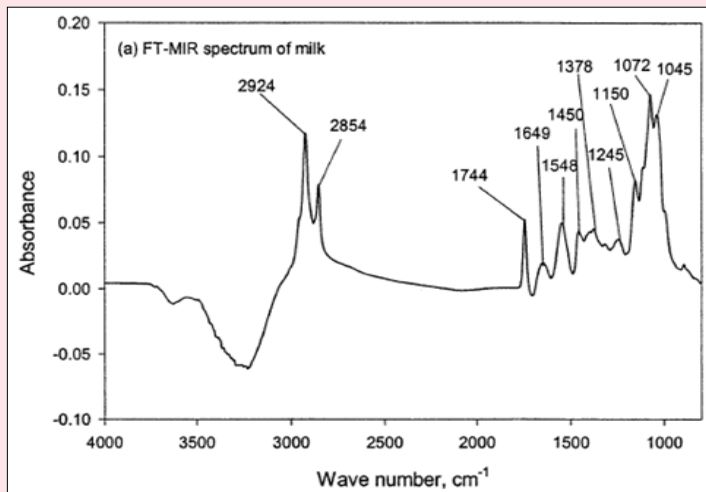
Milk control



MIR



MIR spectra of each cow



Milk composition

Fat
Proteins
Urea
Lactose
...

Fast
Cheap

2. New tools to assess milk quality



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Fatty Acids profile



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Mid-infrared prediction of bovine milk fatty acids across multiple breeds, production systems, and countries

H. Soyeurt,^{*†1,2} F. Dehareng,^{‡1} N. Gengler,^{*†} S. McParland,[§] E. Wall,[‡] D. P. Berry,[§] M. Coffey,[#] and P. Dardenne[‡]

Component (in g/100ml milk)	N	Mean	SD	SEC	R ² c	SECV	R ² cv	RPDcv
Fat	1745	3.913	0.939	0.041	1.00	0.041	1.00	22.69
C4:0	1759	0.103	0.028	0.008	0.92	0.008	0.92	3.49
C6:0	1770	0.072	0.021	0.006	0.93	0.006	0.92	3.60
C8:0	1761	0.047	0.014	0.004	0.93	0.004	0.92	3.66
C10:0	1753	0.111	0.035	0.009	0.93	0.010	0.92	3.60
C12:0	1760	0.134	0.042	0.011	0.93	0.011	0.93	3.74
C14:0	1757	0.446	0.121	0.030	0.94	0.030	0.94	3.97
C16:0	1768	1.190	0.386	0.094	0.94	0.095	0.94	4.07
C17:0	1756	0.027	0.007	0.003	0.81	0.003	0.80	2.24
C18:0	1758	0.389	0.134	0.053	0.84	0.055	0.83	2.44
C18:1 trans	1758	0.125	0.057	0.025	0.80	0.026	0.79	2.18
C18:1 cis9	1762	0.744	0.253	0.060	0.94	0.061	0.94	4.16
C18:1 cis	1761	0.801	0.273	0.062	0.95	0.062	0.95	4.40
C18:1	1761	0.929	0.300	0.059	0.96	0.060	0.96	5.00
Saturated FA	1764	2.682	0.724	0.072	0.99	0.074	0.99	9.74
Mono-unsaturated FA	1763	1.069	0.320	0.059	0.97	0.059	0.97	5.42
Poly-unsaturated FA	1760	0.158	0.045	0.022	0.77	0.022	0.76	2.05
Unsaturated FA	1762	1.231	0.361	0.063	0.97	0.065	0.97	5.59
Short chain FA	1759	0.348	0.096	0.023	0.94	0.023	0.94	4.14
Mid chain FA	1761	1.985	0.578	0.103	0.97	0.105	0.97	5.53
Long chain FA	1757	1.571	0.480	0.107	0.95	0.109	0.95	4.41
Odd FA	1755	0.156	0.038	0.016	0.82	0.016	0.82	2.36
Branched FA	1761	0.090	0.024	0.012	0.77	0.012	0.76	2.03
Trans FA	1762	0.159	0.069	0.028	0.83	0.029	0.82	2.36



Fatty Acids profile



Mid-infrared prediction of bovine milk fatty acids across multiple breeds, production systems, and countries

