Taiwan Innotech Expo
台灣創新技術博覽會

乳質檢測儀器國際趨勢研討會
Gobal Application of Milk Quality Analytic Instruments

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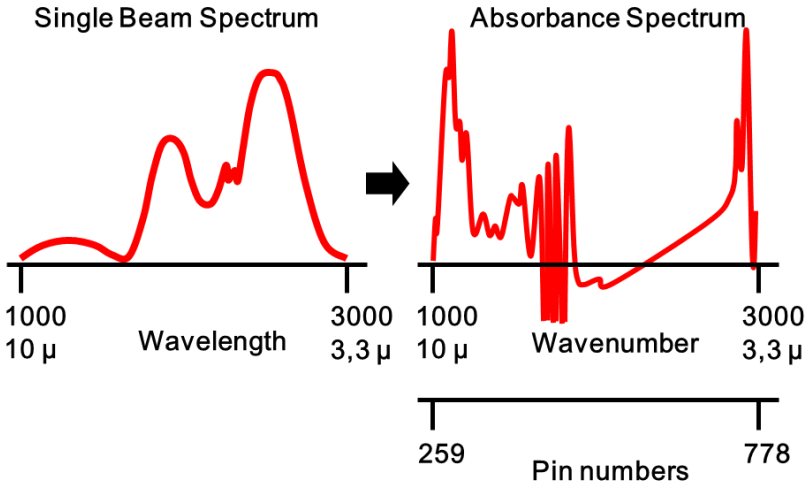
ANALYTICS BEYOND MEASURE

FTIR技術在乳品質快速檢測之應用
Application of FTIR Technology in Milk Quality Analysis



孫丕忠 Terence Sun

今日儀器股份有限公司 Today's Instruments Co., Ltd.

September 24th 2020, Taipei, Taiwan

FTIR 檢測技術之應用 What Can FTIR Offer Us Today?

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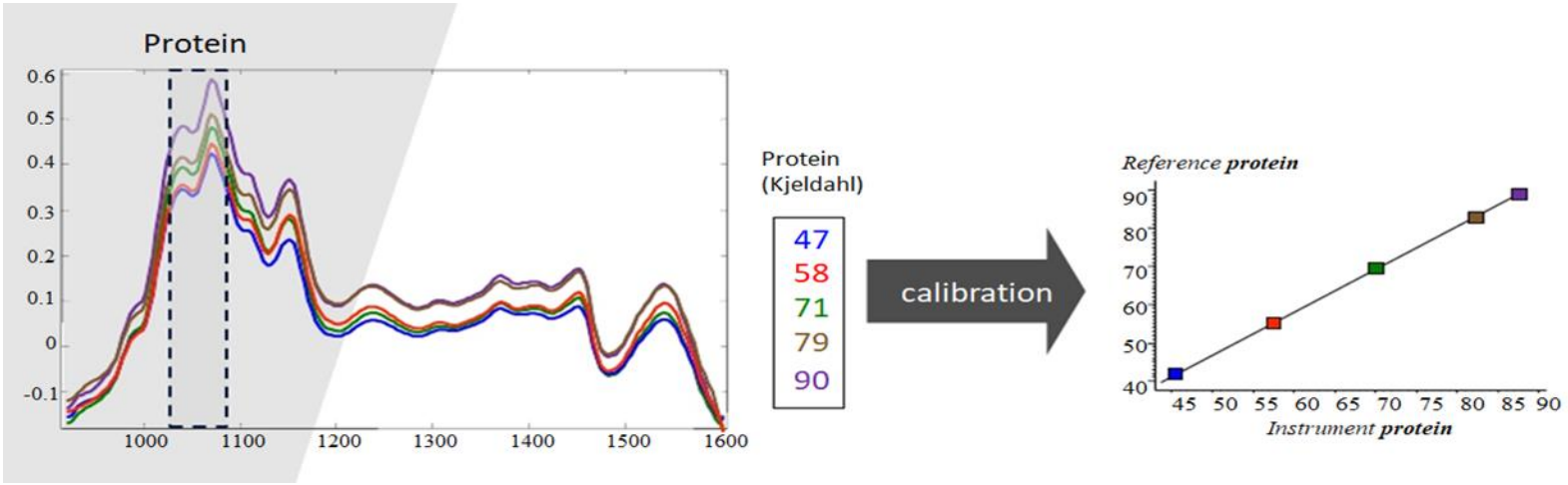
ANALYTICS BEYOND MEASURE

- 自1970年代以來，中紅外線(MIR)光譜分析儀，已成為例行檢測牛奶中主要成分 (脂肪、蛋白質、碳水化合物) 的快速方法

  Mid-Infrared (MIR) spectroscopy has been available since the 1970's as a rapid method for routine measurements of the main constituents in milk (fat, protein, carbohydrates)

- 傅立葉轉換式紅外光譜 (FTIR) 是獲取 MIR 光譜分析的最新方法

  Fourier Transform Infrared (FTIR) spectroscopy is the state-of-the-art method for acquiring MIR spectra analyzing



液態乳 MIR 光譜　Mid IR Spectra of Liquid Milk

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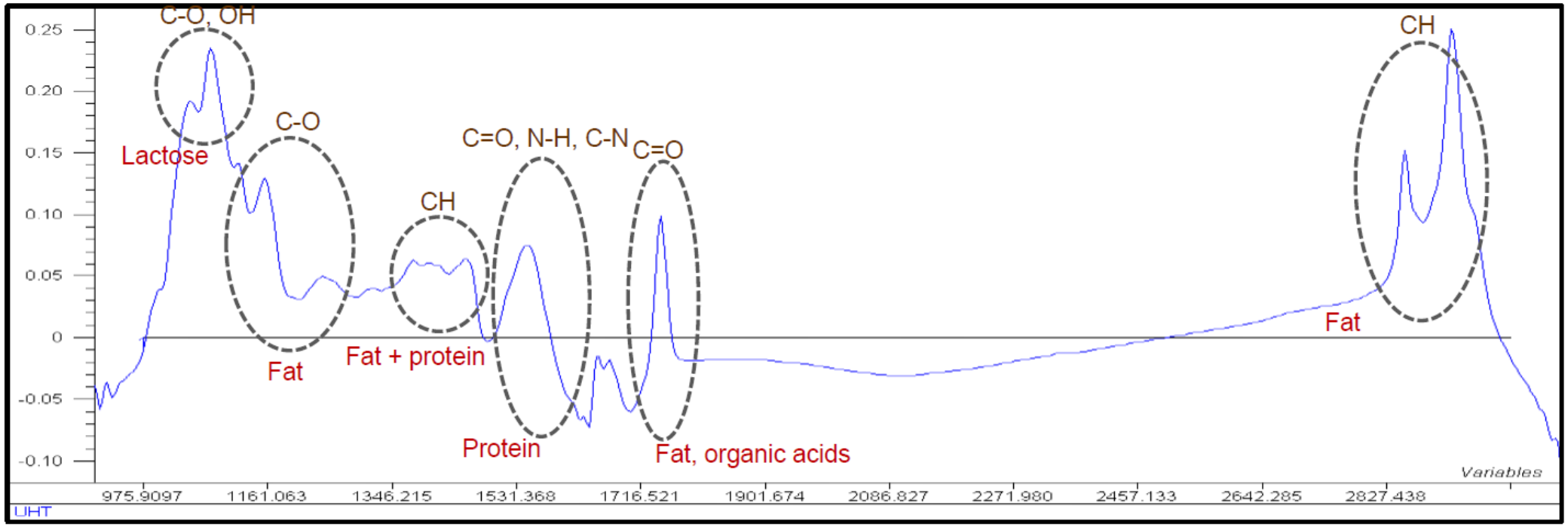
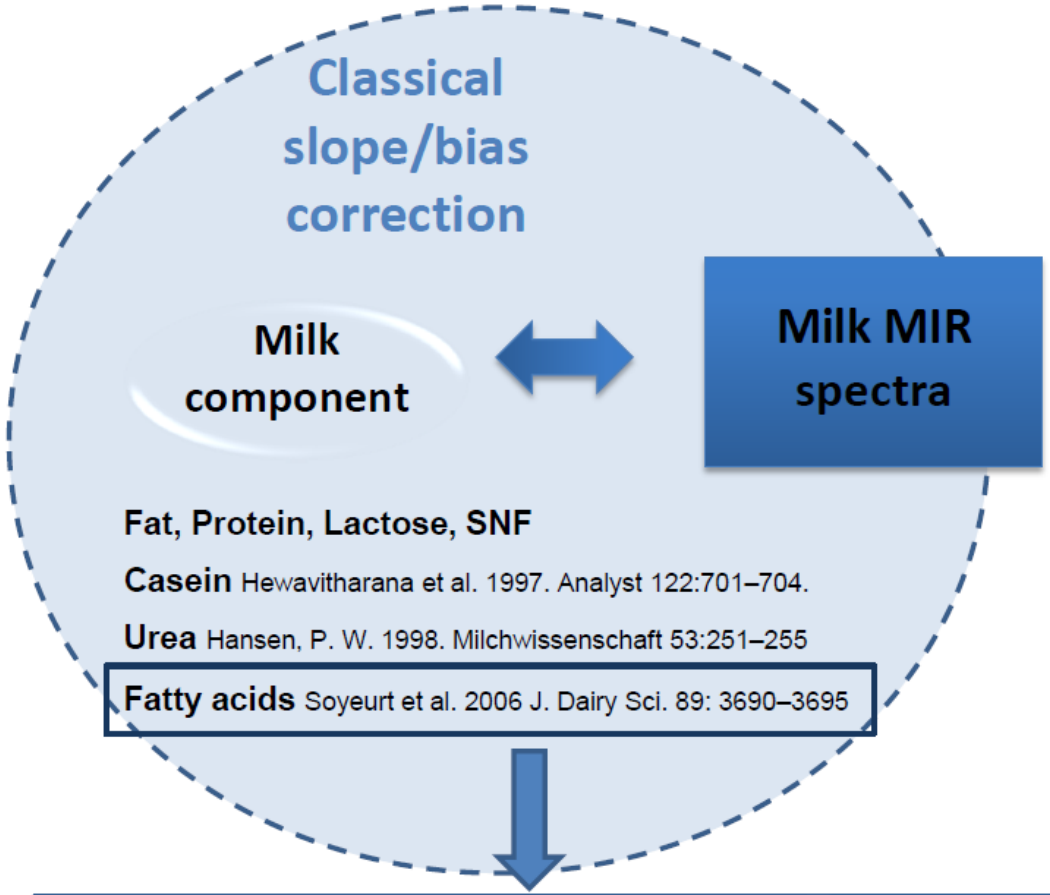


