

# 國際畜政聯盟認證乳質分析機種趨勢 ICAR Certified Milk Quality Analyzers



今日儀器公司 Today's Instruments Co., Ltd.  
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TLRI, Tainan, Taiwan, 12th September 2018



越南與台灣乳業發展論壇  
Dairy Industry Development -  
Vietnam and Taiwan  
2018/09/12



Dedicated Analytical Solutions
乳製品生產流程  
Production of Milk Products

## The Milky Way

Dedicated Analytical Solutions
牛乳之價值鏈  
The Milk Value Chain - Overview

**生乳計價 Milk Payment - value chain**  
**(CMT – Dairy – Consumer):**

- ▶ Composition (Fat, Protein, Solids)
- ▶ Health improving parameters  
*(e.g. Fatty Acids)*
- ▶ Hygienic quality *(total bacteria count)*
- ▶ Milk quality *(e.g. Fatty Acids, Freezing Point Depression)*
- ▶ Safety & quality  
*(e.g. Abnormal Spectrum Screening)*

CMT

↔

**牛群性能改良 Herd Improvement - value chain**  
**(CMT – DHIA – Farmer/Cow):**

- ▶ Productivity *(Fat, Protein, Yield)*
- ▶ Mastitis *(Somatic Cells, Microorganisms)*
- ▶ Feeding *(Fat, Protein, Urea, Fatty Acids)*
- ▶ Cattle diseases *(Acetone/BHB/ Ketosis)*
- ▶ Breeding *(all parameters, Yield)*
- ▶ Quality *(Free Fatty Acids)*

Dedicated Analytical Solutions
參考方法 (標準方法)  
International Reference Methods

**參考方法 International Reference Methods**

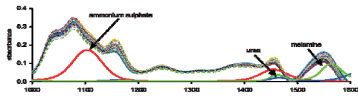
<b>Fat</b>	
Gravimetric method (Röse-Gottlieb)	ISO 1211   IDF 1 AOAC 905.02 (IDF-ISO-AOAC-Codex) AOAC 989.05 (IDF-ISO-AOAC)
Gravimetric method (modified Mojonnier)	
<b>Protein</b>	
Titrimetric method (Kjeldahl)	ISO 8968   IDF 20 AOAC 991.20 (IDF-ISO-AOAC) AOAC 991.21 AOAC 991.22 (IDF-ISO-AOAC) AOAC 991.23 (IDF-ISO-AOAC-Codex)
<b>Casein</b>	
Titrimetric method (Kjeldahl)	ISO 17997   IDF 29 AOAC 927.03 AOAC 998.05 AOAC 998.06 AOAC 998.07
<b>Lactose</b>	
HPLC method is foreseen to provide the reference to routine methods by ISO   IDF and its international standardisation is underway (ISO DIS 22662   IDF 198). In the meantime, standardised methods as referred to in "Part II, other methods" can be used.	
<b>Urea</b>	
Differential pH-method (Reference method)	ISO 14637   IDF 195
<b>Somatic cell count</b>	
Microscope method (Reference method)	ISO 13366-1   IDF 148-1

- ▶ 國際認可之標準檢驗方法  
International Standard Methods
- ▶ 檢測程序較複雜  
Detection procedure is more complicated
- ▶ 檢測所需的時間較長  
Long time required for testing
- ▶ 使用較多的人力  
Need more manpower
- ▶ 無法一次檢測大量的樣品  
Unable to detect large numbers of samples at the same time
- ▶ 準確度易受人為操作影響  
Accuracy is affected by operations

## 常規檢驗方法 International Routine Methods

### 常規檢驗方法 International Routine Methods

<b>Fat</b>	
Automated turbidimetric I	AOAC 969.16
Automated turbidimetric II	AOAC 973.22
<b>Protein</b>	
Automated dye-binding (Amido Black)	AOAC 975.17 (FIL-ISO-AOAC)
<b>Fat-protein-lactose</b>	
Mid infra red (MIR) spectrometric	ISO 9622   IDF 141 AOAC 972.16
<b>Urea</b>	
(taken into account with the underway revision of ISO 9622   IDF 141)	
<b>Somatic cell count</b>	
Electronic particle counter (Coulter Counter)	International standards withdrawn
Fluoro-opto-electronic methods	ISO 13366-2   IDF 148-2 AOAC 978.26