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C6:0	1770	0.072	0.021	0.006	0.93	0.006	0.92	3.60
C8:0	1761	0.047	0.014	0.004	0.93	0.004	0.92	3.66
C10:0	1753	0.111	0.035	0.009	0.93	0.010	0.92	3.60
C12:0	1760	0.134	0.042	0.011	0.93	0.011	0.93	3.74
C14:0	1757	0.446	0.121	0.030	0.94	0.030	0.94	3.97
C16:0	1768	1.190	0.386	0.094	0.94	0.095	0.94	4.07
C17:0	1756	0.027	0.007	0.003	0.81	0.003	0.80	2.24
C18:0	1758	0.389	0.134	0.053	0.84	0.055	0.83	2.44
C18:1 trans	1758	0.125	0.057	0.025	0.80	0.026	0.79	2.18
C18:1 cis9	1762	0.744	0.253	0.060	0.94	0.061	0.94	4.16
C18:1 cis	1761	0.801	0.273	0.062	0.95	0.062	0.95	4.40
C18:1	1761	0.929	0.300	0.059	0.96	0.060	0.96	5.00
Saturated FA	1764	2.682	0.724	0.072	0.99	0.074	0.99	9.74
Mono-unsaturated FA	1763	1.069	0.320	0.059	0.97	0.059	0.97	5.42
Poly-unsaturated FA	1760	0.158	0.045	0.022	0.77	0.022	0.76	2.05
Unsaturated FA	1762	1.231	0.361	0.063	0.97	0.065	0.97	5.59
Short chain FA	1759	0.348	0.096	0.023	0.94	0.023	0.94	4.14
Mid chain FA	1761	1.985	0.578	0.103	0.97	0.105	0.97	5.53
Long chain FA	1757	1.571	0.480	0.107	0.95	0.109	0.95	4.41
Odd FA	1755	0.156	0.038	0.016	0.82	0.016	0.82	2.36
Branched FA	1761	0.090	0.024	0.012	0.77	0.012	0.76	2.03
Trans FA	1762	0.159	0.069	0.028	0.83	0.029	0.82	2.36
C14:1 cis	1757	0.038	0.014	0.008	0.71	0.008	0.70	1.83
C16:1 cis	1763	0.065	0.024	0.012	0.73	0.013	0.72	1.88
C18:2	1748	0.096	0.026	0.015	0.68	0.015	0.67	1.73
C18:2 cis-9, cis-12	1743	0.062	0.022	0.012	0.74	0.012	0.72	1.89
C18:3 cis-9, cis-12, cis -15	1749	0.020	0.008	0.004	0.70	0.004	0.68	1.77
C18:2 cis 9, Trans 11	1739	0.028	0.020	0.010	0.75	0.010	0.74	1.94
Omega 3	1743	0.026	0.009	0.005	0.69	0.005	0.68	1.74
Omega 6	1750	0.103	0.028	0.015	0.71	0.016	0.69	1.81



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C14:0	1757	0.446	0.121	0.030	0.94	0.030	0.94	3.97
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C17:0	1756	0.027	0.007	0.003	0.81	0.003	0.80	2.24

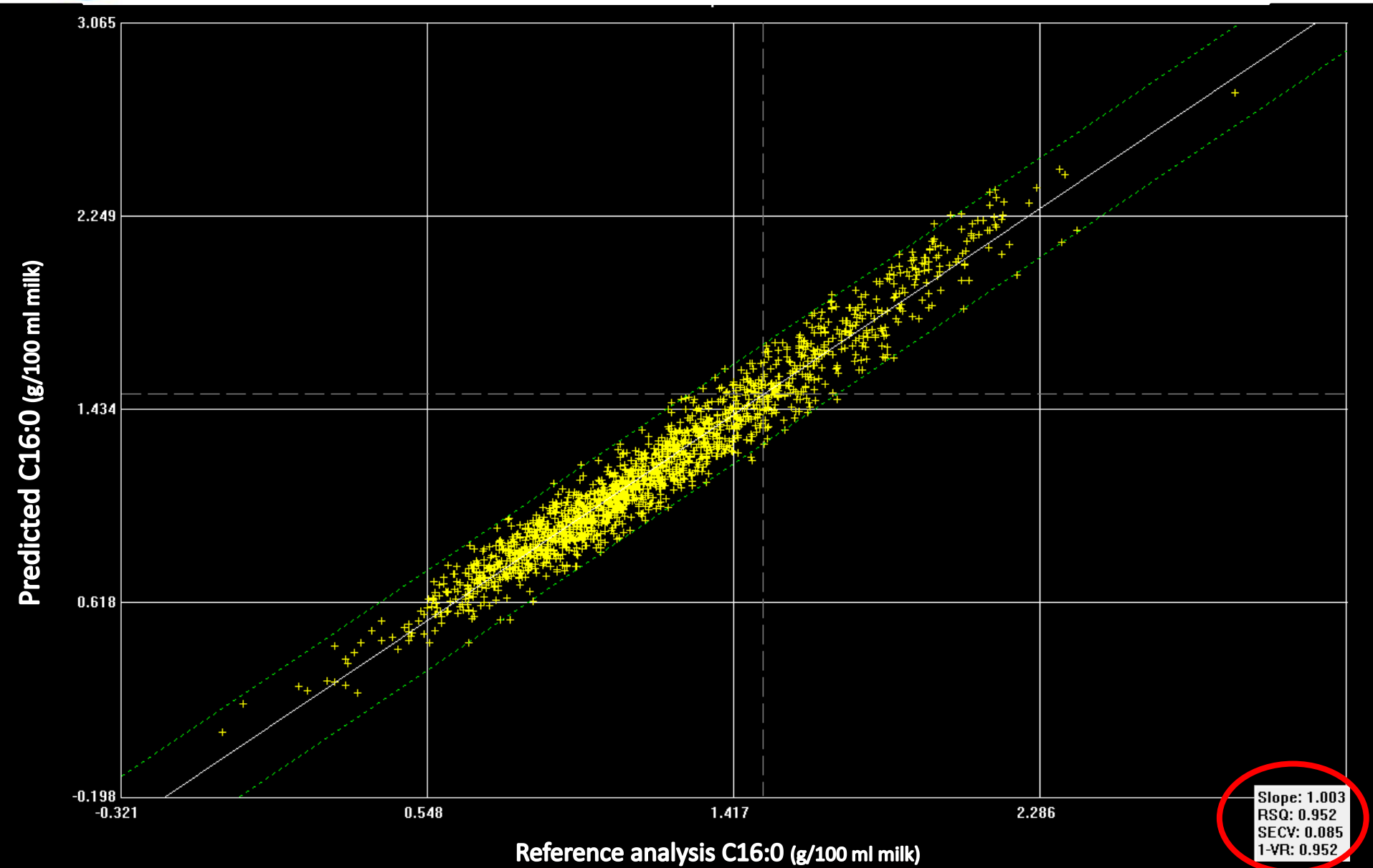
Robustness:

- ✓ Milks from Belgium, Luxembourg, Ireland, UK, France and Germany
- ✓ Different breeds
- ✓ Different feeding systems

C18:0	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
C18:1 trans	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
C18:1 cis9	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
C18:1 cis	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
C18:1	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
Saturated FA	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
Mono-unsaturated	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
Poly-unsaturated	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
Unsaturated FA	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
Short chain FA	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
Mid chain FA	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
Long chain FA	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
Odd FA	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
Branched FA	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
Trans FA	1758	0.000	0.000	0.000	0.00	0.000	0.00	0.00
C14:1 cis	1757	0.038	0.014	0.008	0.71	0.008	0.70	1.83
C16:1 cis	1763	0.065	0.024	0.012	0.73	0.013	0.72	1.88
C18:2	1748	0.096	0.026	0.015	0.68	0.015	0.67	1.73
C18:2 cis-9, cis-12	1743	0.062	0.022	0.012	0.74	0.012	0.72	1.89
C18:3 cis-9, cis-12, cis -15	1749	0.020	0.008	0.004	0.70	0.004	0.68	1.77
C18:2 cis 9, Trans 11	1739	0.028	0.020	0.010	0.75	0.010	0.74	1.94
Omega 3	1743	0.026	0.009	0.005	0.69	0.005	0.68	1.74
Omega 6	1750	0.103	0.028	0.015	0.71	0.016	0.69	1.81



e.g. C16:0





Minerals

J. Dairy Sci. 92:2444–2454

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Potential estimation of major mineral contents in cow milk using mid-infrared spectrometry

H. Soyeurt,^{*1} D. Bruwier,^{*} J.-M. Romnee,[†] N. Gengler,^{*‡} C. Bertozzi,[§] D. Veselko,[#] and P. Dardenne[†]

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[†]Walloon Agricultural Research Centre, Quality Department, B-5030 Gembloux, Belgium

[‡]National Fund for Scientific Research, B-1000 Brussels, Belgium

[§]Walloon Breeding Association, B-5590 Ciney, Belgium

[#]Milk Committee, B-4651 Battice, Belgium

Component (mg/kg milk)	#N	Mean	SD	SEC	R ² c	SECV	R ² cv	RPDcv
Sodium	815	358.7	78.9	55.2	0.51	55.9	0.50	1.41
Calcium	899	1167.5	147.9	61.3	0.83	63.3	0.82	2.33
Phosphorus	812	1017.1	112.6	54.8	0.76	56.4	0.75	2.00
Magnesium	885	101.2	12.9	7.0	0.71	7.3	0.68	1.78
Potassium	877	1514.1	154.7	100.6	0.58	104.6	0.54	1.48



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Potassium	877	1514.1	154.7	100.6	0.58	104.6	0.54	1.48



Ability to make cheese

Cheese making	#N	Mean	SD	SEC	R ² c	SECV	R ² cv	RPDcv
Proteins (g/100g milk)	4322	3.36	0.4	0.019	0.99	0.02	0.99	20.3
Caseins (g/100g milk)	845	2.68	0.35	0.077	0.95	0.079	0.95	4.43
pH	318	6.62	0.1	0.057	0.64	0.061	0.6	1.57
Titration acidity (°D)	451	16.63	1.8	0.77	0.82	0.8	0.8	2.25
Rennet Coagulation Time (RCT, s)	252	899	213	123	0.67	136	0.59	1.57
Curd-firming time (k20, s)	268	221	34	25.4	0.44	27.1	0.36	1.25
Curd firmness 30 minutes after rennet addition (a30, mm)	212	34.5	4.1	2.8	0.54	3.1	0.44	1.33
Cheese yield fresh (g coagulum/100g milk)	258	26.80	6.50	2.60	0.83	2.80	0.81	2.27
Cheese yield DM (g DM coagulum/100g DM milk)	243	61.88	6.40	2.58	0.84	2.73	0.82	2.34



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But also:

Beta-hydroxybutyrate (Roos et Al., 2007)

Acetone (Heuer et Al., 2001...)

