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# QUALITY ASSURANCE TOOLS IN MILK-TESTING LABORATORIES – THE VIEW OF AN INSTRUMENT MANUFACTURER

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ANALYTICS BEYOND MEASURE

ICAR THE GLOBAL STANDARD FOR LIVESTOCK DATA

Annual Conference

ICAR2018.NZ

7 – 11 February 2018

Aotea Centre

Auckland,

New Zealand

INTERBULL

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ANALYTICS BEYOND MEASURE

# INTERNATIONAL CERTIFICATIONS OF MILK ANALYSERS

# CONFUSION

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# OVERVIEW ON INTERNATIONAL VALIDATIONS

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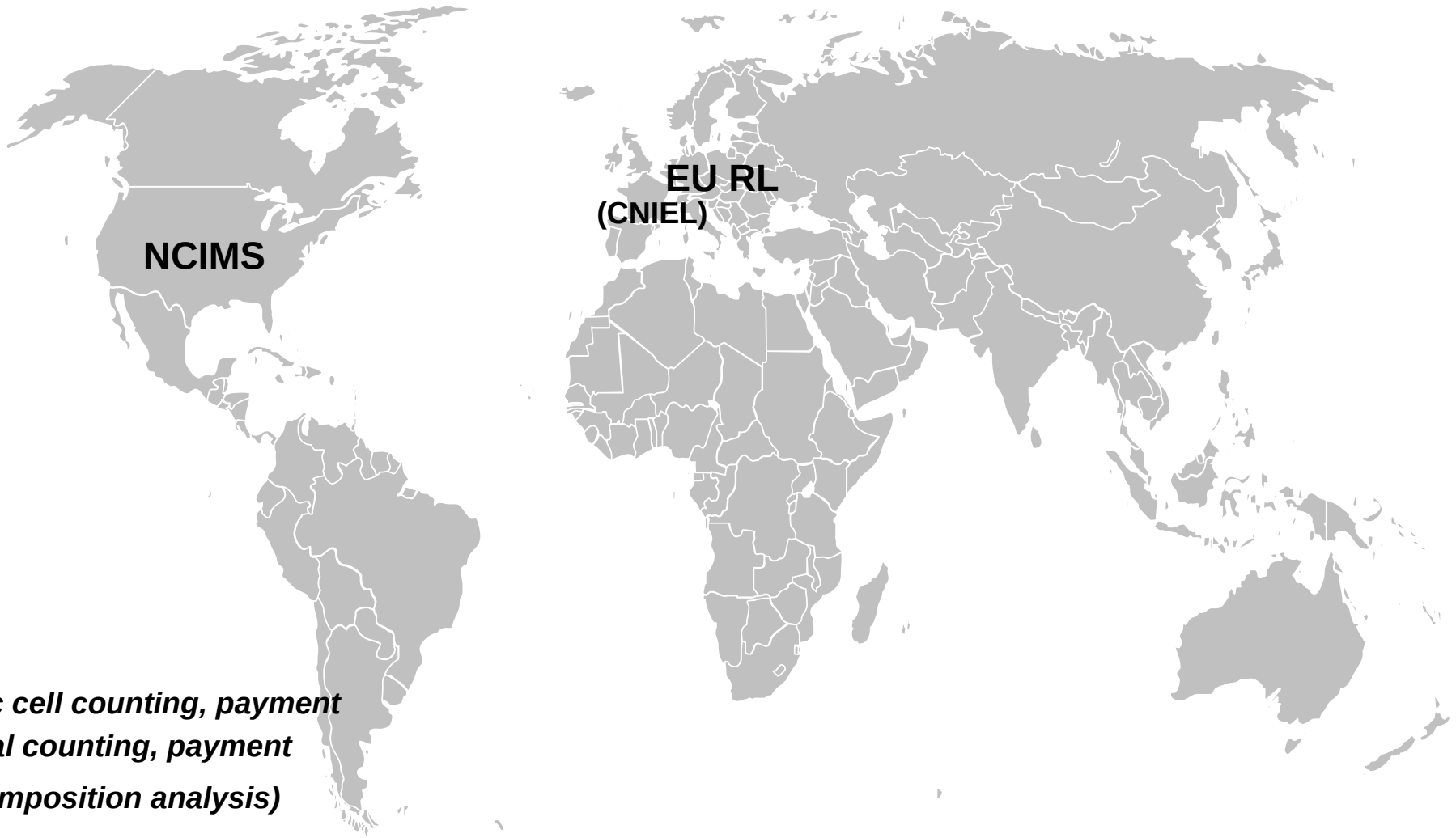
ANALYTICS BEYOND MEASURE




***Somatic cell counting, payment***
***Bacterial counting, payment***
***(Milk composition analysis)***

# SOMATIC CELL COUNT – EU RL

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- Validation testing according to **ISO 13366-1. ISO 13366-2. ISO 8196-3** (IDF 148-1. IDF 148-2.