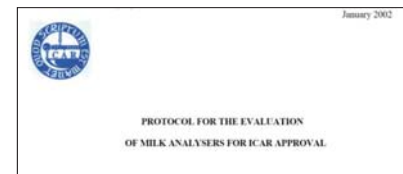


- ▶ 國際認可之常規檢驗方法  
International Routine Methods
- ▶ 檢測程序較簡單  
Simple detection procedure
- ▶ 檢測所需的時間較短  
Short time required for testing
- ▶ 使用較少的人力  
Use less manpower
- ▶ 可一次檢測大量的樣品  
Easy to test large numbers of samples at the same time
- ▶ 準確度不易受人為操作影響  
Accuracy is less affected by operations

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## ICAR 認證乳質分析設備 Milk Analyzers and ICAR Certification



- ▶ ICAR 乳質分析設備評估規範  
ICAR offers the possibility to certify milk analyzers in accordance to the ICAR “**Protocol for evaluation of milk analyzers**” developed and endorsed by the ICAR Milk Analysis Sub-Committee.
- ▶ 統一認證，國際認可  
It is an international harmonized protocol that serves the interest of milk recording world-wide.
- ▶ ICAR 認證遵循 **ISO 8196-3 / IDF 128-3**  
標準方法  
The ICAR certification will follow the ISO 8196-3 IDF 128-3 with particular attention for the DHI analysis.

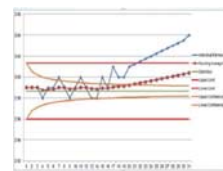
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## ICAR 認證乳質分析設備-技術評估項目 Course of Operation of Technical Evaluation

### ▶ 技術評估項目 Course of Operation of Technical Evaluation

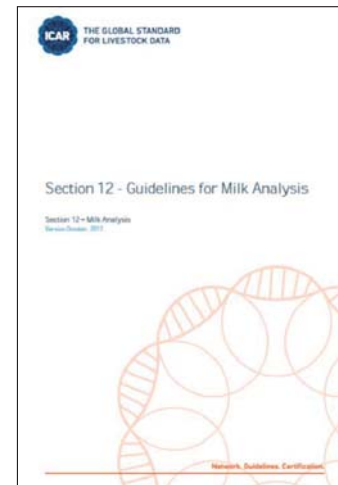
- ▶ 最低必要評估內容 Minimum necessary assessments
  - ▶ Assessment of preliminary instrumental fittings
    - ▶ Daily precision
    - ▶ Carry-over effect
    - ▶ Linearity
    - ▶ Measurement limits, Lower / Upper limit
  - ▶ 整體準確性評估 Evaluation of the overall accuracy
    - ▶ Assessment repeatability, accuracy of the mean, accuracy, exactness of calibration
  - ▶ 其他資訊調查 Additional informative investigations
    - ▶ Ruggedness (Effect of major milk components interaction, biochemical change in components, preservatives added, milk intake temperature, storage conditions)
  - ▶ 實用的方便性 Practical conveniences
    - ▶ Speed, Robustness, Monitoring facilities & service, Validation of precision conditions



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## ICAR 乳質分析指南 ICAR Guidelines for Milk Analysis



- ▶ 牛、山羊和母羊乳中 **脂肪、蛋白質、乳糖、尿素及體細胞數** 測定方法指南  
The guidelines concern methods for **Fat, Protein, Lactose, Urea & Somatic Cells** determinations in Cow, Goats and Ewes milk
- ▶ 參考方法 Reference Methods
  - ▶ 用以校正儀器(常規檢驗)的方法  
The methods used to calibrate the instrumental (routine) methods.
  - ▶ 須為國際認可的標準方法(如ISO, IDF, AOAC等)  
Should be internationally standardized methods (i.e. ISO, IDF, AOAC methods)
- ▶ 常規檢驗(儀器)方法 Routine (Instrumental) Methods
  - ▶ 須為標準方法或經由國際具公信力組織(如ICAR)認可  
Routine methods should be either standard methods, or methods approved at the international level by ICAR

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## ICAR 乳質分析指南 ICAR Guidelines for Milk Analysis

### ▶ DHI實驗室分析品質控制 Quality control for DHI laboratories

Table 1. Components of a quality control and recommended frequencies.