- **Position of the peaks → Qualitative analysis**
- **Intensity of the peaks → Quantitative analysis**

※ Frédéric Dehareng & Clément Grelet, ICAR 2019 Conference, Technical session 7: Challenges in Creating Additional Value from Milk Analysis

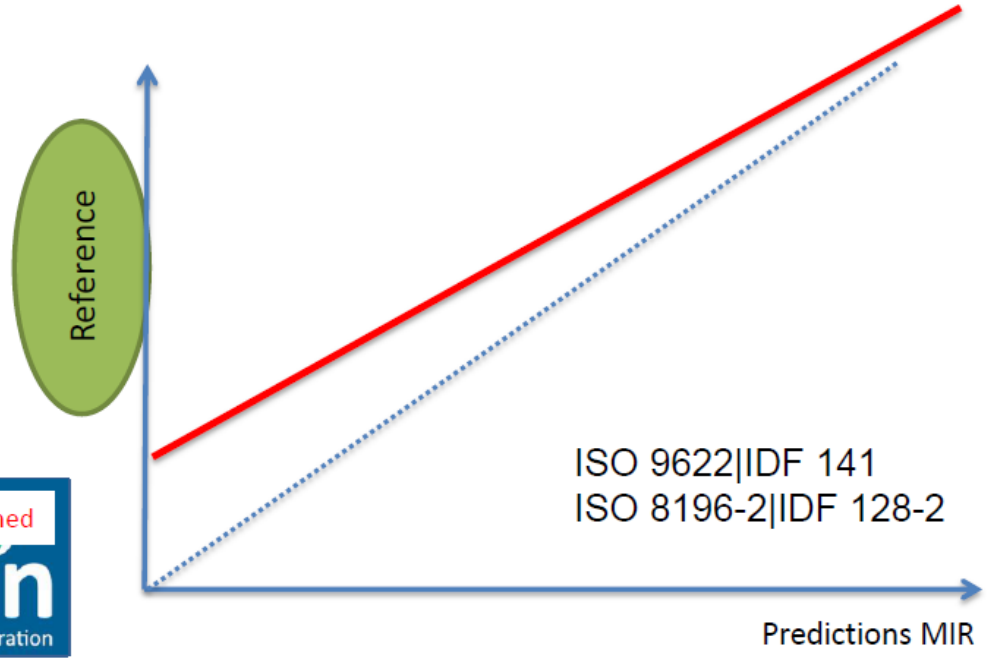
ANALYTICS BEYOND MEASURE



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



Wallonie recherche CRA-W
Walloon Agricultural Research Centre
To address today's questions and to prepare tomorrow's challenges
www.cra.wallonie.be

※ Frédéric Dehareng & Clément Grelet, ICAR 2019 Conference, Technical session 7: Challenges in Creating Additional Value from Milk Analysis

ANALYTICS BEYOND MEASURE

國際乳業聯盟(IDF) 公告

Bulletin-International Dairy Federation, IDF

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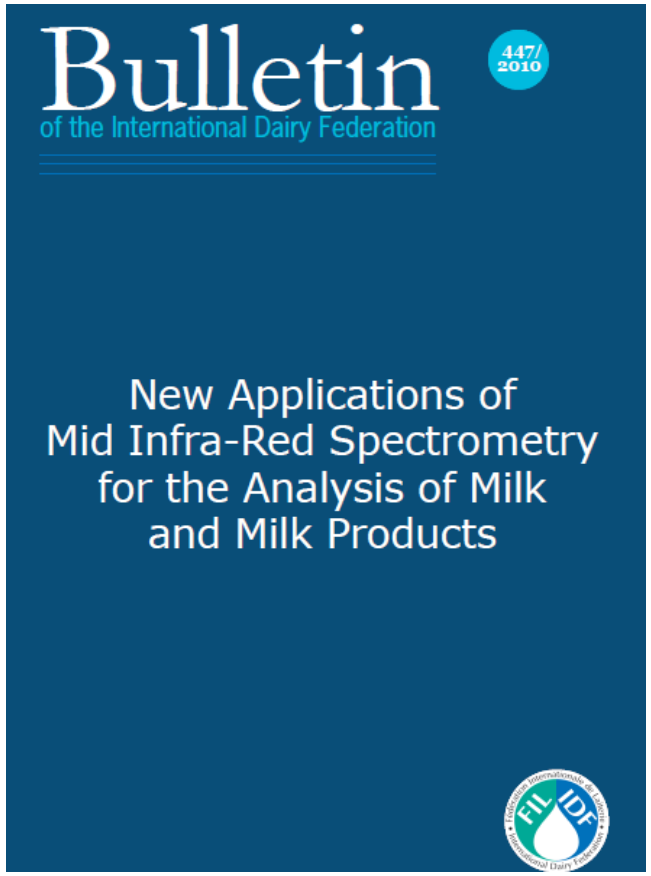
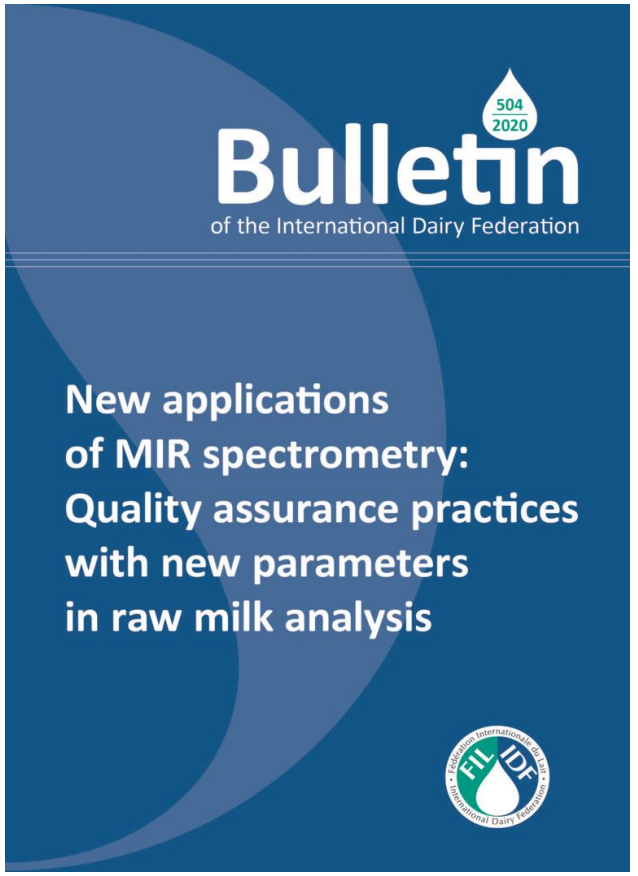


IDF 447 Published 2010



IDF 504 Published 2020

FTIR 可檢測項目

A Broader Range of Robust Calibrations

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ANALYTICS BEYOND MEASURE

Mass Balance/Standardization/Payment

Fat,
Protein (true & crude),
Casein
Lactose,
Total Solids
Solids non Fat
Urea
Ketosis Screening (BHB and Acetone)
Free Fatty Acids
Fatty Acids Profiling

End Product Control

Low Lactose
Glucose
Galactose

Quality Components

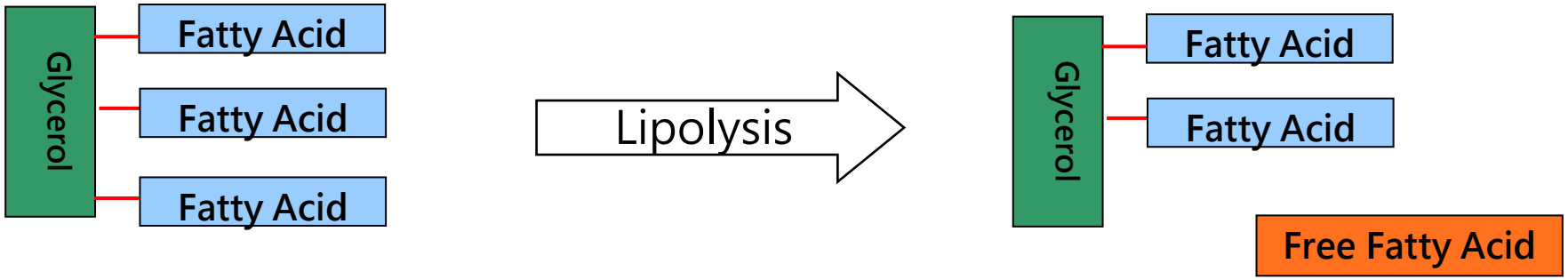
pH
Total Acids
Citric acids
Density
Fatty Acids Profiling (SFA, MUFA, PUFA, SCFA, MCFA, LCFA, De novo FA, Mixed FA, Preformed FA, Trans FA, C14, C16, C18, C18:1.....etc.)
Free Fatty Acids
Freezing Point Depression

Milk Parameters

20+ standard milk components
All included with the FTIR

+ ASM Module (Abnormal Milk Screening, Adulterant Screening), Good Products (Melamine.....etc.)