| Item | |
|---|---|
| **1. Repeatability (r) in % per cell count leve** | |
| low (100 k) | |
| medium (500 k) | |
| high (1,500 k) | |
| **2. Carry-over (CO) in % per cell count leve** | |
| low (500 k) | C |
| medium (1,000 k) | C |
| high (3,000 k) | C |
| **3. Linearity ($r_c$) in %** | |
| **4. Lower limit of quantification** | |
| **5. Upper limit of quantification** | |
| **6. Intra-laboratory reproducibility ($R_{intra\text{-}lab}$) in % per cell count level** | |
| low (50-200 k) | |
| medium low (201-400 k) | |
| medium (401-650 k) | |
| medium high (651-1,000 k) | |
| high (1,000 -1,500 k) | |

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# SOMATIC CELL COUNT – EU RL

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## Issued certificates

After a successful validation process of a test kit is certified. The table belo

| Test kit name | |
|---|---|
| Supplier - manufacturer | Foss Analytical A/S |
| Analyte | Somatic cell count |
| Matrices | Raw cow´s milk |
| Reference method | ISO 8196-3; ISO 13366-2; EURL MMP Criteria |
| Certificate number | 2016LR62 |
| First approval date | 17-10-2017 |
| Expiry date | 16-10-2021 |
| Status | Valid |
| Certificate issued by | Lloyd's Register Nederland B.V. |
| PDF 1 | Download |

http://microval.org/en/issued-certificates/

# SOMATIC CELL COUNT – NCIMS

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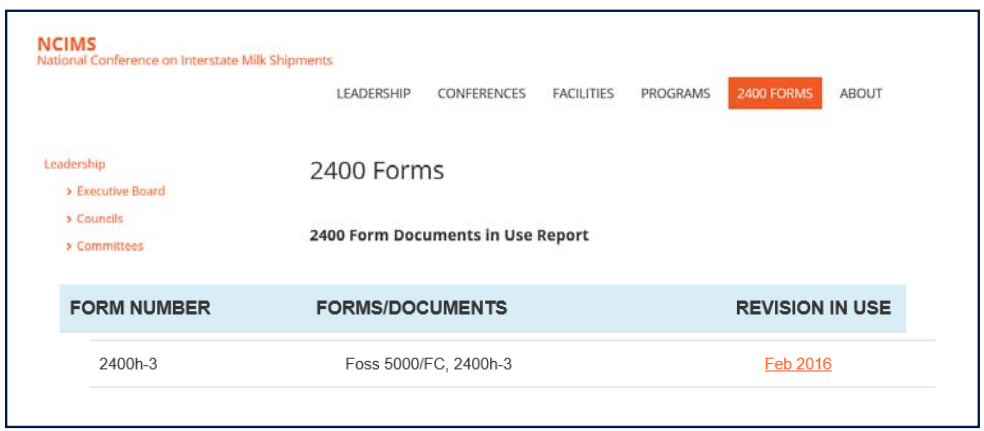
ANALYTICS BEYOND MEASURE

- Validation: correlation to reference method (i.e., microscopy (IDF 148-1))




- 2400-form:

**ELECTRONIC SOMATIC CELL COUNT**

**Fossomatic™ 5000/FC**
**(Raw Commingled Cow, Sheep, Goat, Water Buffalo and Camel Milk)**
**IMS #16**

**(Unless otherwise stated all tolerances ±5%)**

http://ncims.org/forms/

# MILK COMPOSITION

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- Validation testing according to **ISO 8196-3** (IDF 128-3) and specifications of CNIEL

| Item | | | |
|---|---|---|---|
| **1. Accuracy ($s_{y,x}$) in g/l** | | | |
| Fat | | | |
| Protein | | | |
| **2. Repeatability ($S_r$) in g/l** | | | |
| Fat | | | |
| Protein | | | |
| **3. Linearity ($r_c$) in %** | | | |
| Fat | | | |
| Protein | | | |
| **4. Carry-over (CO) in %** | | | |
| Fat | | | |
| Protein | | | |
| **5. Stability according to ISO 8196-3** | | | |