Control	Frequencies	Mode
<b>Reference methods</b>		
- External control	Quarterly	IPS
- Internal control	Weekly (for each check of the mean bias)	CRMs, SRMs, IRMs
<b>Routine methods</b>		
- External control	Quarterly	IPS/IEC
- Internal control	(see 7.2)	IRMs

IPS: Interlaboratory Proficiency Study.  
CRMs: Certified Reference Materials.  
IEC: Individual External Control.  
SRMs: Secondary Reference Materials.  
IRMs: In-house Reference Materials.

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## ICAR 乳質分析指南 ICAR Guidelines for Milk Analysis

### ▶ DHI實驗室常規方法的內部控制 Internal control on routine methods for DHI laboratories

Table 2. Frequencies and limits for checking routine methods.

Checks	Frequencies	F P L	Limits	SCC
<b>Instrumental fittings</b>				
Homogenization	Monthly	$\leq 0.05$ % units or $\leq 1.43$ % relative	(a)	None
Carry-over	Monthly	$\leq 1$ %	(a)	( $\leq 2$ %)
Linearity (curving)	Quarterly	$\leq 1$ % of range	(a)	( $\leq 2$ % of range)
Intercorrection	Quarterly	$\pm 0.02$	(a)	none
<b>Calibration</b>				
Mean bias	Weekly	$\pm 0.02$ % units	(b)	$\pm 5$ % relative
Slope	Quarterly	$1.00 \pm 0.02$ $(1.00 \pm 0.03)^*$ $(1.00 \pm 0.05)^{**}$	(b) (c) (c)	$1.00 \pm 0.05$ $(1.00 \pm 0.07)^*$ (c)
<b>Overall daily stability</b>				
Repeatability (sr)	Daily/every	$0.014$ % units	(a)	$5$ % relative
Start-up		$0.020$ % units*	(a)	
Daily/short-term stability	$\geq 3$ /hour	$\pm 0.05$ % units	(a)	$\pm 10$ % relative
Zero-setting	$\geq 4$ /day	( $\pm 0.03$ % units)	(c)	( $\leq 5000$ cf/ml)

(a): Limit stated in ISO 9622 | IDF 141 or ISO 13366 | IDF 148  
(b): Limit stemming from specifications of ISO 9622 | IDF 141 or ISO 13366 | IDF 148  
(c): Tentative (indicative) limit as there is no value specified in corresponding international standards  
\*: Limit for first generation instruments  
\*\*: Limit for lactose

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## ICAR 乳質分析實驗室能力測試 ICAR Proficiency Test

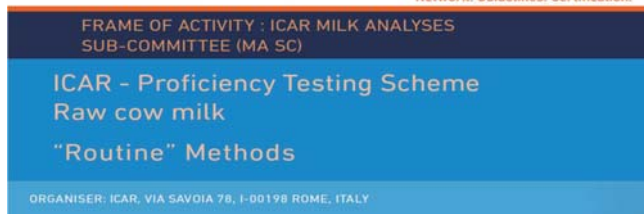


### 國際驗證

ICAR 牛乳分析實驗室能力測試 每年3月及9月

ICAR Proficiency Test held in every March and September since 2016

Network, Guidelines, Certification.



實驗室能力測試 比對項目 (Parameters) : Fat, Protein, Lactose, Urea, Somatic Cells

2017年3月 新增比對項目 (Mar. 2017 New Parameters) : BHB, PAGs (ELISA Method)

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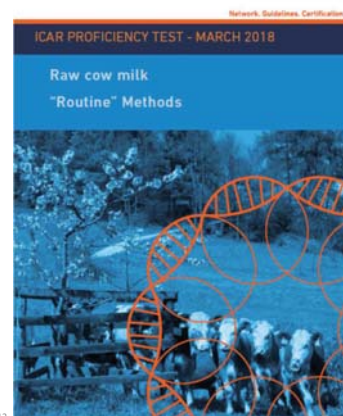
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## ICAR 乳質分析實驗室能力測試報告 ICAR Proficiency Test Report