Proteins profile (Bonfatti et Al., 2011...)

Milk contaminants

e.g. **Melamine detection** (Smirnov, 2011...)

...

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3. New tools for dairy farmer: OptiMIR Project

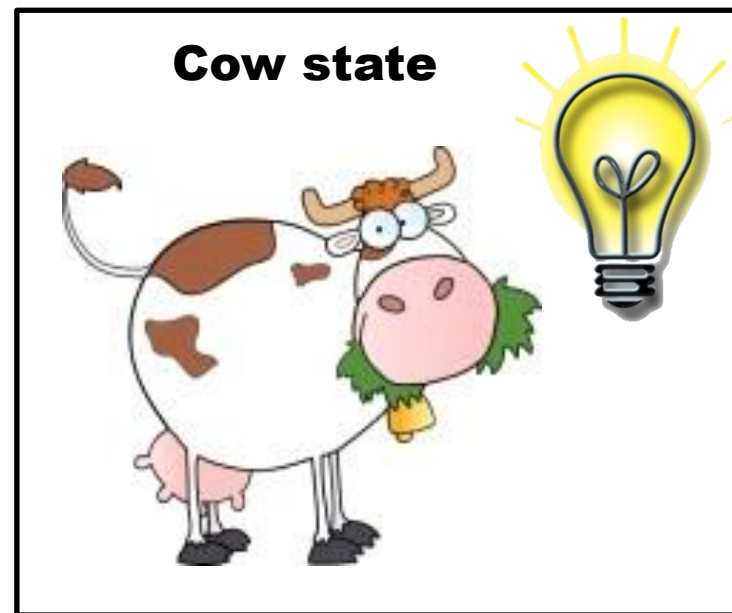
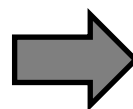
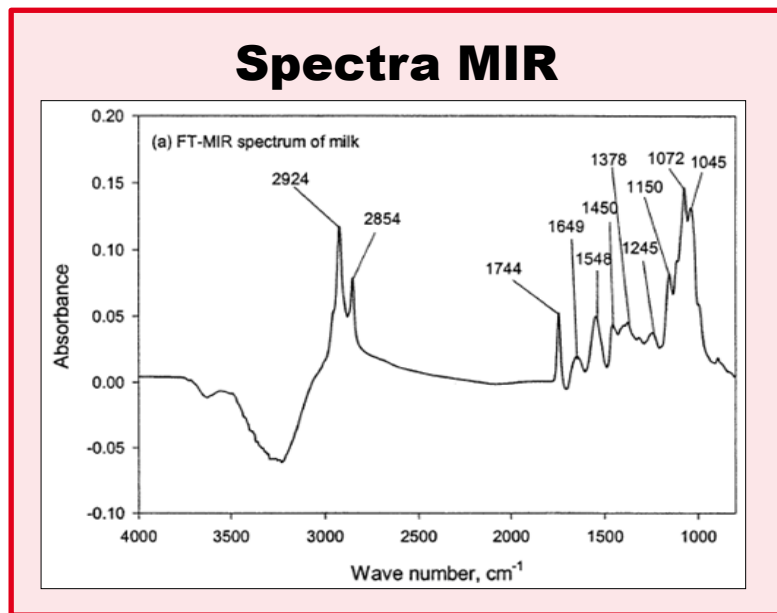


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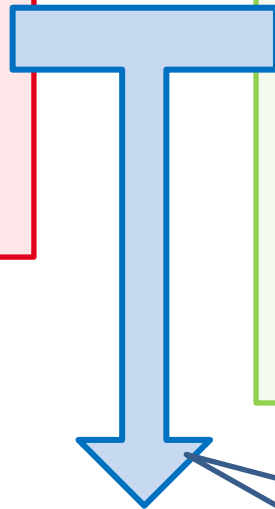
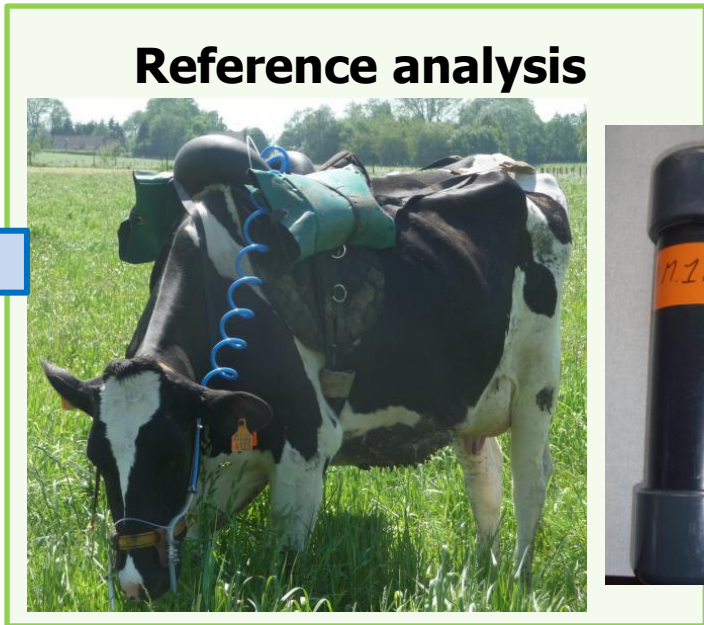
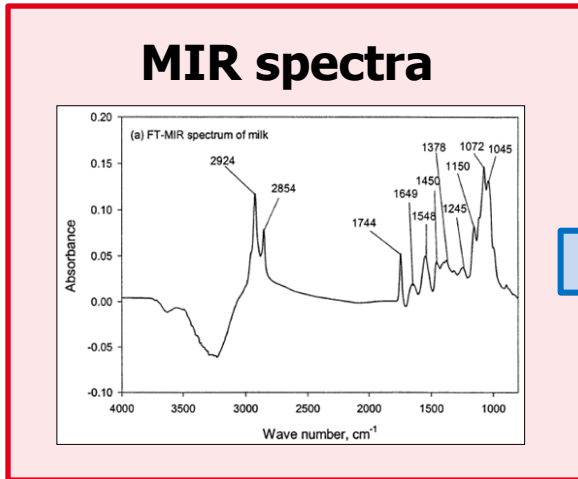