*= not determined

# BACTERIAL COUNT (EU RL AND NCIMS)

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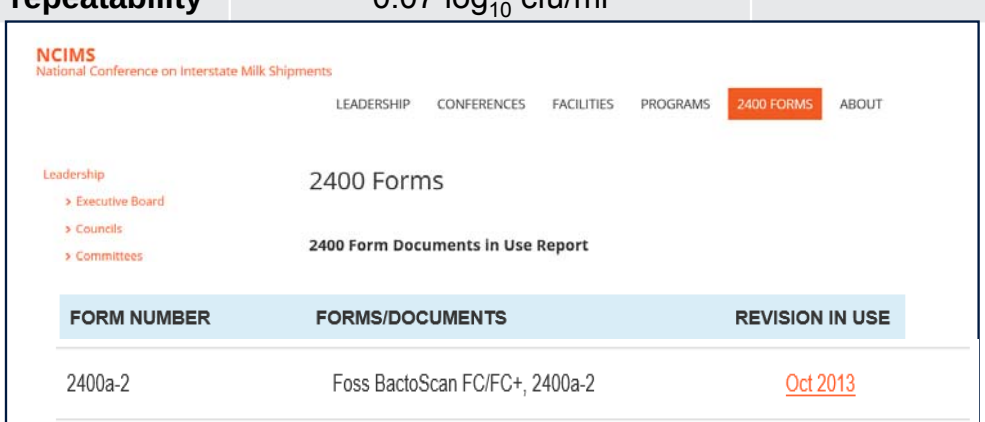
- Validation testing according to **ISO 4833-1, ISO 4833-2, ISO 16140-2**

| Item |
|---|
| 1. Repeatability (r) |
| 2. Carry-over (CO) |
| 3. Linearity ($r_c$) in % |
| 4. Lower limit of quantification |
| 5. Limit of detection |
| 6. Upper limit of quantification |
| 7. Accuracy ($s_{y,x}$) in range 1 x $10^4$ – 1 x $10^6$ cfu/ml |
| 8. Interlaboratory repeatability |

0.07 $\log_{10}$ cfu/ml



NCIMS
National Conference on Interstate Milk Shipments

LEADERSHIP CONFERENCES FACILITIES PROGRAMS 2400 FORMS ABOUT

Leadership
› Executive Board
› Councils
› Committees

2400 Forms

**2400 Form Documents in Use Report**

| FORM NUMBER | FORMS/DOCUMENTS | REVISION IN USE |
|---|---|---|
| 2400a-2 | Foss BactoScan FC/FC+, 2400a-2 | Oct 2013 |

Lloyd's Register LRQA

CERTIFICATE OF COMPLIANCE

MICROVAL

HEREBY DECLARES THAT THE CERTIFICATION ASSESSMENT BY

LLOYD'S REGISTER QUALITY ASSURANCE

has demonstrated that

BactoScan FC and FC+™

Manufactured and supplied by:
Foss Analytical A/S
Foss Allé
DK-3400 Hillerød
Denmark

has been validated and revealed to be at least equivalent to the reference methods as demonstrated by the validation study report. The summary of the validation report is available on the MicroVal website: www.microval.org

Reference method:
1. EN-ISO 4833-1:2013 Microbiology of the food chain – Horizontal method for the enumeration of microorganisms – Part 1: Colony count at 30 degrees C by the pour plate technique.
2. EN-ISO 4833-2:2013 Microbiology of the food chain – Horizontal method for the enumeration of microorganisms – Part 2: Colony count at 30 degrees C by the surface plating technique.

Scope:
Raw cow milk

The validation and certification has been performed in accordance with EN ISO/DIS 16140-2:2013, the EURL MMP criteria and the MicroVal Rules and Certification Scheme version 8.

Certificate no.: 2013LR45

First approval date: 26 February 2015
Expiry date: 26 February 2019

ISSUED BY: Lloyd's Register Nederland B.V.
Rotterdam, The Netherlands

Certificate no.: 2013LR45 26-02-2015 Page 1 of 1

# NEW ICAR SERVICE:
# MILK ANALYSER CERTIFICATION

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ICAR THE GLOBAL STANDARD FOR LIVESTOCK DATA

ANALYTICS BEYOND MEASURE

- Particular attention for DHI analyses
- The ICAR certification will follow the ICAR **Protocol for evaluation of milk analysers and ISO 8196-3** (*Milk — Definition and evaluation of the overall accuracy of alternative methods of milk analysis*)
- Harmonised protocol that serves the interest of milk recording world-wide → avoid national certifications