游離脂肪酸 Free Fatty Acids



影響游離脂肪酸含量之可能因素 Free Fatty Acids are Influenced by :

- The mechanic treatment of the milk (milking machine design) Installation and operation
- The general hygiene/bacterial status of the milk
- Physiological status of the cow (stage of lactation, diseases, composition of fodder, seasonality etc.
- Milk handling on the farm and in the factory

ANALYTICS BEYOND MEASURE

游離脂肪酸含量對乳品品質之影響
FFA in Dairy Products and Suppliers Milk

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ANALYTICS BEYOND MEASURE

游離脂肪酸太高導致之問題 Problems caused by higher FFA levels:

- Rancidity in high-fat dairy products i.e. butter, cream
  (FFA has a rancid and soapy taste and smell)
- High levels of FFA influences the shelf life
  (i.e. reduced shelf life of milk powder products)
- Affects/reduce cheese yield
  (FFA is not included in the coagulum)
- Adversely affect the flavour and quality of milk dairy products

檢測游離脂肪酸之應用 Why test suppliers milk:

- Farmers, who deliver milk with high FFA, can be helped to correct the problem
- Raw milk with high FFA levels cannot be “repaired”, the damage is done, and the enzymatic process continues
- by well designed and monitored milking machines and bulk tanks
- by consistent balanced cow diets
- by proper drying-off procedures

以FTIR技術檢測游離脂肪酸之優點
FFA Calibration and Benefits with FTIR Tech.

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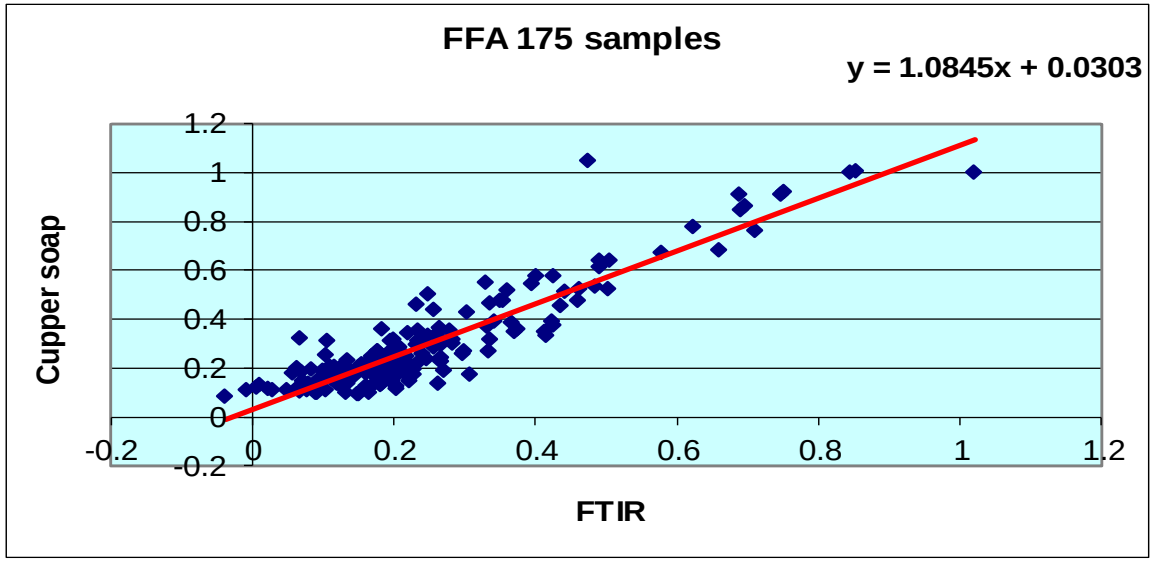
FTIR Milk Analyzers can be an excellent method for FFA-screening by:

- Reducing analysis costs
- Giving high test frequency and thereby a much better overall picture of FFA levels
- Saved time and costs if using BDI, FIA or copper soap method today
- Quick follow up tests to solve the problem if a high FFA result detected, ex. Errors in milking equipment, unbalanced cow diets
- Improve the shelf life and quality of dairy products



- Normal concentration of FFA in raw milk "ab farm" is < 0.7 mmol FFA / 100g Fat
- Sound cow's milk contains ~0.5 mmol FFA/100 g Fat
- FFA > 1 mmol FFA / 100g Fat is considered abnormally high / 'defective'

檢測游離脂肪酸之國家
Countries Measuring Free Fatty Acid

FFA is today measured in:

- The Netherlands
- Norway
- France
- Belgium
- Japan
- Italy
- UK
- Canada
- USA
- Denmark
- Spain
- Brazil
- The Czech Republic
- Taiwan

FFA included in Payment Scheme

冰點檢測 Freezing Point Depression (FPD)

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Has someone added water to the milk?

Freezing point of authentic ab farm milk is quite constant and lower than **-0.520℃ degrees Celsius (ca -0.538 "Hortvet")**, and has therefore for a long time been used as a milk quality parameter.

In 1989, J. Koops et al proved that combining data from **Infra Red analyzer** with that of **electrical conductivity** can give a good prediction of the actual freezing point of a milk sample.

FTIR technology is the third generation IR analyzers used for this purpose, with which we can now provide an **accuracy of < 4 m°C**, which ensures an extremely **efficient screening** and very **low cost** per positive sample.

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冰點檢測之應用

Application of Freezing Point Depression (FPD)