Prediction tools fast, cheap, via milk control organisations

- Informations on :
- **fertility** (pregnancy...)
 - **feeding** (acidosis, ketosis, energy balance...)
 - **health** (mastitis...)
 - **environmental impact** (methane...)

NEW

Methane emissions



Chemometrics



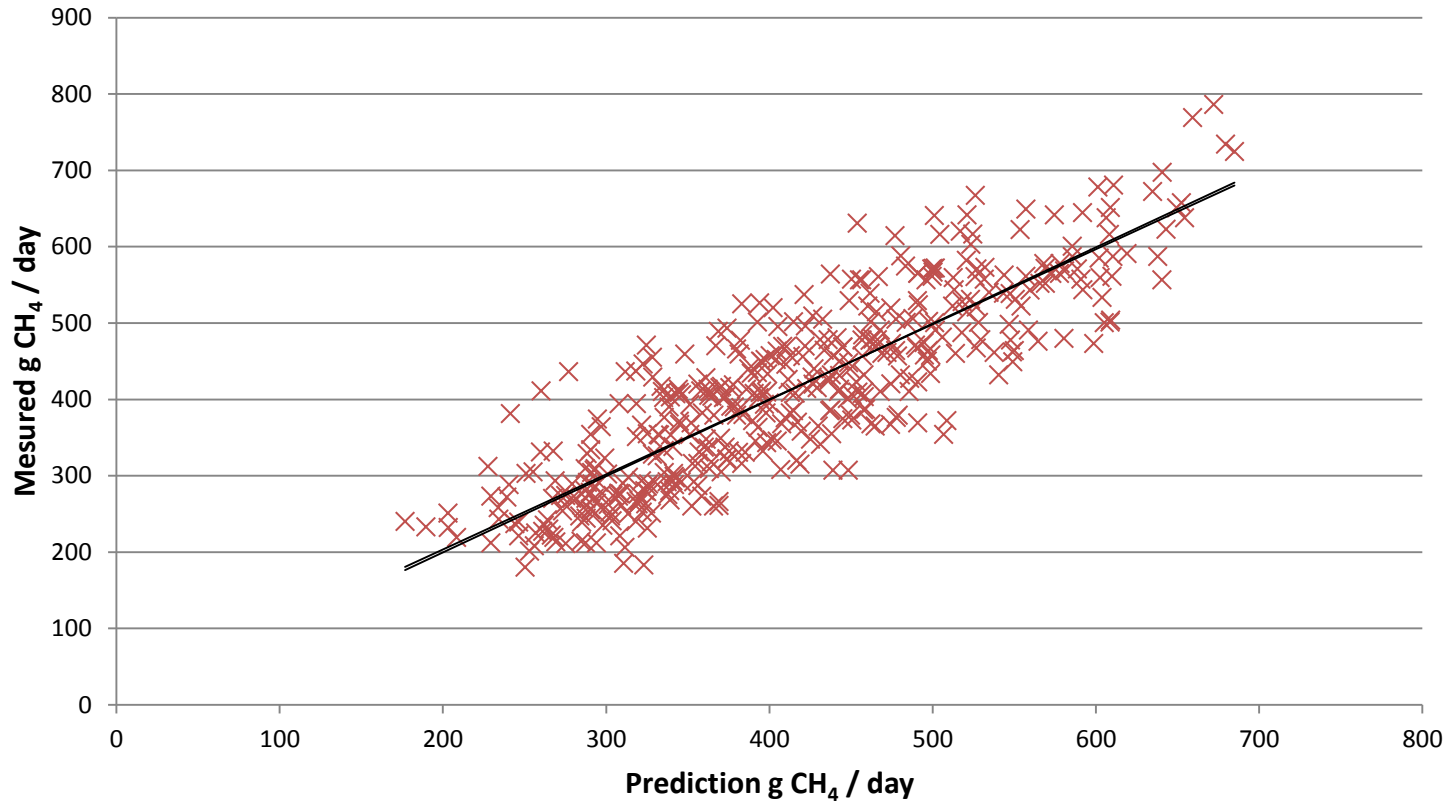
Equation to predict methane production by dairy cows