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ANALYTICS BEYOND MEASURE

# SPECTRAL ANALYSIS OF MILK

# FTIR ANALYSIS OF MILK SAMPLES

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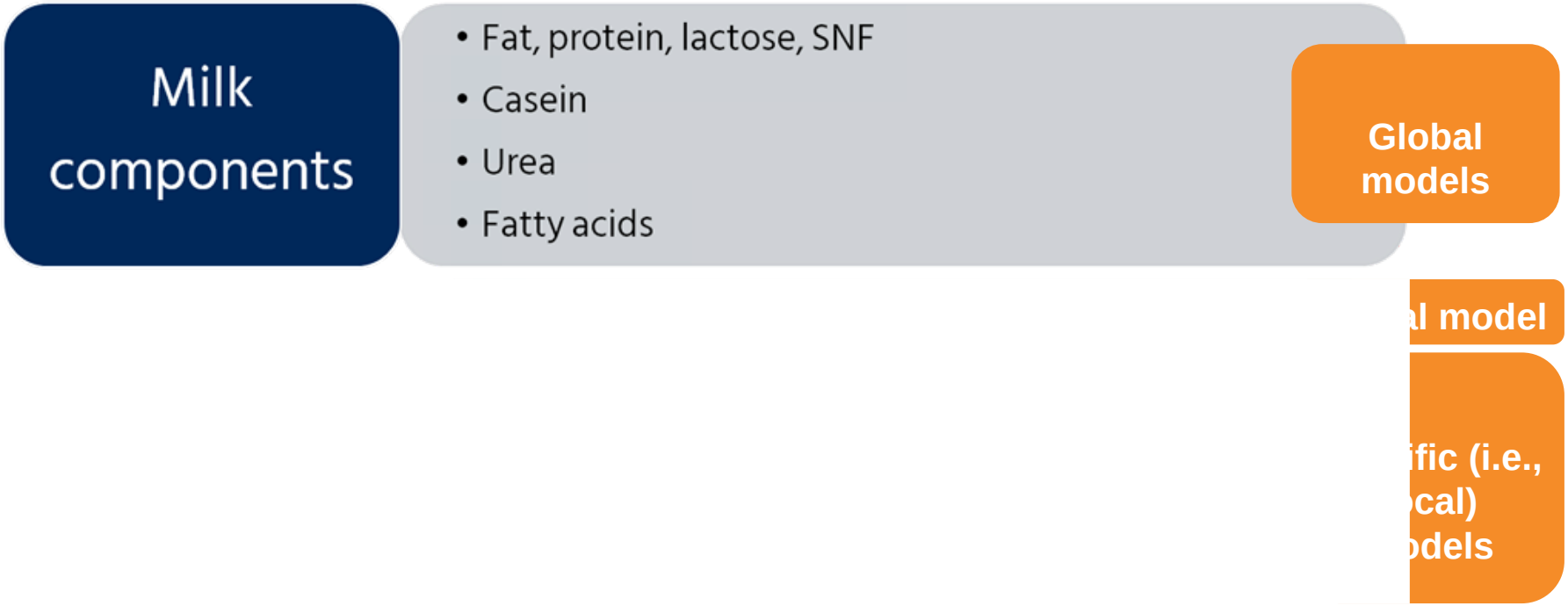
ANALYTICS BEYOND MEASURE




according to Scott et al., ICAR 2016 Annual

# STANDARDISATION OF FTIR ANALYSERS

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- Introduced in 1995
- Based on running FTIR Equalizer (frequency: 1/month)
- Prerequisite for global transfer and application of calibrations


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ANALYTICS BEYOND MEASURE

# NEW PARAMETERS FOR MILK TESTING AND REFERENCE METHODS

# EXAMPLE 1: KETOSIS SCREENING

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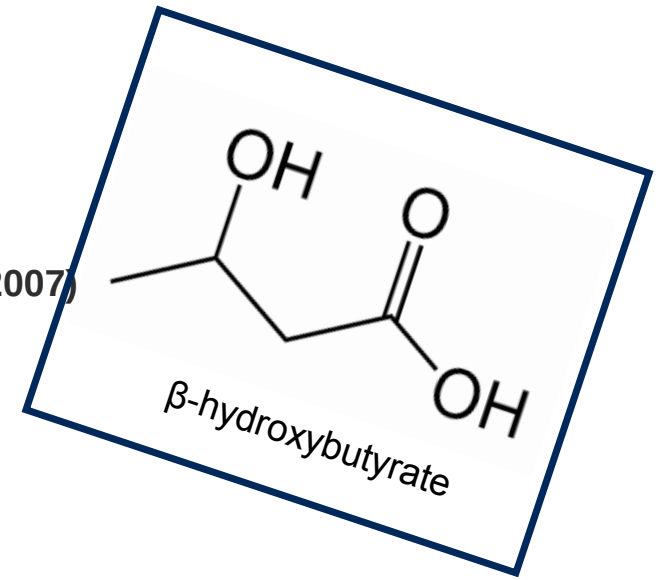
- History:
  - FTIR-based prediction of milk acetone **(Hansen, 1999)**
  - FTIR-based prediction of milk β-hydroxybutyrate **(deRoos et al., 2007)**




- Concentrations:
  - Milk urea: typically 1.5-4.5 mmol/l
  - Milk β-hydroxybutyrate: typically 0.0-0.4 mmol/l

- Quality assurance as key to success when establishing ketosis screening service