29個國家共45個實驗室參加2018年3月的能力比對

45 laboratories in 29 countries participated in the PT in Mar. 2018



ICAR Proficiency Test (PT) "Routine" methods – March 2018

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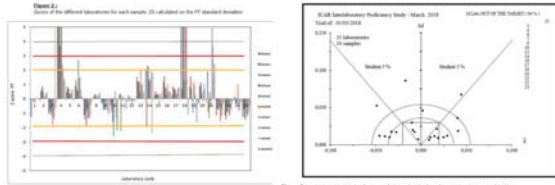
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# ICAR 乳質分析實驗室能力測試報告 ICAR Proficiency Test Report

**Table I:** Ranking of the laboratories

Nb	%	N°	d	Sd	D	Method
1	4	12	- 0,003	0,008	0,008	IR
2	8	8	+ 0,009	0,007	0,011	IR
3	12	15	+ 0,010	0,010	0,015	IR
4	16	1	- 0,007	0,017	0,019	IR
5	20	2	+ 0,015	0,012	0,019	IR
6	24	5	+ 0,019	0,009	0,021	IR
7	28	24	- 0,009	0,020	0,022	IR
8	32	17	+ 0,025	0,010	0,027	IR
9	36	20	+ 0,017	0,021	0,028	IR
10	40	16	+ 0,029	0,012	0,031	IR
11	44	11	- 0,010	0,030	0,031	IR
12	48	9	- 0,027	0,017	0,032	IR
13	52	23	- 0,035	0,010	0,036	IR
14	56	22	- 0,033	0,018	0,037	IR
15	60	25	- 0,040	0,011	0,041	IR
16	64	13	+ 0,041	0,019	0,045	IR
17	68	19	+ 0,002	0,046	0,046	⊙
18	72	7	- 0,046	0,012	0,047	IR
19	76	6	+ 0,040	0,036	0,054	IR
20	80	10	- 0,049	0,052	0,071	IR
21	84	14	+ 0,044	0,067	0,081	IR
22	88	3	- 0,017	0,086	0,088	IR
23	92	21	+ 0,102	0,219	0,242	IR
24	96	4	+ 0,274	0,241	0,365	IR
25	100	18	+ 0,423	0,411	0,590	IR

每個比對項目的能力比對結果  
The PT results of each parameter



**Table III:** Percent of the different laboratories for each sample

Sample	1	2	3	4	5	6	7	8	9	10
1	0,18	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04
2	0,18	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04
3	0,18	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04
4	0,18	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04
5	0,18	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04
6	0,18	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04
7	0,18	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04
8	0,18	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04
9	0,18	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04
10	0,18	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04	0,04

**Table IV:** Outlier identification

Sample	1	2	3	4	5	6	7	8	9	10
Outliers	3	10	11	3	3	3	3	3	3	3
Cochran	3	10	11	3	3	3	3	3	3	3
Grubbs	4	10	11	4	4	4	4	4	4	4
ar	0,008	0,004	0,004	0,005	0,005	0,004	0,004	0,004	0,004	0,007
Stat	0,044	0,026	0,025	0,028	0,045	0,037	0,038	0,037	0,033	0,060



# 台灣乳質分析設備現況 Taiwan Milk Analyzers Status

- ▶ 畜產試驗所 DHI 實驗室，使用 ICAR 認證的乳質分析設備，檢測牛乳中脂肪、蛋白質、乳糖、總固形物、尿素、酪蛋白、檸檬酸、脂肪酸含量、游離脂肪酸、酮症及體細胞數

TLRI DHI Lab uses ICAR-certified Milk Analyzers to detect Fat, Protein, Lactose, TS, Urea, Casein, Citric Acid, Fatty Acids, Free Fatty Acid, Ketosis and Somatic Cells in milk



- ▶ 超過70%的乳品工廠，使用 ICAR 認證的乳質分析設備，檢測生乳計價及品質控管

More than 70% of dairy companies use ICAR-certified Milk Analyzers for raw milk payment test and quality control