Walloon Agricultural Research Centre





Methane production



Phenotypic calibrations	#N	SD	SEC	R ² c	SECV	R ² cv	RPDcv
Methane (g/v/j)	452	126	62	0.76	69	0.70	1.83

Walloon Agricultural Research Centre



New tools = new perspectives

Milk composition

- ✓ Assess Nutritional quality
- ✓ Adapt process to milk quality
- ✓ Quality control
- ✓ Geographic origin determination
- ✓ Traceability...

Cow status

- ✓ Reduce economic losses
 - Avoid metabolic disorders
 - Adapt feeding
 - Improve fertility management...
- ✓ Bring the dairy sector more efficient
 - Genetic selection....

4. International networks



Grelet C.¹, Dehareng F.¹, Nguyen H.N.¹, Vanlierde A.¹, Colinet F.² & Dardenne P.¹

¹ *Walloon Agricultural Research Center (CRA-W), Gembloux, Belgique*

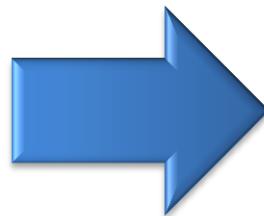
² *University of Liège, Gembloux Agro-Bio Tech, Gembloux, Belgique*

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Issue:

To create new calibration we need:

- 1. Reference Analysis**
- 2. Variability**



Important costs!

Solutions : international collaboration



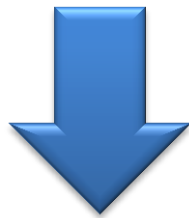


Creation of the first milk spectral database



Walloon level → European level

Integration of new organisation by adding samples



- ✓ Important database
- ✓ Great variability (geographic, breed, feeding systems, seasons...) : increasing robustness
- ✓ Shared costs

