ICAR Reference Laboratory Network - 4th Meeting, Niagara Falls, 16 June 2008

- Agenda -

8.00 : Opening - Welcome - Round table for presentation

8.20 : Introduction: ICAR Reference Laboratory Network history and objectives
ICAR analytical strategy - International anchorage & harmonisation  
(O. Leray, Cecalait, FR)

8.50 : Interlaboratory reference systems and centralised calibration – Prerequisites and standard optimum procedures
(O. Leray, Cecalait, FR)

9.10 : Discussion

9.40 : The way to reference systems and centralised calibration for milk recording testing - Present status in Germany
(C. Baumgartner, MPR, DE)

10.00 : Health break

10.20 : Reference system and centralised calibration for milk recording testing in Argentina
(R. Castañeda, Inti-Lacteos, AR)

10.40 : Reference system and centralised calibration for milk (payment) testing in USA.
(D. Barbano, Cornell University, USA)

11.00 : Assessment of laboratory performances and analytical equivalence in milk testing in North America,
(P. Sauvé, Canadian Laboratory Services, CA)

11.30 : Discussion

12.00 - Closure of the meeting
- INTRODUCTION - GENERAL OBJECTIVES -

**History:** ICAR Session in Ottawa 1994,
=> Analytical Quality Assurance (AQA) policy by ICAR

**General objective:** Develop an international AQA system for DHI based on harmonised laboratory practices.

**Goal:** Confidence, equivalence, comparability
=> within / between countries,
=> worldwide: international genetic evaluation.

**Implementation by MA SC (MTL WG):**
> **Guidelines** for the harmonisation of analytical practices:
  Analytical methods, Quality Assurance,

> **International network** of reference laboratories for milk recording analytical performances

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**ROLES OF THE LABORATORY NETWORK**

ICAR Reference Laboratory Network is *expected* to operate as an international platform for milk recording as to

- diffuse/promote GLP and AQA based on international guides and standards => communication (*Internet, website*)

- provide precision traceability and anchorage to consensual international "true values" to routine labs via network members => analytical data harmonisation (*PTs, RM*)

- a mean for developing collaborations for laboratory purposes => Co-operation (*Education, training*)

*Model & explanation provided every year to ICAR member organisations*
THEORETICAL STRUCTURE

Missions / activities expected
- Eligibility criteria -

- 1- National ring test organizer
- 2- Reference Material supplier
- 3- Master laboratory for centralized calibration
- 4- Teaching and training in laboratory techniques
- 5- Information on analytical methods
- 6- Evaluation of analytical methods/instruments
- 7- Research on analytical methods
- 8- National regulatory control of analyses
- 9- Routine testing where only 1 or 2 labs/country
ICAR Reference Laboratory Network
Composition & evolution
from 1998 to 2008

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ICAR Reference Laboratory Network
Membership in 2008

38 laboratory members from 32 countries as follows:

Argentina (1) Austria (1) Belgium (2) Canada (1) 
Cyprus (1) Czech Republic (1) Denmark (1) Estonia (1) 
Finland (1) France (1) Germany (1) Hungary (1) 
Ireland (1) Israel (1) Italy (1) Korea (1) 
Latvia (2) Lithuania (1) The Netherlands (1) New Zealand (1) 
Norway (1) Poland (1) Slovak Repub. (1) Slovenia (1) 
South Africa (3) Spain (1) Sweden (1) Switzerland (1) 
Tunisia (2) United Kingdom (1) U.S.A. (2) Zimbabwe (1)

(n) : number of member(s)

among which: 38 members for cow
16 members for goat
14 members for sheep
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ICAR Reference Laboratory Network
- Evolution since 1998 -

### Evolution of the proportions of national roles from 1998 to 2007 (end of year)

<table>
<thead>
<tr>
<th>YEAR</th>
<th>NRTO</th>
<th>RMS</th>
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### Evolution of the composition and national roles from 1998 to 2007 (end of year)

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NRTO = National Ring Test Organiser  
RMS = Reference Material Supplier  
MLCC = Master Laboratory for Centralised Calibrations  
TLT = Training in Laboratory Techniques  
IAM = Information on Analytical Methods  
EAMI = Evaluation of Analytical Methods/Instruments  
RAM = Research on Analytical Methods  
NRCA = National Regulatory Control of Analyses  
DHIA = Dairy Herd Improvement Analyses  
Payment = Analyses for milk payment  
Membership = Officially nominated by ICAR National Committees
Eligibility criteria declared in 2008