# FTIR – BHB PREDICTION MODEL

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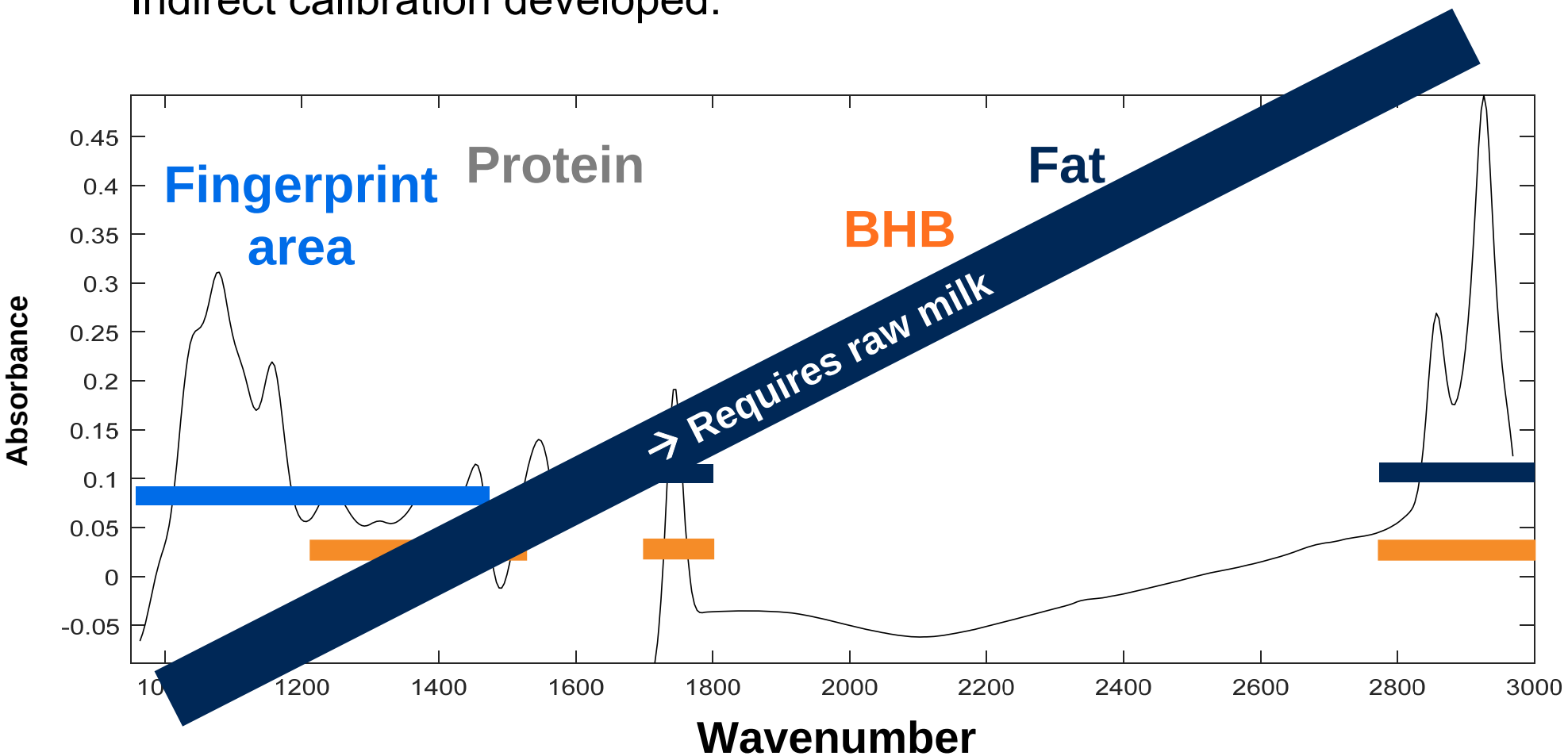
Indirect calibration developed:

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**Correlation to wet-chemistry method: 0.82 (de Roos et al., 200**

