

Frame of activity : ICAR Milk Analyses  
Sub-Committee (MA SC)



# **ICAR - PROFICIENCY TESTING SCHEME**

## **CHEMICAL "REFERENCE" METHODS**

### **RAW COW MILK**

Organisers: ICAR, Via Savoia 78, 00191 Rome, Italy

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## ICAR Proficiency Test (PT) Chemical “Reference” methods – March 2016

### 1. Introduction

Dear Participant,

Thank you for participating in the ICAR Proficiency Test (PT) March 2016 !

In this report you will find sections 2 and 3 which are dedicated to “your” quality assurance management and section 4 dedicated to the “general” ICAR statistical elaboration for each parameter.

The proficiency test is a tool to help evaluate the performance of the laboratory process and to support your laboratory quality assurance system. It's aim is to provide independent data for you to monitor, evaluate and ultimately improve your processes as you see fit.

From the analyses of the data received we have identified some aspects that if evaluated and managed may serve to improve some control steps of your quality management ISO 17025.

When the PT samples arrive to your laboratory they can be viewed as being from a ‘customer’ that is asking you to provide timely, precise and accurate results.

In tables A,B,C,D,E,F,G if all the information is reported correctly from the participant, then the cells are filled in green, otherwise they are highlighted in red for your attention, so you can review and verify any causal reasons internally. The cell is grey if you have not participated for that parameter. The control charts H will help you to follow your performance over the time.

- A) In table A you find your participation codes and the information if all the results from the samples received, have been sent to the PT provider.
- B) In table B is indicated if the results have been sent on time.
- C) In table C is indicated if the results have been reported in the correct unit of measurements. For example in a result excel file it was requested to report the total nitrogen and not the crude protein.
- D) In this table is reported if some of your results are outlier for Grubbs or Cochran test.

## ICAR Proficiency Test (PT) Chemical “Reference” methods – March 2016

- E) The evaluation of repeatability of the results should be one of the first controls before communication of the data. In table E the absolute difference between replicates is compared with the repeatability limit of the relevant “reference” method indicated. If one or more results have a result out of the limit, the cell is in red. It may be that you have deployed a chemical method that is different from the reference method indicated. If the repeatability is bigger it will be evaluated internally with the precision of the specific method used. You can find all the detailed information of your data in Table II in the section Statistical elaboration for each parameter.
- F) In table F the results of your Z-Score<sub>PT</sub> (standard deviation calculated on this proficiency test) and the Z-Score<sub>FIX</sub> (standard deviation of the reference method) are summarized. If you have obtained all the -2<Z-Score results<+2 the cell will be filled in green. If you have obtained one or more results in the moderate or poor performance range the cells will be filled in yellow or red respectively.
- G) In table G the ranking of your lab will be green if the mean of difference and standard deviation of difference value are in the box of figure 2 of each parameter. Limits are only indicative and so far do not constitute standard values; they indicate what is normally reachable by labs for their self evaluation. According the results obtained the MA SC will decide eventually to revise. For this round the somatic cell ranking is not considered to evaluate the performance of the lab.
- H) The control charts have been created using your data obtained with the Z-Score<sub>PT</sub> and Z-Score<sub>FIX</sub>. The standard deviation of the method will be the same in the next PT round and it will facilitate the comparison of your performance over the time.

Your Control Charts will be updated with data as the next ICAR PT rounds happen and will be reported in the subsequent ICAR PT reports.

The sample preparation and statistical elaboration have been done by ICAR Sub-contractor Actalia, accredited to ISO 17043.

In the second part of the report the statistical elaboration followed the template approved by ICAR’s Milk Analyses Sub Committee chaired by Dr. Gavin Scott (NZ). You find the statistical elaboration for all the ICAR interested parameters, fat, protein, lactose, urea and somatic cell.

We think it is important to show you, as ICAR member, the reproducibility of the ICAR laboratories, even if you have not participated in this PT round.

For each parameter the SR=standard deviation of reproducibility has been calculated after the outlier elimination. If you have participated, and your results are in the repeatability limits, you can use this value for the calculation of your uncertainty of measurement.

## ICAR Proficiency Test (PT) Chemical “Reference” methods – March 2016

ICAR would like to see, in the next years, the part 4 of this report, completed with the results, reference and/or routine methods, from all the ICAR countries for the parameters indicated.

We are sure with your support and contribution it will grow to benefit all!