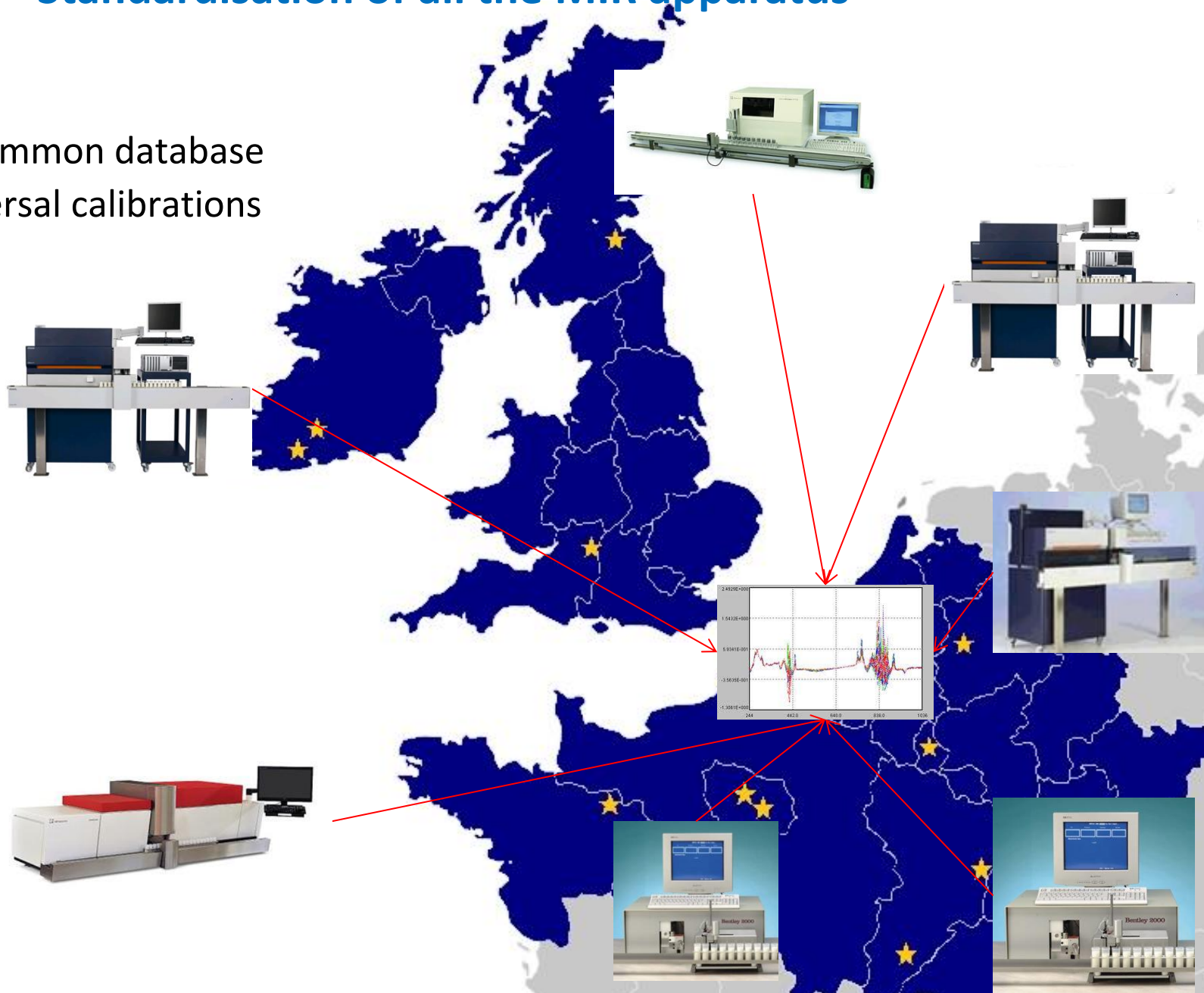
We are open to collaborations





Standardisation of all the MIR apparatus

- ✓ Create common database
- ✓ Use universal calibrations



5. Lab to the sample : milkinir project

An exemple of On line application



Grelet C.¹, Dehareng F.¹, Vanlierde A.¹, Nguyen H.N.¹, Colinet F.² & Dardenne P.¹

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On line measurement



MILKINIR: Development of an automatic system to measure milk composition and quality during milking



Objectives:



- ✓ On-Line measure
- ✓ Research: cinetic of milking
- ✓ Farm management: daily predictions directly during milking

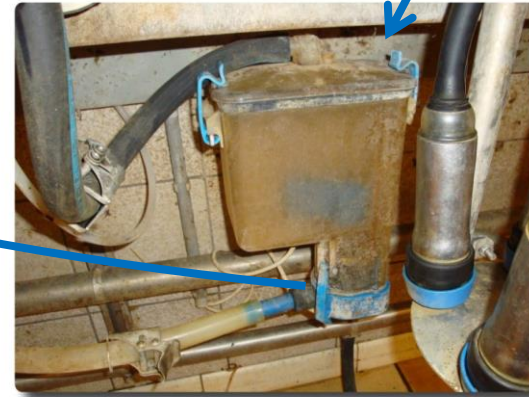


NEW

First NIR automatic sensing system at the CRA-W milking room



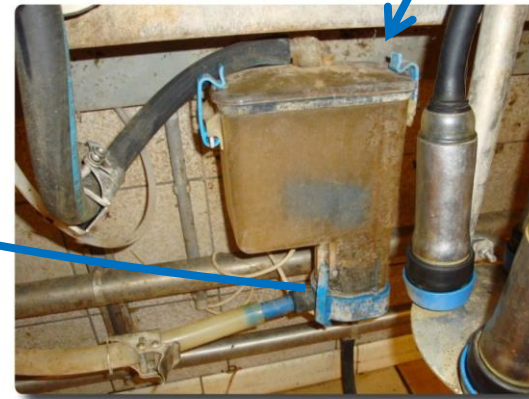
Fiber Optic



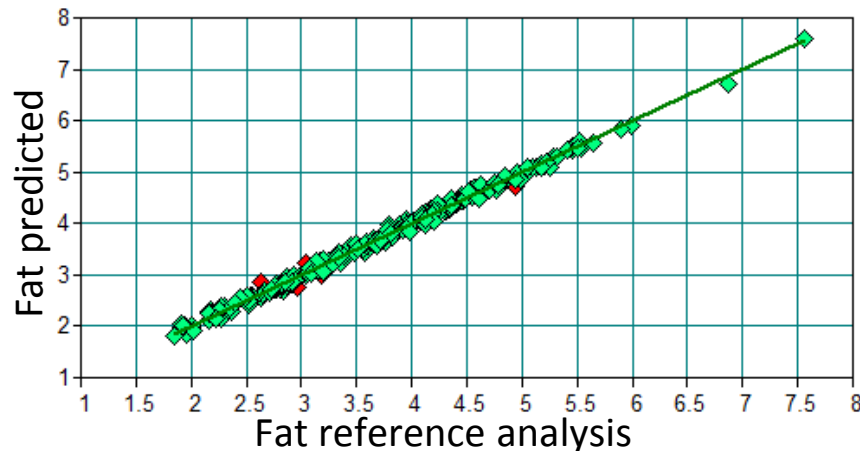
- NIR: Lower price
 - Allows analyze non-homogenized milk (optic pathway)
 - More robust
- NIR spectrometer not in the milking room
- Fiber Optic to deliver and collect the radiation between spectrometer and sample