# QA PROGRAMME IN CANADA

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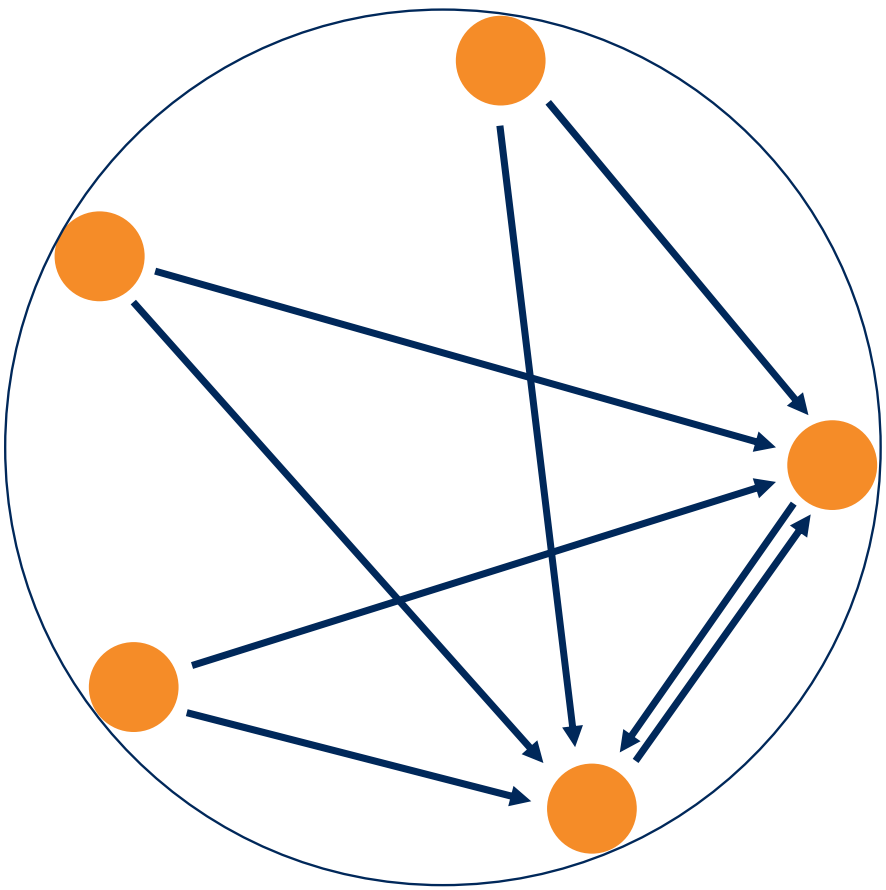
- All laboratories offering ketosis screening participate in QA programme:



**Valacta, reference results (wet chemistry method) for 100 random samples**
**Provision of BHB pilot samples**

Frequency: 1/month

ANALYTICS BEYOND MEASURE

# QA PROGRAMME IN FRANCE

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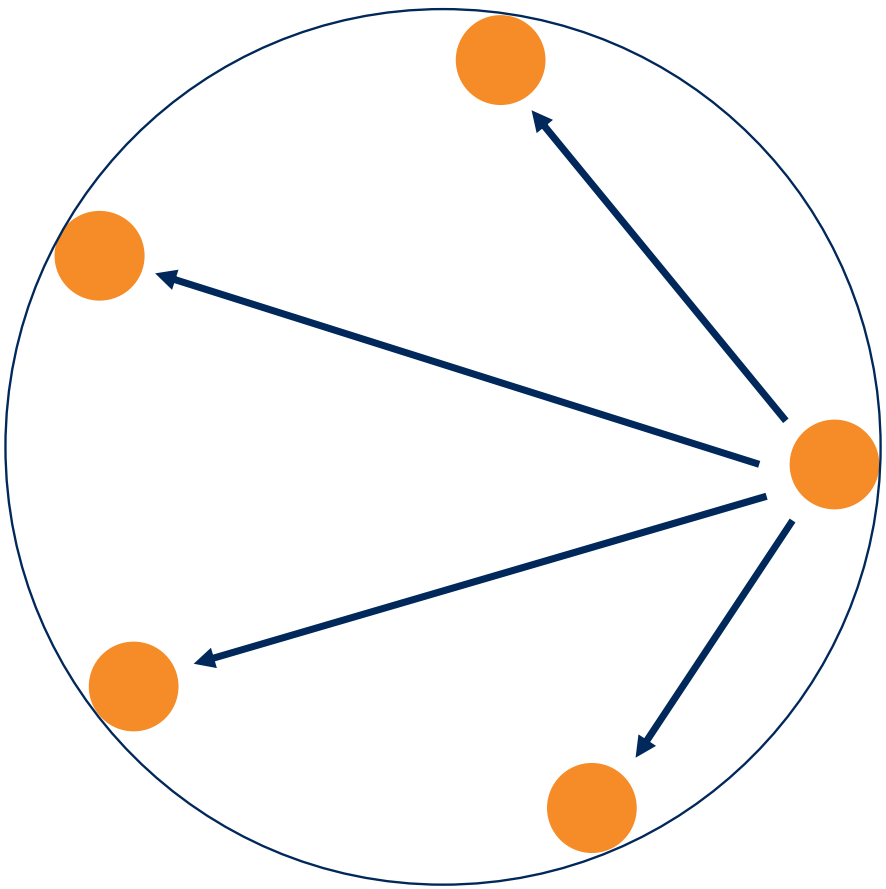
- All laboratories offering ketosis screening participate in QA programme:



**Reference laboratory, wet chemistry method**
**10 reference samples for BHB (0.05-0.25 mmol/l) and 5 samples for acetone (0.10-0.20 mmol/l)**

Frequency: 1/month

# IDF GUIDELINE

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- Action Team S03b:
  New applications of IR spectrometry
- Best practise cases on working with new FTIR-based parameters
- New guideline to be published in 2018