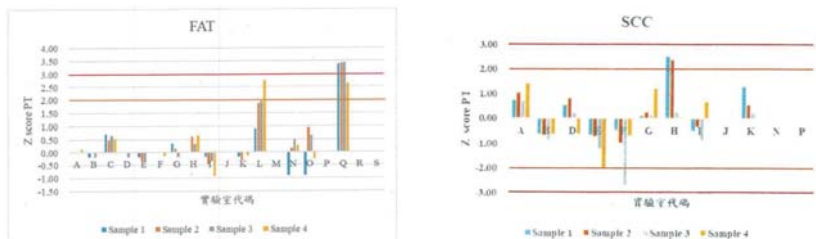
# 台灣乳質分析實驗室能力測試 Taiwan Proficiency Test

## 台灣驗證

台灣牛乳分析實驗室能力測試 每年4月及10月 由畜試所舉辦  
Taiwan Proficiency Test held in every April and October by TLRI

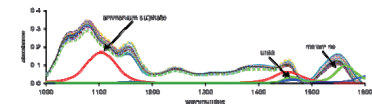
依循 ICAR 乳質分析能力測試之方法，對乳品工廠計價檢測實驗室進行能力試驗  
Follow ICAR Proficiency Test methods to do PT for dairy companies payment lab

實驗室能力測試 比對項目 (Parameters) : Fat, Protein, Lactose, SNF, Somatic Cells



# FTIR 檢測技術於乳質分析之新應用 New Applications of FTIR in Milk Analysis

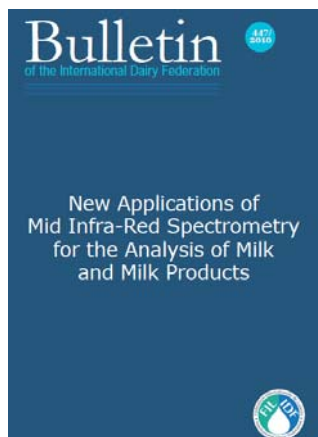
- Mid-Infrared (MIR) spectroscopy has been available since the 1970's as a rapid method for routine measurements of the main constituents in milk (fat, protein, carbohydrates)
- Fourier Transform Infrared (FT-IR) spectroscopy is the state-of-the-art method for acquiring MIR spectra analyzing:
  - Conventional parameters
    - Fat, Protein (true & crude), Casein, Lactose, Solids, Urea, Citric Acid, Free Fatty Acids, PH, Freezing Point Depression
  - New parameters
    - Fatty Acids
    - Ketosis Screening (BHB and Acetone)
    - Abnormal Milk Screening
    - Others



## 國際乳業聯盟(IDF) 公告 Bulletin-International Dairy Federation, IDF

### Catch the next big wave in milk testing

- Published December 2010
- Analysis of results gained using different equipment in 11 different laboratories across Europe
- Assessment of accuracy, reproducibility and repeatability



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## 脂肪酸組成 Fatty Acids Profile

### 為什麼要檢測脂肪酸 Why is Fatty Acid profile interesting:

- ▶ Consumers want to get the right fat from their diet
  - ▶ The fat should be unsaturated rather than saturated
  - ▶ High cholesterol levels causes heart diseases
- ▶ The dairies need to offer **value added products** to stay profitable
- ▶ Improved nutritional image of milk



### 脂肪酸含量之應用 The Fatty Acid Profile can be used for:

- ▶ **Payment analysis of milk according to:**
  - ▶ Unsaturated fatty acids
  - ▶ Saturated fatty acids
- ▶ The farmers can use the results to optimize the feed of the dairy herd regarding target amounts of unsaturated fatty acids in the raw milk
  - ▶ Short term by changes in the feeding
  - ▶ Long term by breeding in order to favor a specific fatty acid profile in milk



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

### 影響游離脂肪酸含量之因素 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

### 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



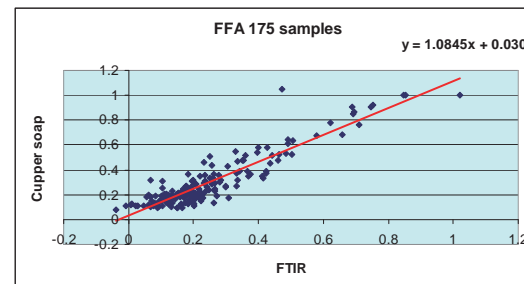
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## 以FTIR技術檢測游離脂肪酸之優點 FFA Calibration and Benefits with Milk Analyzers