NEW

First NIR automatic sensing system at the CRA-W milking room



Fiber Optic



Fat	
$R^2_{\text{Cross Validation}}$ *	99.43
$RMSE_{CV}$ **	0.0735
RPD***	13.3

Centre wallon de Recherches agronomiques



Classical NIR predictions On Line

Component	R^2_{cv} * [%]	RMSE _{cv} *	RPD *	N	Range	Units
Fat	99.5	0.07	13.6	293	1.85 - 7.55	g/100g
Prot	96.9	0.07	5.7	290	2.46 - 4.38	g/100g
Casein	98.8	0.06	9	59	1.91 - 4.02	g/100g
Lactose	71.5	0.09	1.9	441	3.95 - 5.28	g/100g
DM	97.4	0.15	6.3	94	10.77 - 16.74	g/100g
FFA	32	2.79	1.2	239	0.24 - 16.97	meq/100g Fat
Cells	1.6	256	1	234	119 - 1099	x1000/ml
Urea	23.2	9	1.1	468	1 - 50	mg/100g
pH	18.7	0.05	1.1	68	6.44 - 6.92	-
Cryoscopy	98.3	10	7.6	42	247 - 537	m°C

Classical NIR predictions On Line

Component	R ² _{cv} * [%]	RMSE _{cv} *	RPD *	N	Range	Units
Fat	99.5	0.07	13.6	293	1.85 - 7.55	g/100g
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Component	R ² _{cv} * [%]	RMSE _{cv} *	RPD *	N	Range
FA profile					
C4:0	51.6	0.02	1.4	41	0.02 - 0.14
C6:0	64.1	0.01	1.7	41	0.02 - 0.12
C8:0	47.7	0.01	1.4	41	0.02 - 0.08
C10:0	48.4	0.03	1.4	41	0.04 - 0.20
C12:0	67.6	0.03	1.8	40	0.05 - 0.25
C14:0	81.3	0.07	2.3	41	0.20 - 0.95
C14:1	34.1	0.01	1.2	40	0.02 - 0.09
C16:0	77.6	0.17	2.1	41	0.47 - 2.09
C16:1	87.8	0.02	2.9	41	0.03 - 0.23
C17:0	67.2	0.005	1.8	40	0.012 - 0.054
C18:0	81.3	0.08	2.3	41	0.16 - 0.91
C18:1 trans	20.9	0.02	1.1	40	0.03 - 0.13
C18:1 cis	93.4	0.15	3.9	39	0.47 - 2.69
C18:2	64.7	0.01	1.7	40	0.04 - 0.15
C18:3	76.1	0.01	2	39	0.03 - 0.12
CLA	35.5	0.004	1.3	36	0.010 - 0.029
SAT	93.6	0.18	4	40	1.09 - 4.81
MONO	92.5	0.17	3.7	40	0.60 - 3.05
POLY	66	0.02	1.7	36	0.06 - 0.21
UNSAT	97.9	0.09	6.9	42	0.69 - 3.23
SCFA	90	0.03	3.2	41	0.12 - 0.65
MCFA	94.8	0.13	4.4	42	0.85 - 3.62
LCFA	96.9	0.14	5.7	42	0.84 - 3.85
Omega-3	34.8	0.01	1.2	40	0.01 - 0.05
Omega-6	72.6	0.01	2	41	0.04 - 0.18



Classical NIR predictions On Line

Component	R ² _{cv} * [%]	RMSE _{cv} *	RPD *	N	Range	Units
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Prot	96.9	0.07	5.7	290	2.46 - 4.38	g/100g
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Urea	23.2	9	1.1	468	1 - 50	mg/100g
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Component	R ² _{cv} * [%]	RMSE _{cv} *	RPD *	N	Range
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C4:0	51.6	0.02	1.4	41	0.02 - 0.14
C6:0	64.1	0.01	1.7	41	0.02 - 0.12
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C10:0	48.4	0.03	1.4	41	0.04 - 0.20
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C14:0	81.3	0.07	2.3	41	0.20 - 0.95
C14:1	34.1	0.01	1.2	40	0.02 - 0.09
C16:0	77.6	0.17	2.1	41	0.47 - 2.09
C16:1	87.8	0.02	2.9	41	0.03 - 0.23
C17:0	67.2	0.005	1.8	40	0.012 - 0.054
C18:0	81.3	0.08	2.3	41	0.16 - 0.91
C18:1 trans	20.9	0.02	1.1	40	0.03 - 0.13
C18:1 cis	93.4	0.15	3.9	39	0.47 - 2.69
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MONO	92.5	0.17	3.7	40	0.60 - 3.05
POLY	66	0.02	1.7	36	0.06 - 0.21
UNSAT	97.9	0.09	6.9	42	0.69 - 3.23
SCFA	90	0.03	3.2	41	0.12 - 0.65
MCFA	94.8	0.13	4.4	42	0.85 - 3.62
LCFA	96.9	0.14	5.7	42	0.84 - 3.85
Omega-3	34.8	0.01	1.2	40	0.01 - 0.05
Omega-6	72.6	0.01	2	41	0.04 - 0.18

Component	R ² _{cv} * [%]	RMSE _{cv} *	RPD *	N	Range
Minerals					
Ca	61.3	108	1.6	50	908 - 1578
P	69.9	117	1.7	50	831 - 1696
Mg	24.3	13	1.2	50	86 - 152
K	35.9	130	1.3	51	966 - 1729

Thank you for your attention



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