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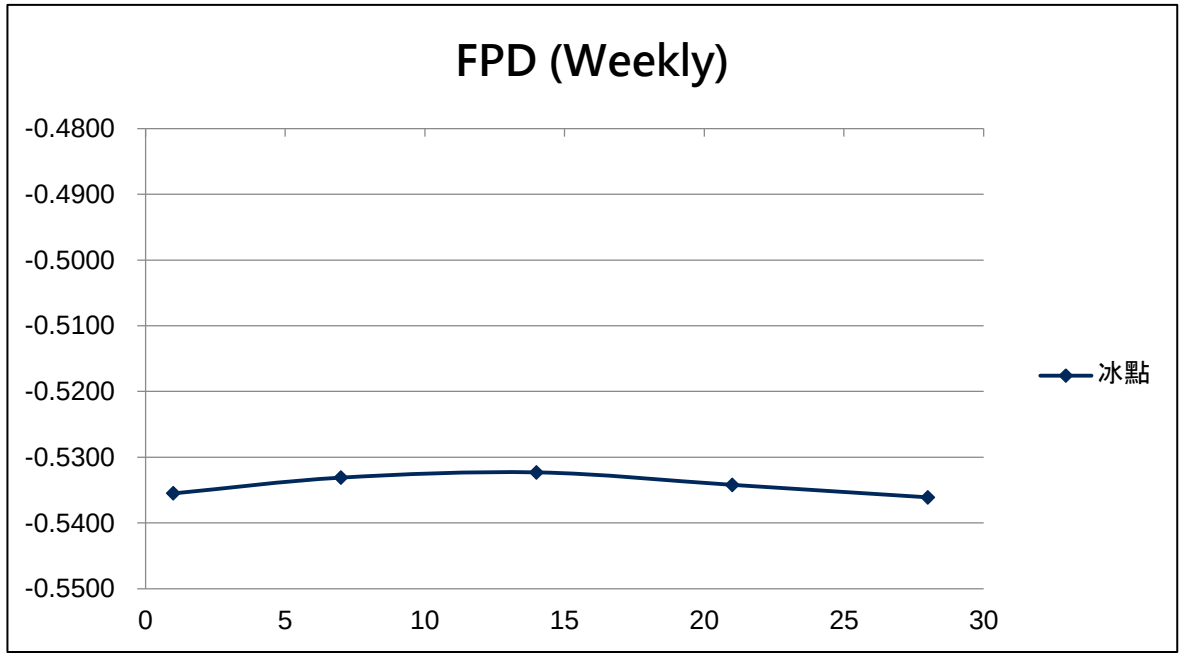
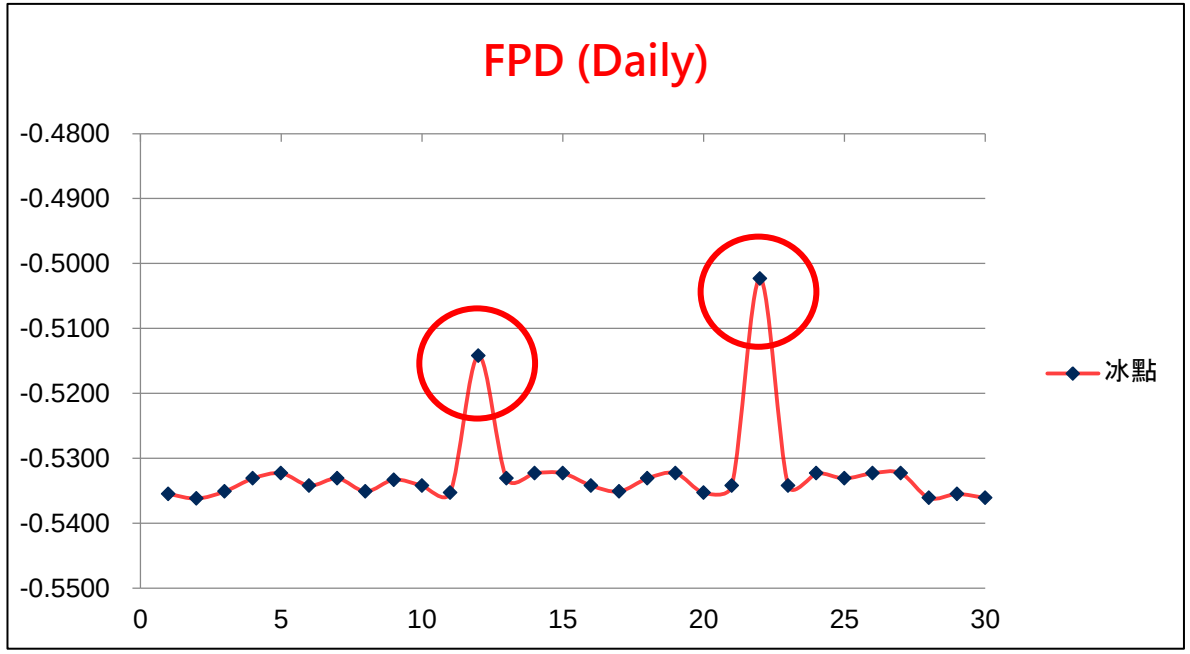
ANALYTICS BEYOND MEASURE





牛乳中摻假檢測 Milk Adulteration

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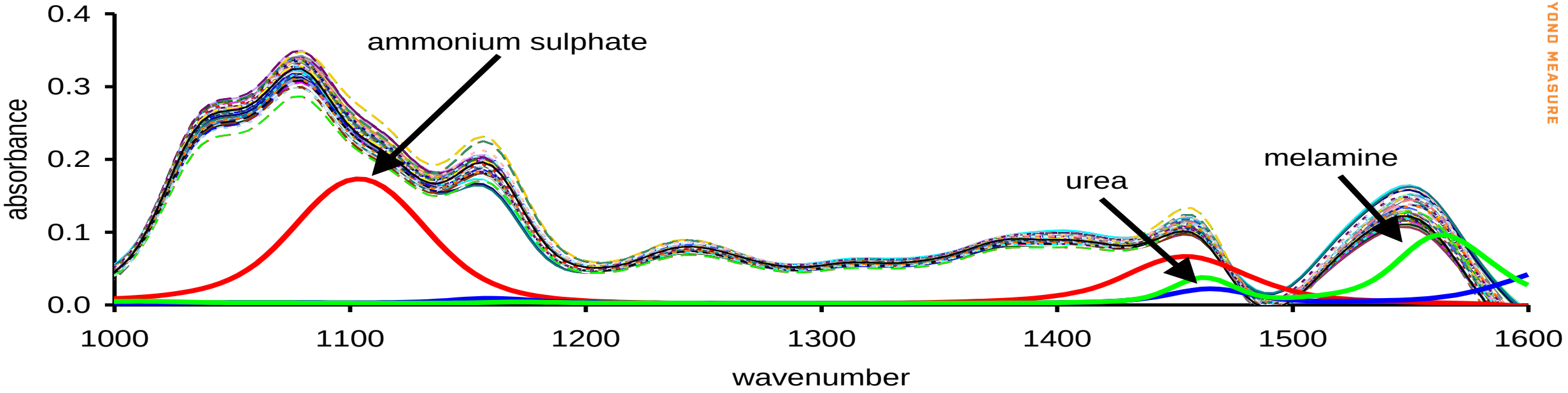
ANALYTICS BEYOND MEASURE



牛乳中摻假檢測 Milk Adulteration

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- FTIR spectra from natural raw milk samples is a unique finger print of normal milk
- Most accurate and widest scope of adulteration screening capabilities in the industry
- Check for deliberate or accidental adulteration while performing normal quality control tests.

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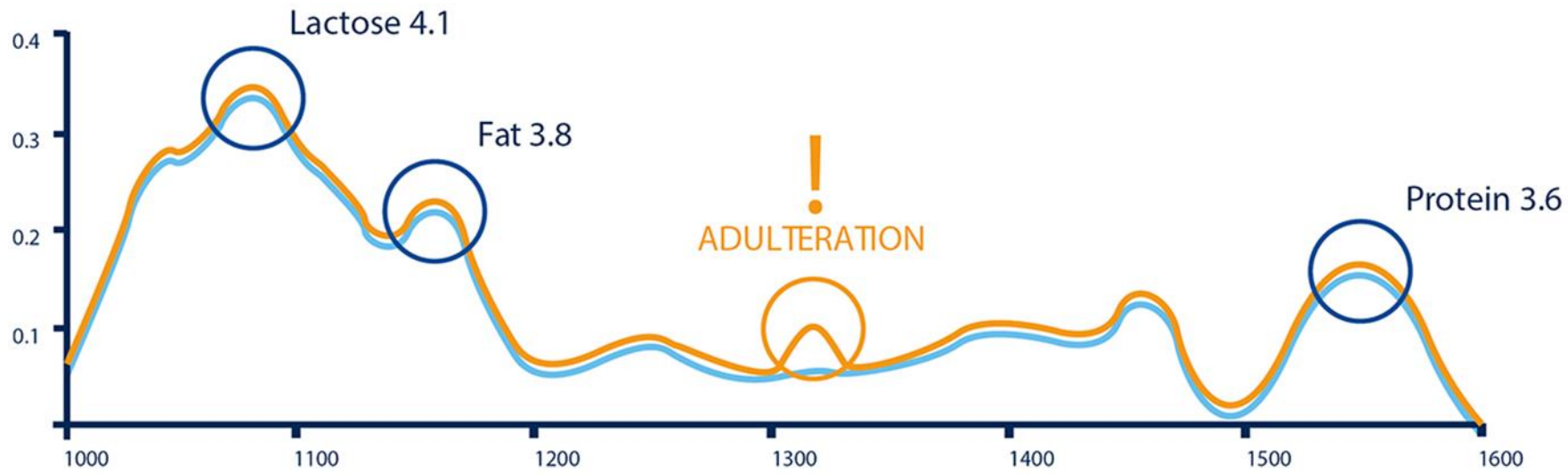
Raw milk sample



摻假物質篩檢

What Can Be Screened Against with The Untargeted Model ?

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- The table shows the Limit of Detection for the adulterants which could be detected as abnormal by the model.
- The LoD´s correspond to a threshold of 3.

Adulterant	LoD	LoD Adulterant	LoD	LoD Adulterant	LoD
Allantoin	300 ppm	Cyanuric acid	170 ppm	Sodium bicarbonate	400 ppm
Amidourea	500 ppm	Cyromazine	300 ppm	Sodium hydroxide	0.06 %
Aminotriazine	800 ppm	Dicyandiamide	300 ppm	Sodium nitrite	200 ppm
Ammonium nitrate	200 ppm	Formaldehyde	400 ppm	Thiourea	500 ppm
Ammonium sulphate	300 ppm	Hydroxyproline	900 ppm	Triuret	800 ppm
Biuret	600 ppm	Maltodextrine	1100 ppm	3-aminotriazole	1100 ppm
CIP agent	1%	Melamine	300 ppm	4-aminotriazole	1400 ppm

發生乳中摻假之可能原因 Screening for Milk Adulteration

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非故意的 Unintentional deviations

Caused by accidents or failures.