### Milk analyzer 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'

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

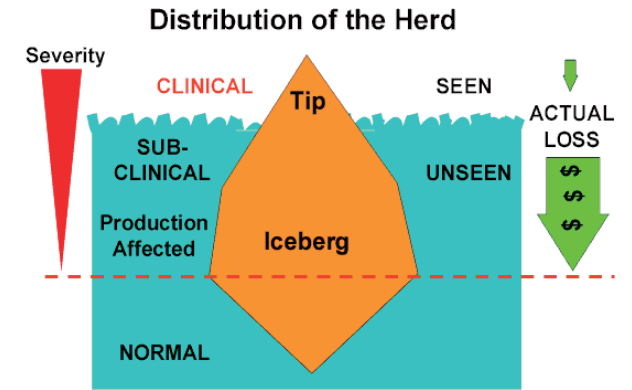
FFA is today measured in:

- ▶ The Netherlands
  - ▶ Norway
  - ▶ France
  - ▶ Belgium
  - ▶ Japan
  - ▶ Italy
  - ▶ UK
  - ▶ Canada
  - ▶ USA
  - ▶ Denmark
  - ▶ Spain
  - ▶ Brazil
  - ▶ The Czech Republic
  - ▶ Taiwan
- } **FFA included in Payment Scheme**



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## 酮症的真實情況 The Real Situation of Ketosis



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## 以FTIR乳質分析設備快速篩檢酮症 Semi-quantitative Method for Ketosis Screening

- ▶ **Limit for subclinical ketosis is**
  - ▶ 0.15 mmol/L for Acetone
  - ▶ 0.1 mmol/L for BHB

They may not increase simultaneously
- ▶ **Screening model used for classifying the animals as either "Low risk" or as "High risk" of ketosis**



Low risk

High risk of ketosis

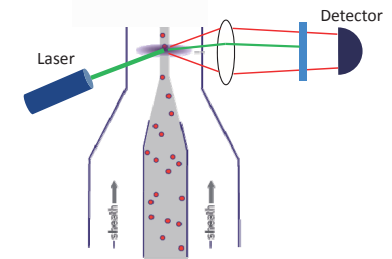
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## 以流式細胞儀檢測體細胞數之新趨勢 New Application of SCC in Milk

**ISO 13366-2 / IDF 148-2 / AOAC 978.26** International Routine Methods for Somatic Cell Count based on **Flow Cytometry technology**

The basic steps are the following:

- ▶ The cells are stained with a fluorescent dye.
- ▶ The sample is stretched to a very thin string (20 µm).
- ▶ The sample passes a focused light beam which excites fluorescence from the dye.
- ▶ The cells are seen as individual light pulses. The fluorescence is collected, filtered and detected.



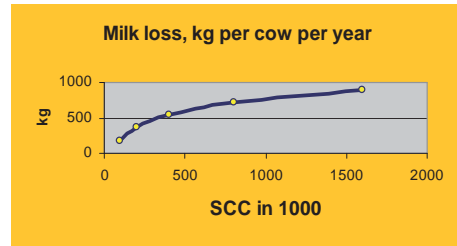
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## 乳房炎造成之影響 Mastitis Consequences