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<th>Proportion %</th>
<th>Lab number with N</th>
<th>Lab % with N</th>
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Evolution of membership and missions/activities from 1998 to 2008
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Evolution of membership and missions/activities from 1998 to 2008

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International interlaboratory proficiency studies

From 1996: International proficiency scheme organised by ICAR
Frequency: twice a year
Participants: members of ICAR ref lab Network
Analytical methods:
- reference methods to calibrate routine methods for fat, protein and lactose
- methods for urea somatic cell counting
Type of milk: cow milk

Participation in international proficiency studies from 1998 to 2008

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Participation in international proficiency studies from 1998 to 2008

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Participation in international proficiency studies from 1998 to 2008

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CONCLUSION ON THE NETWORK IMPLEMENTATION

Nominations by national organisations:
- **Number**: Stability from 2003 around 38 members ⇒ growth completed
- **Qualification**: Increase of mission numbers (eligibility criteria)

International Proficiency Testing schemes:
- Regular participation of about 50% of laboratory network members
- Improvement of performance from 2003

ICAR AQA Strategy
International anchorage & harmonisation

Olivier Leray, Cecalait, France
ICAR analytical anchorage

**Intent**
- to establish links from local/national/regional levels to the international level
- to harmonise laboratories on a international collective reference

**Means**
- Guidelines, standards, GLP, AQA
- Interlaboratory proficiency studies ⇒ lab trueness traceability
- Reference materials ⇒ trueness improvement
Requirements for the reference

1- Technical:
   ⇒ Use of international reference methods (IDF/ISO)
   ⇒ Compliance with precision figures of the methods

2- Statistical:
   Unbiased and low uncertainty
   ⇒ sufficient number, representativeness of participants

3- Political/economical:
   recognition for the purpose
   ⇒ consensus of participants / organisations based on representativeness

For international genetic evaluation (Interbull), it should be built from results of laboratories from different countries !!!

Possible uses of interlaboratory proficiency studies

1- Measuring laboratory performance
2- Measuring result uncertainty
3- Comparing laboratories (assess equivalence)
4- Providing traceability to international reference
5- Qualifying/selecting reference/expert laboratories
6- Assessing/certifying reference materials
1- Measuring laboratory performance

Laboratory L
- participates with p laboratories, q samples in n replicates
- the estimate of sample S true value is \( \bar{X}_S \)
- means of n replicate (average) are \( \bar{X}_{Lk} \)
- level score (individual bias) is \( \delta_{Lk} = \bar{X}_{Lk} - \bar{X}_S \)

Laboratory score = Average of q level scores :
\[ \bar{d}_L = \frac{\sum \delta_{Lk}}{q} \]
also \( \bar{d}_L = \bar{x}_L - \bar{X} \)

Additionally:
- standard deviation of repeatability \( s_{rL} \)
- standard deviation of differences \( s_{dL} \)
- Euclidian distance (equivalent to SEP) \( D = (\bar{d}_L^2 + s_d^2)^{1/2} \)

Within lab reproducibility :
\[ s_{R_l}^2 = s_{rL}^2(1-1/n) + \bar{d}_L^2 + s_d^2 \]

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Evaluation example : ICAR PT scheme (10 samples in duplicates)

<table>
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<tr>
<th>Tableau I</th>
<th>Ranking of the laboratories</th>
<th>Unit : g / 100 g</th>
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Note : Limits are only indicative and so far do not constitute standard values; they indicate what is normally reachable by labs for their self evaluation.
2- Measuring test result uncertainty

Evaluation example: ICAR PT scheme (10 samples in duplicates)

ISO 5725-2: (replacing laboratory variable by trials)
Precision of Laboratory L → $s_{L_{\text{ref}}}$ and $s_{R_{\text{L,ref}}}$

ISO 5725-6: Reference method for q samples and n replicates
uncertainty $\approx \pm u_{0.975} \cdot (s_{R_{\text{ref}}}^2 - s_{L_{\text{ref}}}^2 \cdot (1-1/nq))^{1/2}$
(in calibration) $\approx \pm u_{0.975} \cdot (s_{R_{\text{ref}}}^2 - s_{L_{\text{ref}}}^2)^{1/2}$ (1)

ISO 8196: Routine (alternative) method
uncertainty $\approx \pm u_{0.975} \cdot (s_{L_{\text{ref}}}^2 + s_{y,x}^2)^{1/2}$

From (1) + (2) ⇒ Overall uncertainty of routine testing results
$\approx \pm u_{0.975} \cdot (s_{R_{\text{L,ref}}}^2 + s_{L_{\text{ref}}}^2 + s_{y,x}^2)^{1/2}$
3- Comparing laboratories