- Agent in the cows feed that is transported on to the milk
- Cleaning agent
- Water

刻意的 Intentional deviations

Driven by economic gains:

- amount (weight and/or volume)
- milk fat content
- milk protein content
- dry matter content
- the total bacterial counts and somatic cell counts

ANALYTICS BEYOND MEASURE

快速完成摻假篩檢 Security Screening in 30 Seconds

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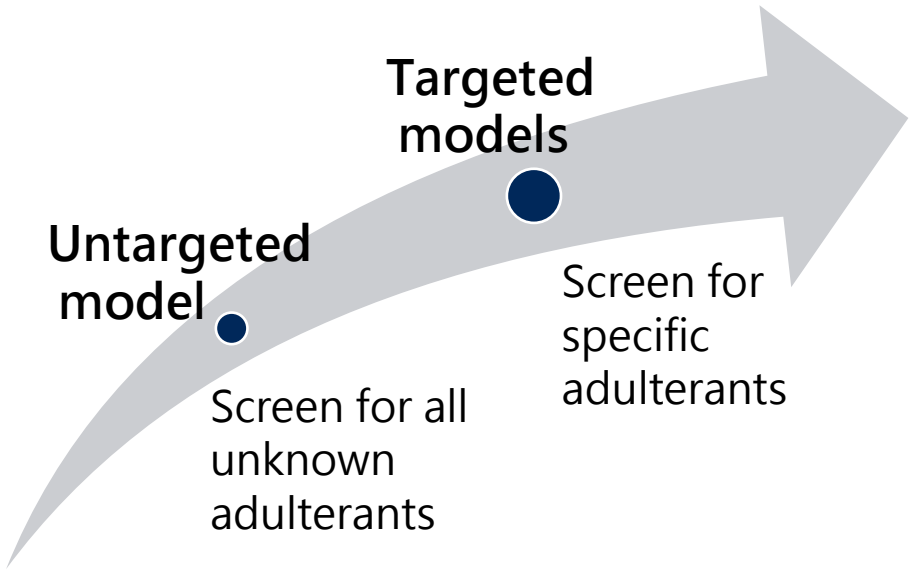

Rapid inexpensive screening of your milk

脂肪酸組成檢測 Fatty Acids Profiling

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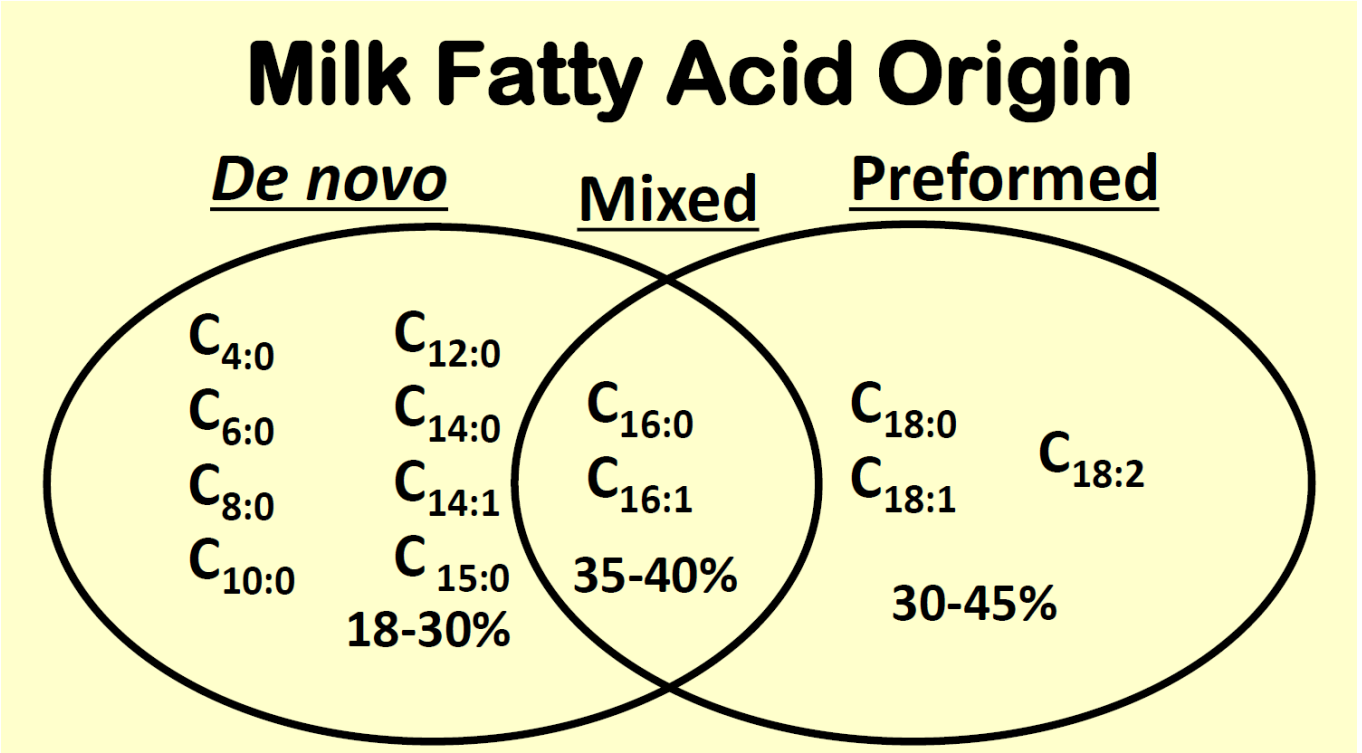
ANALYTICS BEYOND MEASURE

Fatty Acids Profiling

- SFA, MUFA, PUFA, Trans FA,
- SCFA, MCFA, LCFA,
- **De novo FA, Mixed FA, Preformed FA**
- C4, C6, C8, C10, C12, C14, C15, C16, C17, C18, C18:1.....etc.



※ D. M. Barbano, etc., Analytica 2019, Rome, Italy. Cows health and feed efficiency improvement through milk analyses and optimized health and feed management.

脂肪酸來源檢測 Fatty Acids Origin

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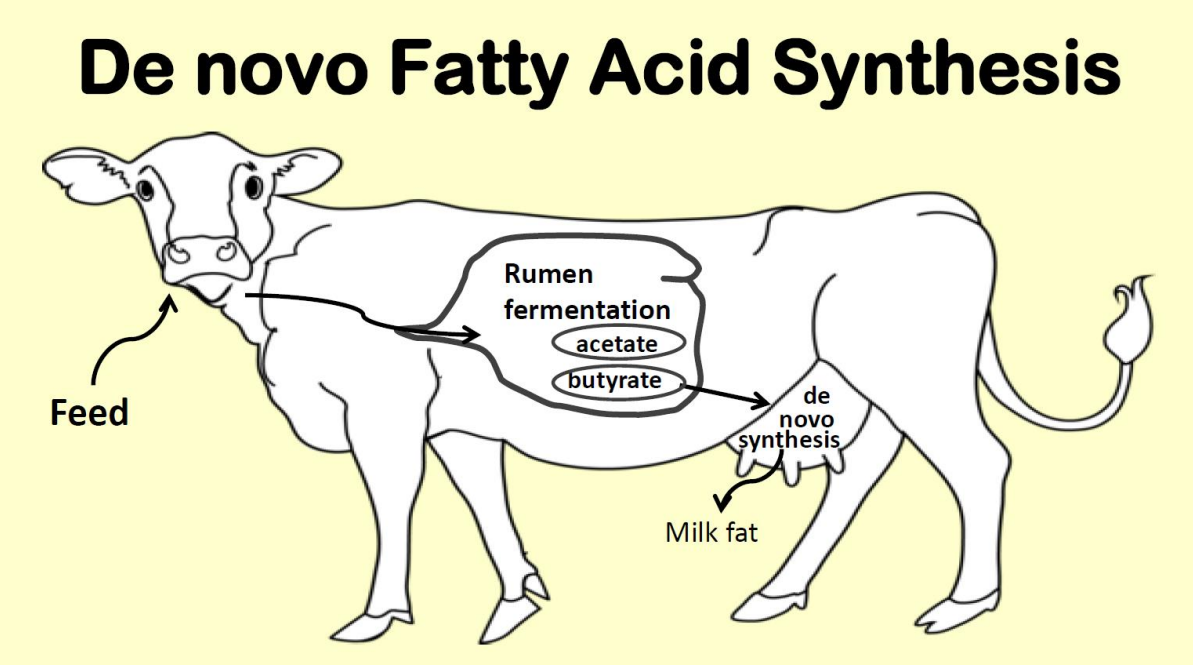
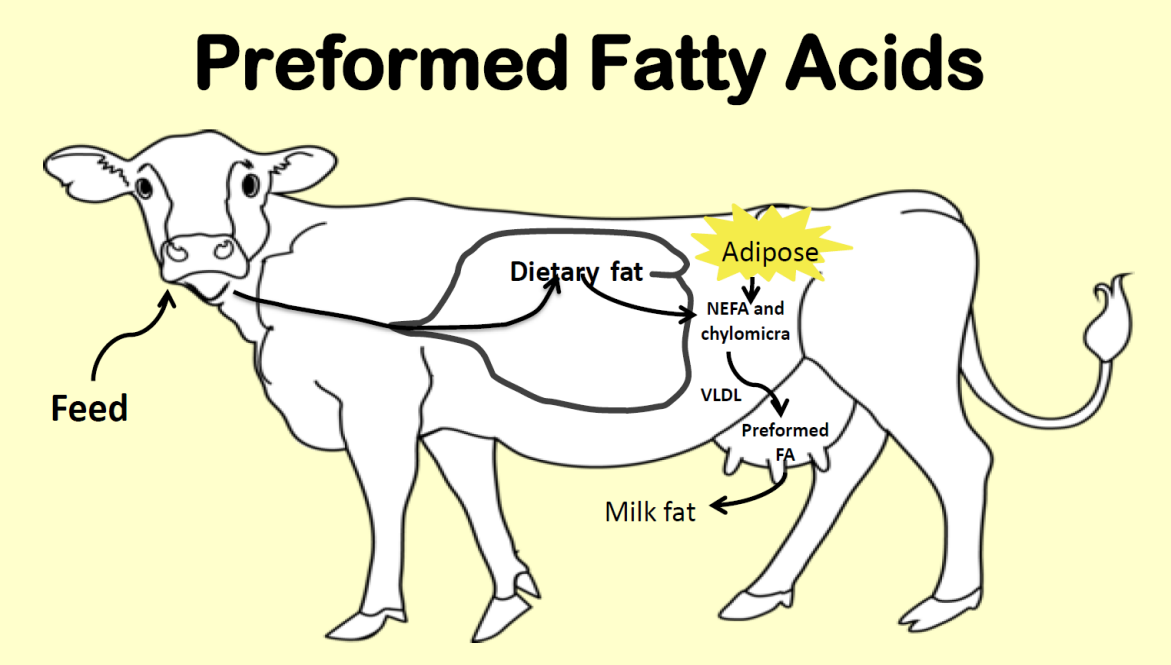
Cows health and feed efficiency improvement through milk analyses and optimized health and feed management