### Lower compositional value

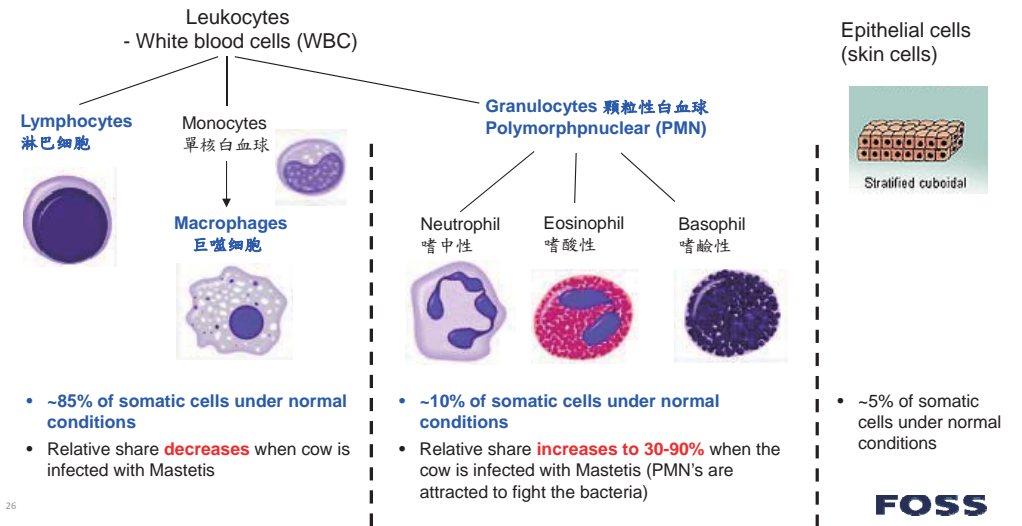
Factor	Increase	Decrease	Effect for the Dairy
Fat		↓	Economy
Free Fatty Acids	↑		Taste problem
Total casein		↓	Less cheese yield
Lactose		↓	Economy
Sodium	↑		Taste problem
Chloride	↑		Taste problem
Calcium		↓	Quality of the product
Lipase	↑		Taste problem
Bacteria	↑		Low quality and economy