# EXAMPLE 2: DIFFERENTIAL SOMATIC CELL COUNT

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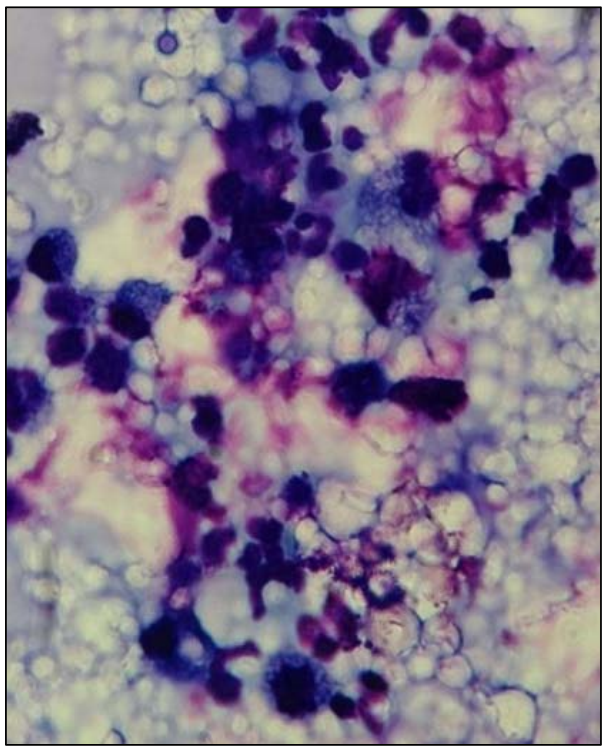
- Introduced in 2016 **(Damm et al., 2017; Schwarz, 2017)**

- Three types of immune cells in milk **(e.g., Oviedo-Boyso et al., 2007)**:
  - Lymphocytes
  - Macrophages
  - Polymorphonuclear neutrophils (PMN)

- Differential Somatic Cell Count (DSCC):
  - →Represents the combined proportion of the PMN and lymphocytes in percent
  - →The percentage of Macrophages is 100 – DSCC