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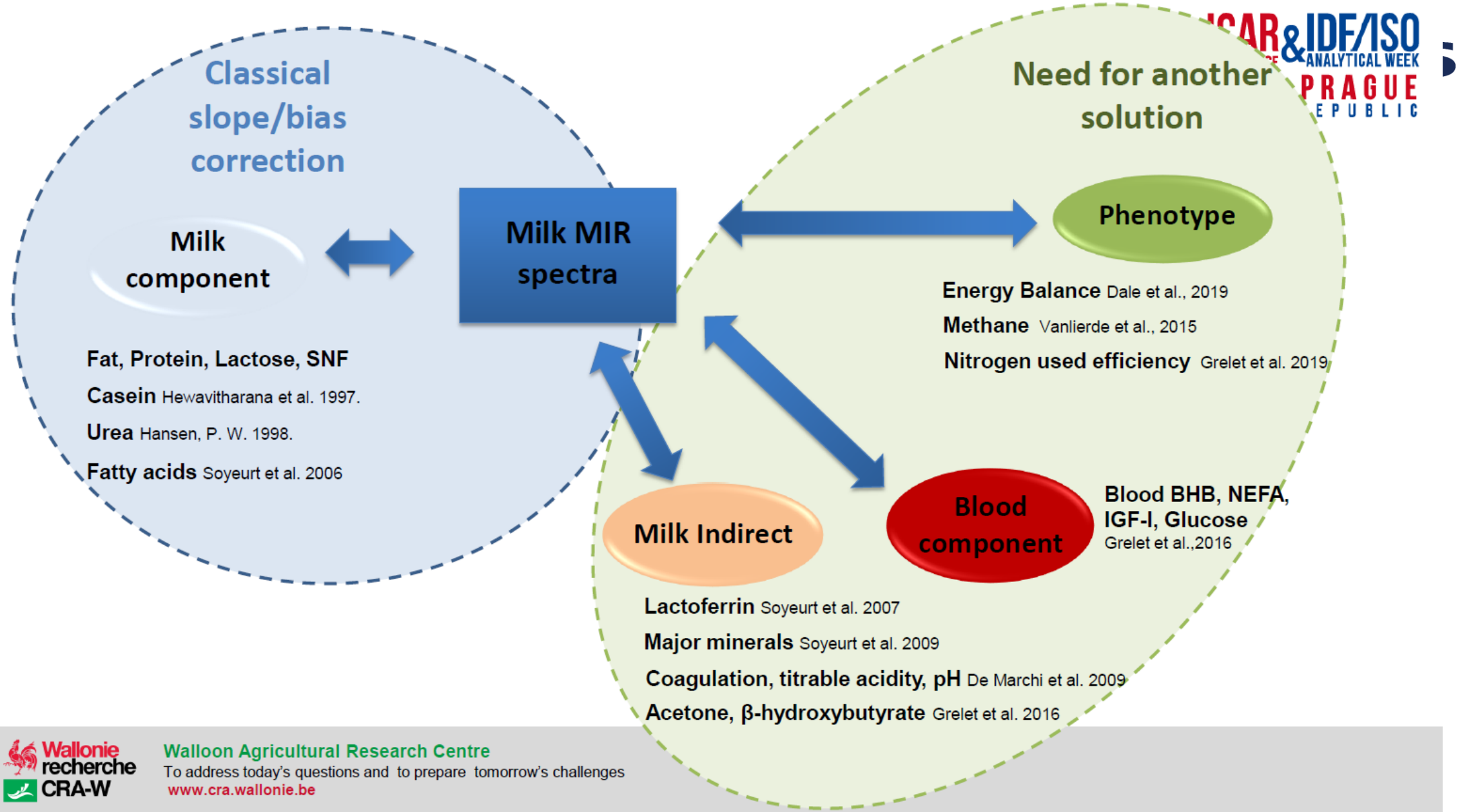

※ Frédéric Dehareng & Clément Grelet, ICAR 2019 Conference, Technical session 7: Challenges in Creating Additional Value from Milk Analysis

ICAR 2019 會議 – FTIR 新的檢測模式

ICAR 2019 Conference – Focus on New Tools

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Hall Panorama

8:30-10:30	**Technical Session 7** **Challenges in Creating Additional Value from Milk Analysis** **Chairpersons: Silvia Orlandini and Jere High**
8:30-8:50	S07(T)-OP-1 **Additional value of cell differentiation in the course of DHI testing** Folkert Onken
8:50-9:10	S07(T)-OP-2 **Pregnancy testing in dairy cows using a PAG test in milk samples: Different thresholds for different stages of the pregnancy** Daniel M. Lefebvre
9:10-9:30	S07(T)-OP-3 **New quality assurance challenges with recent mid-infrared models** Frédéric Dehareng
9:30-9:50	S07(T)-OP-4 **Implementation of a routine Fourier-transform infrared procedure for fatty acid analysis in milk** Daniel M. Lefebvre
9:50-10:10	S07(T)-OP-5 **Routine infrared phosphorous determination in ex-farm milk giving better insight in the phosphorous cycle on dairy farms** Harrie van den Bijgaart
10:10-10:30	Question and Discussion

ICAR 2019 CONGRESS

ANALYTICS BEYOND MEASURE

ICAR 2019 會議 – FTIR 新的檢測模式

ICAR 2019 Conference – Focus on New Tools

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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-04 **The use of fatty acid profiles from milk recording samples to predict body weight change of dairy cows in early lactation in commercial dairy farms**
Franziska Dettmann

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

S07(T)-PP-07 **From new milk-testing parameters to new DHI services – The view of an instrument manufacturer**
Daniel Schwarz

FTIR 檢測模式-品質確保的新挑戰

New Quality Assurance Challenges with FTIR Models

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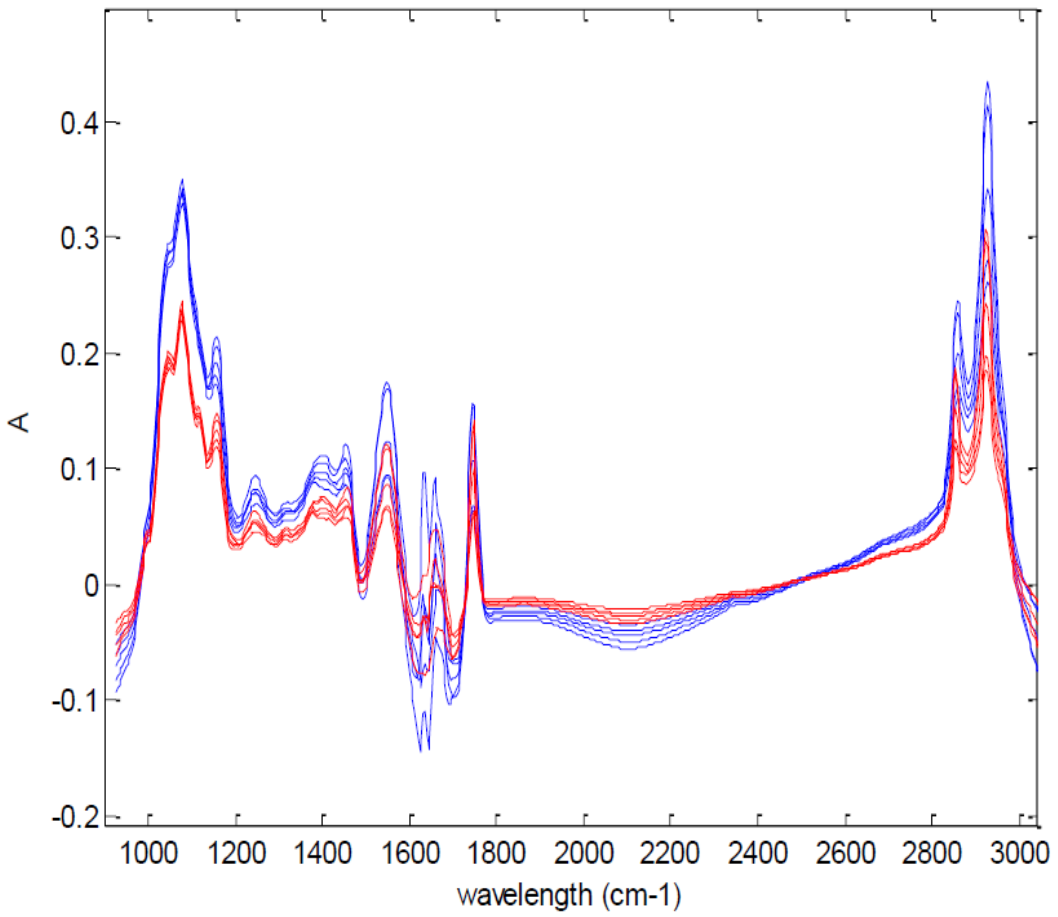