Same PT study

With scores of laboratories L1 and L2

\[ d_{L1} = \bar{x}_{L1} - \bar{x} \quad \text{and} \quad d_{L2} = \bar{x}_{L2} - \bar{x} \]

Between lab performance comparison is made through the difference

\[ d_{1,2} = \bar{x}_{L1} - \bar{x}_{L2} \quad \Rightarrow \quad d_{1,2} = d_{L1} - d_{L2} \]

Comparison between laboratories L1 & L2

Figure 1 – Between laboratory comparison through an interlaboratory study

\[ D = \bar{x}_{L1} - \bar{x}_{L2} = d_{L1} - d_{L2} \]
Parallel national and international PT studies

Thanks to scores of the reference laboratory M

in national study $\bar{d}_{MN}$
in international study $\bar{d}_{MI}$

the virtual error between reference $\Delta = \bar{d}_{MN} - \bar{d}_{MI}$
the effective score of Lab L in the national study $\bar{d}_{LN} = \bar{x}_L - \bar{x}_N$

the virtual international score of Laboratory L is

$$\bar{d}_{LI} = \bar{d}_{LN} - \Delta = \bar{d}_{LN} - \bar{d}_{MN} + \bar{d}_{MI}$$
3- Indirect laboratory comparison

**Different PT studies**

Thanks to scores of reference laboratories M1 and M2

in national studies \( \bar{d}_{MN1} \) and \( \bar{d}_{MN2} \)

in international studies \( \bar{d}_{MI1} \) and \( \bar{d}_{MI2} \)

the virtual bias between reference \( \Delta_1 = \bar{d}_{MN1} - \bar{d}_{MI1} \) and \( \Delta_2 = \bar{d}_{MN2} - \bar{d}_{MI2} \)

the effective scores in national studies \( \bar{d}_{LN1} = \bar{x}_{L1} - \bar{x}_{N1} \) and \( \bar{d}_{L2} = \bar{x}_{L2} - \bar{x}_{N2} \)

the virtual international difference between laboratories L1 and L2 is

\[
D = \bar{d}_{L1} - \bar{d}_{L2} = (\bar{d}_{LN1} - \bar{d}_{LN2}) - (\Delta_1 \cdot \Delta_2)
\]

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**Comparison between laboratories L1 & L2**

\[
D = \bar{d}_{L1} - \bar{d}_{L2} = (\bar{d}_{LN1} - \bar{d}_{LN2}) - (\Delta_1 \cdot \Delta_2)
\]

\[
\Delta_1 = \bar{d}_{MN1} - \bar{d}_{MI1}
\]

\[
\Delta_2 = \bar{d}_{MN2} - \bar{d}_{MI2}
\]

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5- Qualifying/selecting reference laboratories

**Required regular good performance in PTs**

**RMs certification:**
- Regular score compliance in a number of successive trials

**Laboratory anchorage:**
- Regular score compliance throughout time
- Constant bias (better 0) \( \Leftrightarrow \ s_{R_{L,ref}} \approx s_{R_{L,ref}} \)

**Means of success:** Trueness adequacy and stability ensured through RMs and special training, competence, caution.

6- Assessing/certifying reference materials

**Focus is given to reference values determination and reference material quality**

**ICAR protocol:**
- Experimental design for PTs also possible for RMs
- Both tools are dedicated to calibration:
  - same concentration ranges
  - same sample numbers

**Combined use is possible** provided respective specific caution:

- **Reference values:** according to ISO 5725-4 with uncertainty
- **Laboratories:** Qualified / selected on performance for the lowest uncertainty
- **Experimental design:** Consider long term homogeneity/stability
Example: Central RM system

General model: Numerous laboratories and samples; robust reference

Example: Multiple RM system = crossed system

Specific model: Homogeneous laboratory groups; numbers of laboratories and samples limited; good performance must compensate small laboratory number; consensus on group reference better than individual
Example: Mixed system

Intermediate model: Heterogeneous laboratory groups; a few laboratories address samples to a larger group; samples number still limited; more robust reference

Conclusion

International anchorage can provide objective elements on:
- the overall accuracy & uncertainty of milk testing
- the (degree of) analytical equivalence within ICAR

ICAR International Reference Laboratory Network

corner stone for analytical harmonisation in milk recording
## Agenda

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<tr>
<td>8.00</td>
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<td>ICAR analytical strategy - International anchorage &amp; harmonisation</td>
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