**Microscope spot, milk cells**

# THE FOSS DSCC METHOD – SCC AND DSCC SIMULTANEOUSLY

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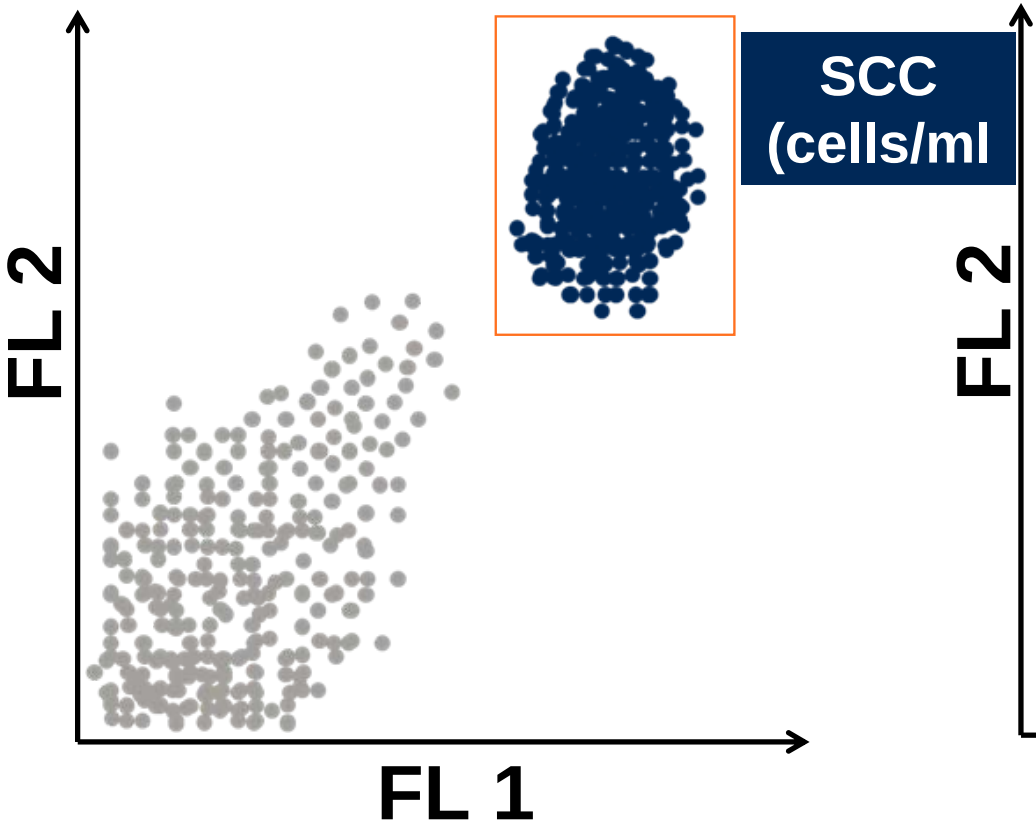
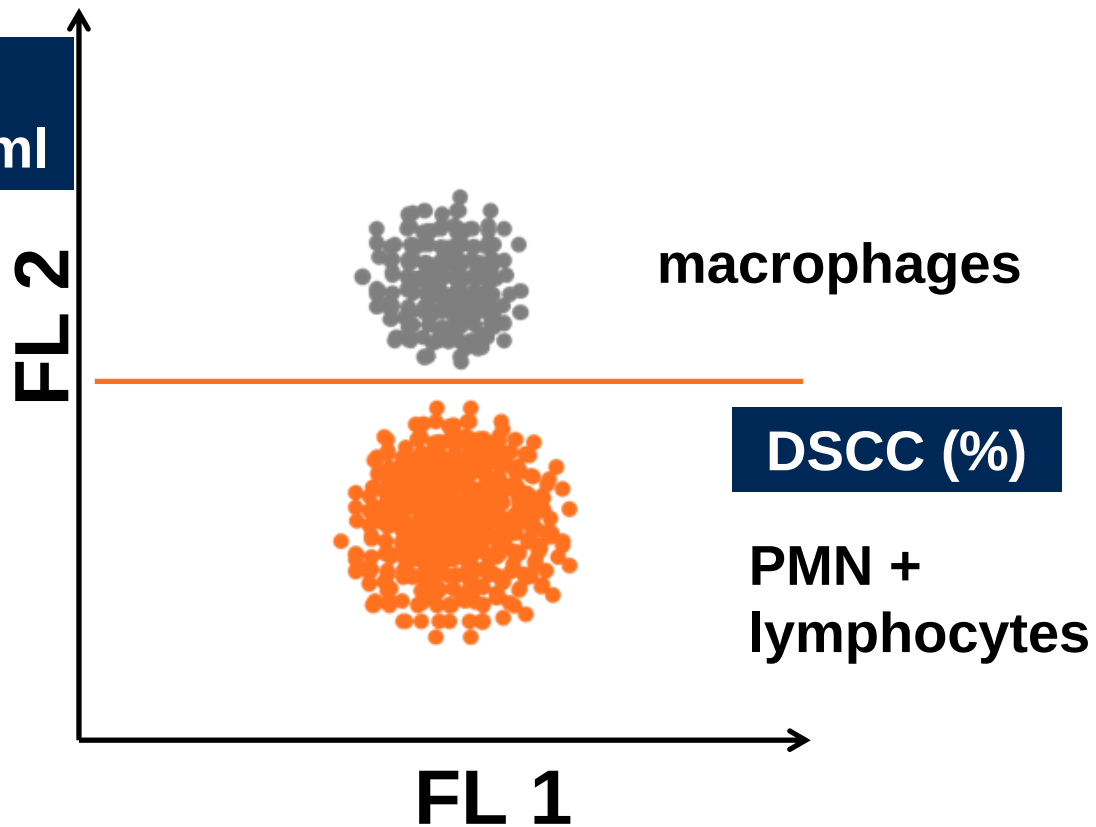
## Analysis of SCC




## Analysis of DSCC




SSC information used supportively for determination of SCC

Damm et al., 2017; Schwarz, 201

# IDF WORK

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- Action Team S15:

Bulletin on improvement of the reference method for somatic cell counting

- Possibilities/technologies for the SCC reference method including DSCC capability

- Bulletin to be published in 2018

# A MESSAGE TO TAKE HOME

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- Raw milk samples hold a wealth of information – milk quality and dairy herd management → new instruments and parameters

- FOSS supports quality assurance through
  - Certification according to internationally-accepted standards
  - Standardisation concept for FTIR analysers
  - Development of guidelines/best practise cases for parameters where reference methods are not yet available


Lloyd's Register LRQA

MICROVAL NEN

CERTIFICATE OF COMPLIANCE

LLOYD'S REGISTER QUALITY ASSURANCE

hereby declares that the certification assessment has demonstrated that

Fossomatic™ 7

for enumeration of somatic cells in raw cow's milk

Manufactured and supplied by:
FOSS ANALYTICAL A/S
FOSS ALLÉ
DK-3400 HILLERØD
DENMARK

has been validated and rfhvealed to be at least equivalent to the reference method as demonstrated by the validation study report. The validation report is available on the Microval website: www.microval.org

Reference method: ISO 13366-1:2008 Milk -- Enumeration of somatic cells - Part 1: Microscopic method (Reference method)

Scope: raw cow's milk

The validation and certification has been performed in accordance with ISO 8196-3, ISO 13366-2, the EURL MMP criteria and the Microval Rules and Certification Scheme version 8.

Certificate no.: 2016LR62

First approval date: 17
Expiry date: 2021

ISSUED BY: Lloyd's Register Nederland B.V.
Rotterdam, The Netherlands

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