同一個樣品，不同的光譜資料
Spectra of same sample could differ

→

不同的測值
Different results

不同的儀器設備
Different Instrument

- 廠牌不同 Different brands apparatus
- 同廠牌 不同機型 Same brand but different model
- 同廠牌 相同機型 Same brand and same model

同一台儀器設備
Same Instrument

- 實驗室 溫度/濕度的影響 Temperature / Humidity in the lab
- 設備維修 Piece replacement
- 設備定期保養 Maintenance operation
- 使用方式/零組件磨損 Use / Wear

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FTIR 檢測模式-品質確保的新挑戰
New Quality Assurance Challenges with FTIR Models

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解決方案
Test Solution

光譜標準化
Standardization of The MIR Spectra

J. Dairy Sci. 98:2150–2160
http://dx.doi.org/10.3168/jds.2014-8764
© American Dairy Science Association®, 2015.

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
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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]
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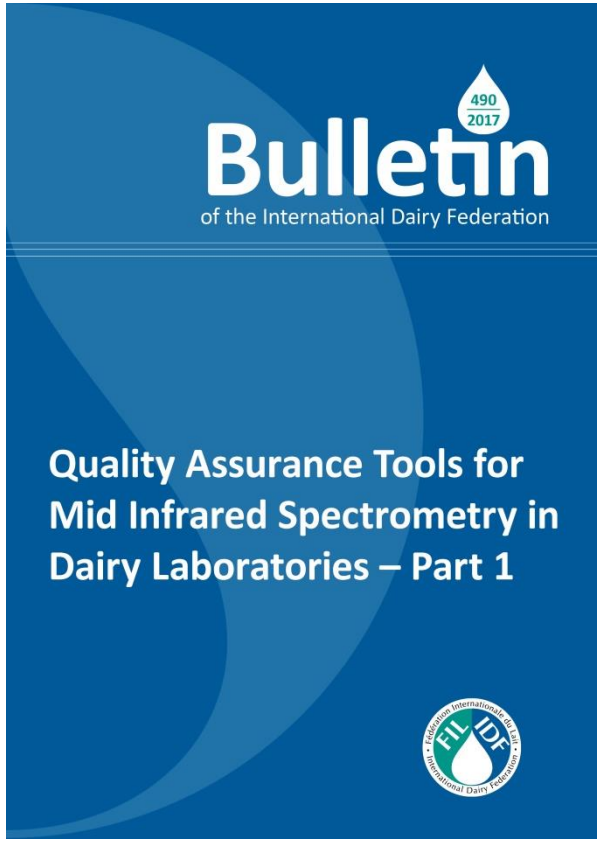
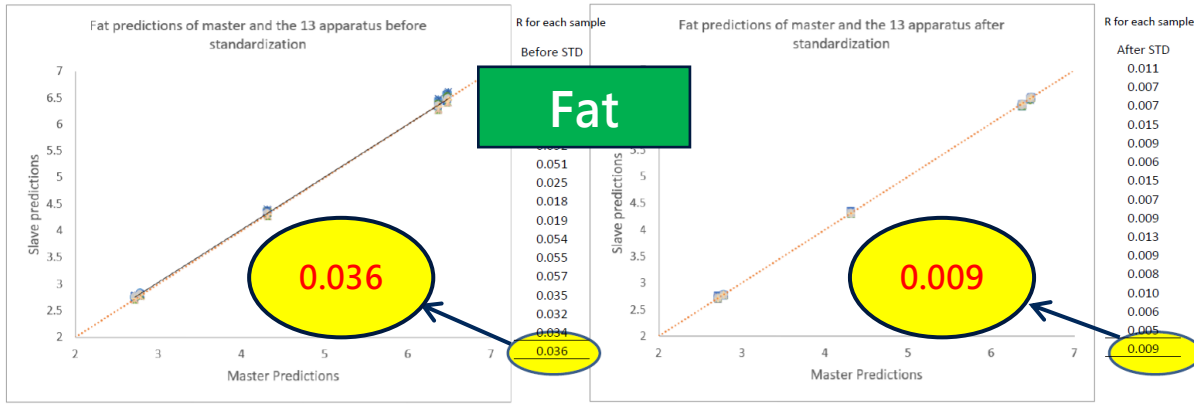


IDF 490 Published 2017

5 個生乳樣品(Raw milk) - Fat, BHB, Phosphorous, Nitrogen use efficiency
6 台 MilkoScan FT+ (France) ＆ 7 台 MilkoScan FT 6000 (Switzerland)

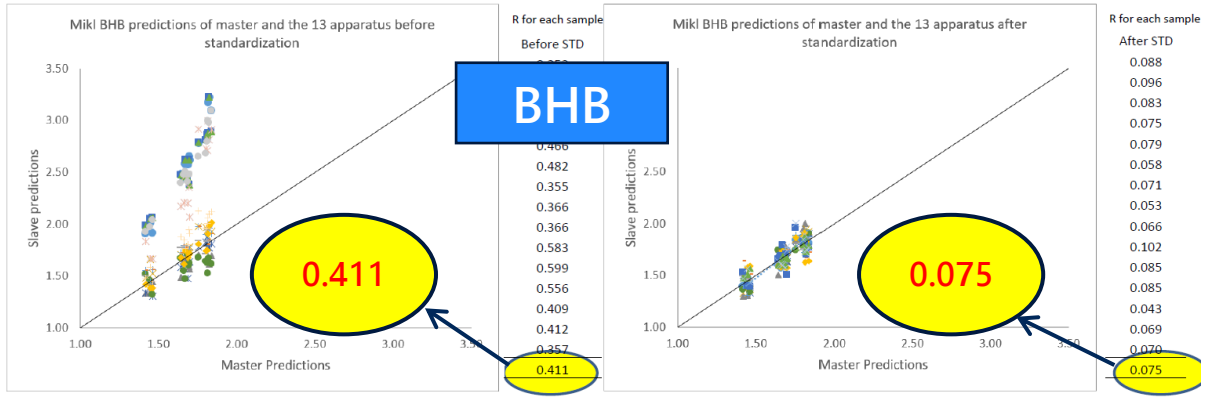
Wallonie agriculture SPW — EMR — FOSS

Fat



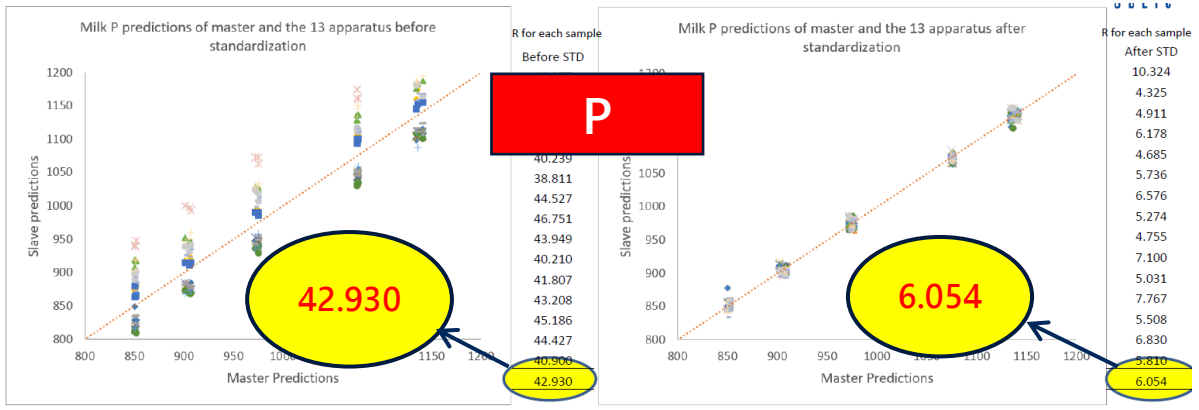
Total Fat	Unit	Calibration							Cross validation with 4 subsets		
		# terms	#N	#WL	Mean	SD	SEC	R^2c	SECV	R^2cv	RPDcv
	g/100ml	7	1792	212	3.912	0.971	0.007	1.00	0.007	1.00	132.99