### Lost milk production

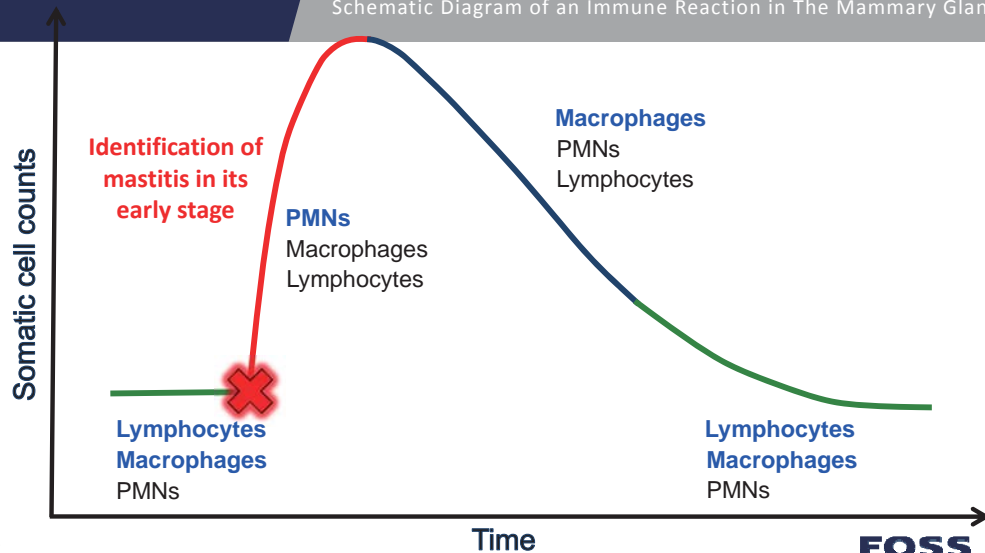


**Worldwide, mastitis is associated with economic losses of \$35 billion annually (Wellenberg et al., 2002)**

## 牛乳中細胞之類別 Type of Cells in Milk

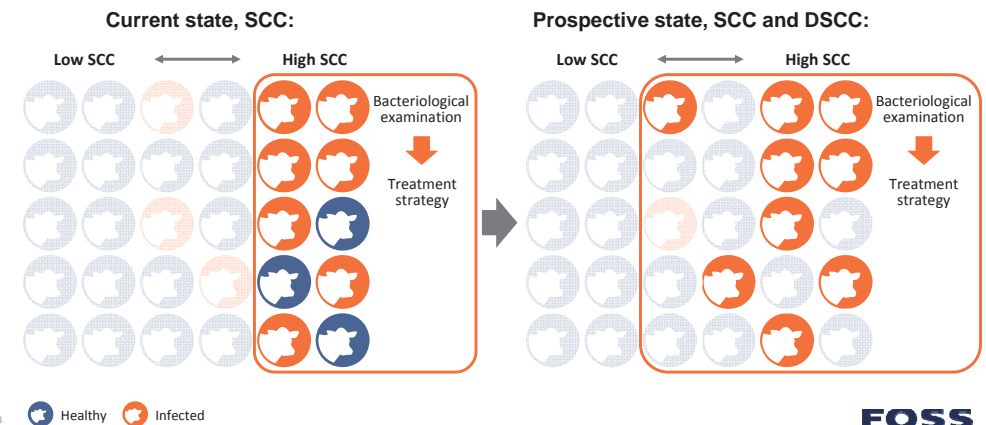


## 乳房炎免疫反應 Schematic Diagram of an Immune Reaction in The Mammary Gland



## 乳房炎監測工具 Enhanced Tool for Mastitis Monitoring

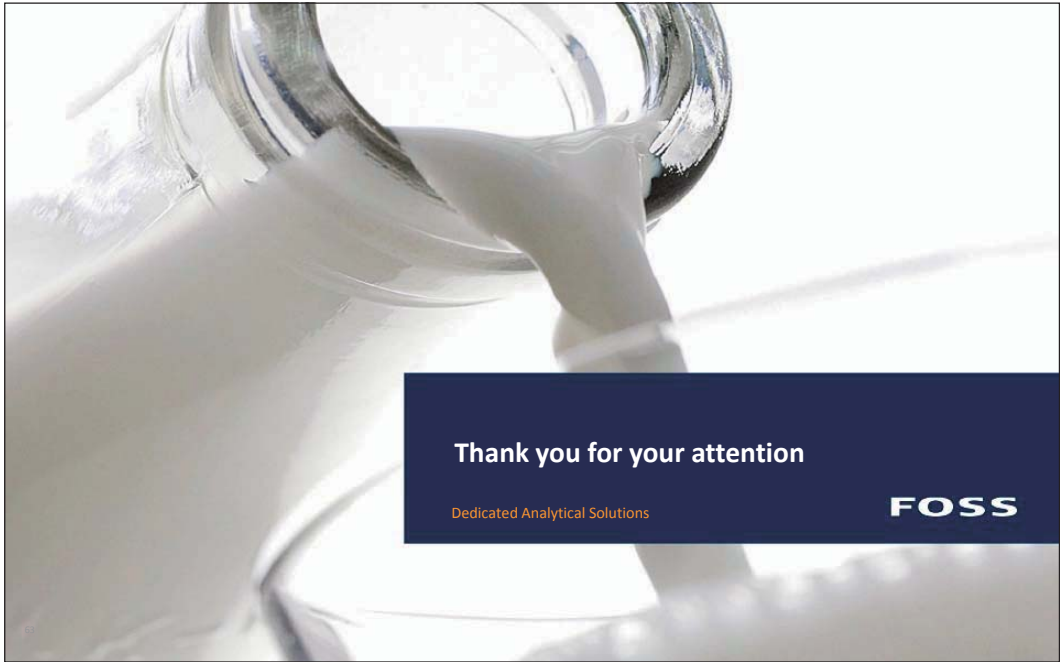
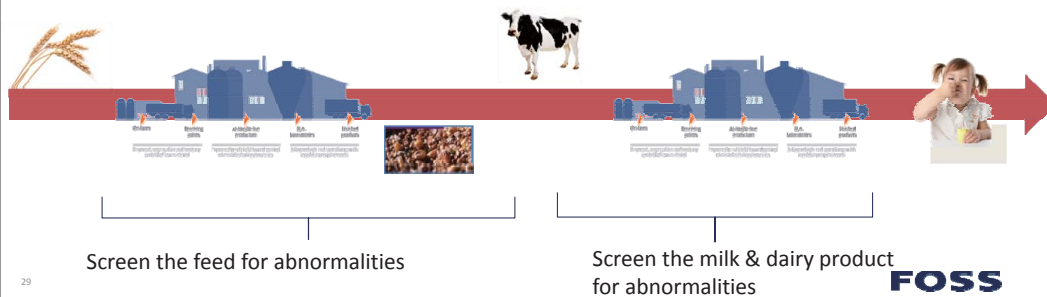
### Targeted selection of suspicious cows Identification of mastitis in its early stage



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