BHB



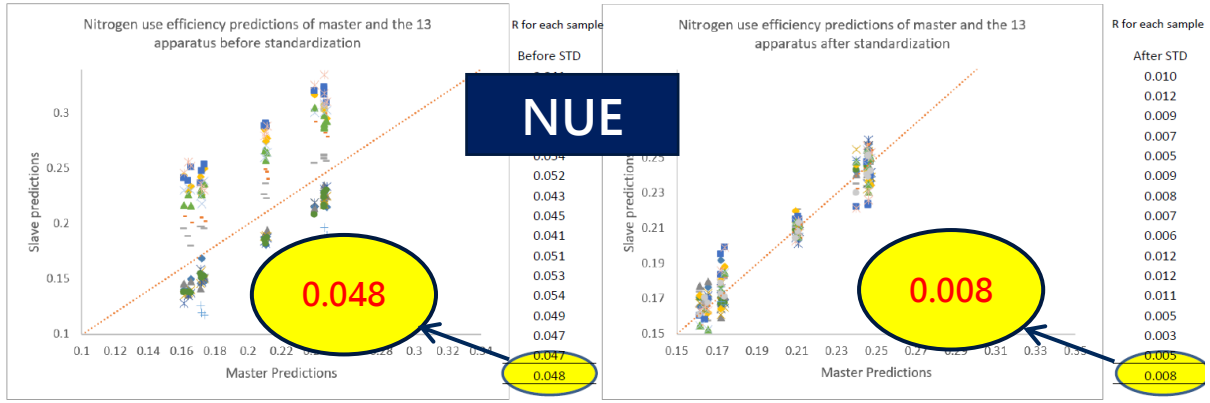
BHB milk	Unit	Calibration								Validation		
		# terms	#N	#WL	Mean	SD	RMSEc	R^2c	RPDc	#N	Mean	RMSEv
	Log(mmol/L)	9	419	212	0.225	0.171	0.086	0.75	1.97	124	0.156	0.065

P



P	Unit	Calibration									Cross validation with 4 subsets		
		# 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

NUE



NUE	N in milk/N Intake, daily ratio	Calibration								Cross-validation with 4 subset			External-cows-validation (25% cows out)		
		# 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

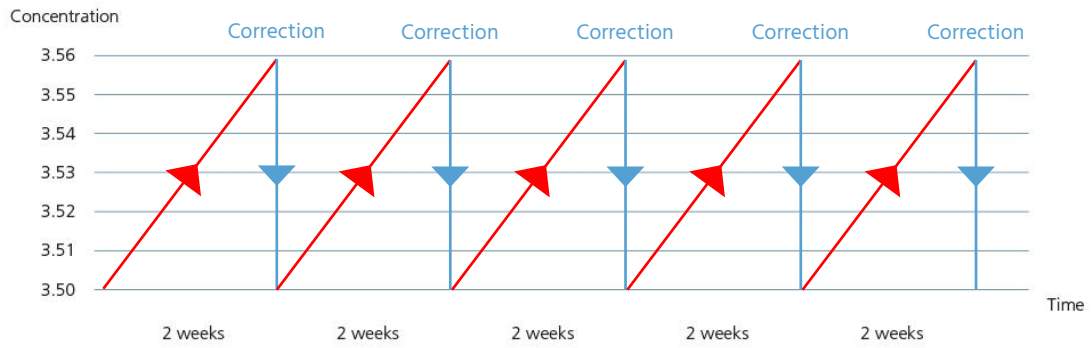
專利光譜標準化技術-預防儀器漂移
Always Standardised-No More Instrument Drift

FOSS

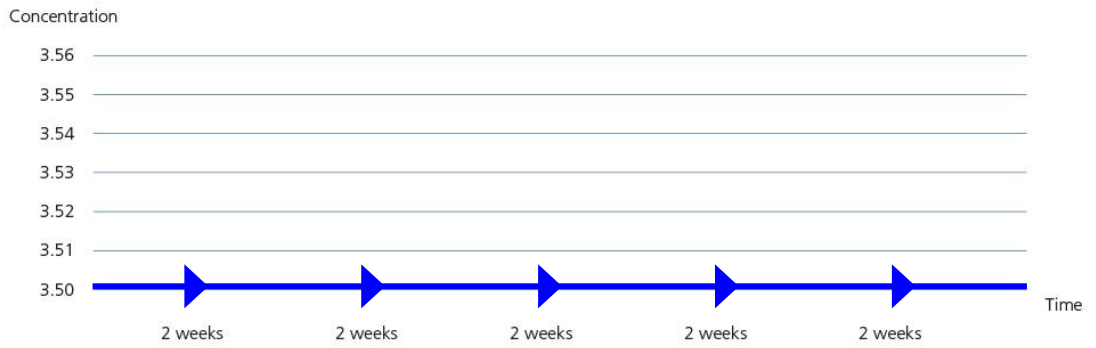
- MilkoScan™FT3 導入FOSS新取得專利之光譜自動標準化技術，此技術無需人工干預，亦不需使用FTIR標準液
- 隨著光譜自動標準化技術新專利的推出，儀器之間不再存在漂移和結果變化的情況
- MilkoScan FT3 每兩小時自動執行光譜標準化一次，消除儀器漂移，使檢測結果持續維持穩定的一致性
- 光譜自動標準化技術，不僅可以年復一年地實現檢測設備穩定的高性能，不再需要執行耗時的標準化檢查，大幅減少高成本的傳統分析校正，同時還提供了提高利潤和優化成本的機會，。

ANALYTICS BEYOND MEASURE

現行方式光譜標準化 Existing Standardised Method



MilkoScan™ FT3 自動光譜標準化 Automatic Standardized



MilkoScan™ FT3
The World's First
Intelligent Milk Analyser

檢測產品種類及項目 Full Coverage Robust Calibrations

FOSS

ANALYTICS BEYOND MEASURE

Milk
Fat, Protein, Casein, Lactose, Low Lactose, Glucose, Galactose, Total Solids, Solids Non Fat, Density, Citric Acid, Urea, Free Fatty Acids, Titratable Acidity and Freezing Point

Cream
Fat, Protein, Lactose, Total Solids and Solids Non Fat and Freezing Point

Whey
Fat, Protein, Lactose, Total Solids, Solids Non Fat and Titratable Acidity

Yoghurt & fermented application
Fat, Protein, Lactose, Total Solids, Solids Non Fat, Glucose, Fructose, Sucrose, Total Sugars and Lactic Acid

Dessert & ice cream application
Fat, Protein, Lactose, Total Solids, Solids Non Fat, Glucose, Fructose, Sucrose, Total Sugars and Lactic Acid

Concentrated & fortified milk
(Conc. Milk, Baby Milk, Infant formula, infant formula powder, Evaporated Milk, Sweetened Condensed Milk)
Fat, Protein, Lactose, Total Solids and Solids Non Fat

Whey concentrates & permeate
(WPC, WPI and concentrated permeate)
Fat, Protein, Lactose, Total Solids, Solids Non Fat and Titratable Acidity

Other Applications

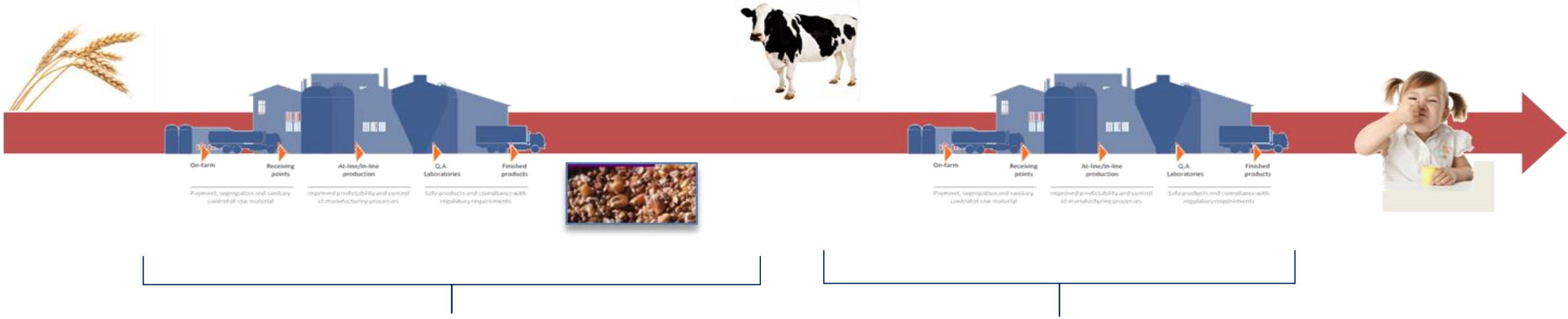
Juice, Soy Sauce.....

- MilkoScan FT3 允許對分析樣品的粘度進行自動調整，因此可對高固體含量的WPC進行可靠的分析

優良食品安全之願景 GoodProduct – A Strong Vision

GoodProduct™

Tools to secure an effective and safe supply chain



ANALYTICS BEYOND MEASURE

Taiwan Innotech Expo
台灣創新技術博覽會

FOSS

2020 台灣創新技術博覽會 2020 Taiwan Innotech Expo

2020 9. 24-26 台北世貿一館 Taipei world Trading Center

永續發展館 Sustainability Theme Zone

國際展區 10號展位 International Pavilion, Booth No. 10

ANALYTICS BEYOND MEASURE

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THANK YOU

ANALYTICS BEYOND MEASURE