The list of all ICAR reference laboratories and those participated in this ICAR PT round with at least one parameter is reported below and upload on ICAR website

<http://www.icar.org/index.php/icar-certifications/milk-laboratories-proficiency-test-2016/>



# ICAR Proficiency Test (PT) Chemical “Reference” methods – March 2016

| ICAR Member with at least one Milk laboratory participating to the Proficiency Test Round (March 2016)                 | ICAR Member with no laboratory participating to the Proficiency Test Round (March 2016) |   |                 |
|--|---|---|-----------------|
| Département Valorisation des Productions agricoles (DVP), Centre Wallon de Recherche Agronomique – CRA -W              | Belgium   | INTI – LACTEOS  | Argentina       |
| T&V – ILVO Eenheid Technologie en Voeding Productkwaliteit en Voedselveiligheid  | Belgium   | Milchprüfung Süd  | Austria         |
| Valacta – Centre d'Expertise en Production Laitière du Québec  | Canada  | Shanghai Dairy Cattle Breeding Center Co., Ltd  | China           |
| Eesti Pollumajandusloomade Joudluskontrolli AS, Milk Analysing Laboratory  | Estonia   | University of Zagreb, Faculty of Agriculture, Department of Dairy Science, Reference Laboratory for Milk and Dairy Products | Croatia         |
| ACTALIA / ACTILAIT / CECALAIT  | France  | Dairy Research Institute, Pragu (MILCOM a.s.)   | Czech Republic  |
| Milchprüfung Baden-Württemberg e.V., Zentrallabor Kirchheim  | Germany   | Eurofins Steins Laboratorium A/S  | Denmark         |
| ÁT. KFT. Tejvizsgáló Laboratórium (Livestock Performance Testing Ltd )   | Hungary   | VALIO R & D Chemistry   | Finland         |
| Teagasc, Technical Services Laboratory   | Ireland   | Institute of Food Safety, Animal Health and Environment BIOR, Laboratory of Food and Environmental Investigations           | Latvia          |
| Central Milk Laboratory – ICBA   | Israel  | State Laboratory for Milk Control Pieno Tyrimai   | Lithuania       |
| Associazione Italiana Allevatori, Laboratorio Standard Latte (LSL- AIA)  | Italy   | MilkTestNZ  | New Zealand     |
| Federazione Latterie Alto Adige Soc. Agr. Coop.  | Italy   | Livestock Improvement Corporation   | New Zealand     |
| Japan Dairy Technical Association  | Japan   | TINE, Centre for R&D  | Norway          |
| Laboratorium Oceny Mleka, Krajowego Centrum Hodowli Zwierząt (KCHZ), Laboratorium Referencyjne z siedzibą w Parzniewie | Poland  | Milk Laboratory, Slovak Agricultural Research Centre  | Slovak Republic |
| PFHBiPM Laboratorium w Parzniewie  | Poland  | Veterinárny a potravinový ústav Bratislava  | Slovak Republic |
| PFHBiPM Laboratorium w Białymstoku zs.w jezewie Starym   | Poland  | University of Ljubljana, Biotechnical Faculty, Zootech. Dept., Laboratory for Dairying                                      | Slovenia        |
| PFHBiPM Laboratorium w Kobiernie   | Poland  | Deltamune PTY Ltd   | South Africa    |
| PFHBiPM Region Oceny Bydgoszcz z/s w Minikowie   | Poland  | Eurofins Steins Laboratorium AB   | Sweden          |
| Laboratorija za ispitivanje kvaliteta mleka, Poljoprivredni fakultet Novi Sad,   | Serbia  | Agroscope, Institute for Food Sciences IFS  | Switzerland     |
| ANPI Dairy Laboratory  | South Africa  | Milk Analysis Laboratory, Hsin-chu Branch, Livestock Research Institute, Council of Agriculture                             | Taiwan          |
| Lab Agroalimentario de Santander   | Spain   | Office de l'Elevage et des Pâturages, Laboratoire de Contrôle Laitier, Direction de l'Amélioration Génétique                | Tunisia         |
| SuisseLab AG   | Switzerland   | Laboratoire d'analyse du lait de Béja, Direction Régionale de l'Office de l'Elevage et des Pâturages                        | Tunisia         |
| Qlip N.V.  | The Netherlands   | National Milk Records plc/Direct Laboratories   | United Kingdom  |
| Cattle Information Service (CIS)   | United Kingdom  |   |                 |
| Eastern Laboratory Services  | USA   |   |                 |

Attached with this report you find the certificate of your participation in the ICAR Proficiency Testing Scheme March 2016.

ICAR would like to stay at your side to support you in any way we can to help improve overall quality management systems for milk analyses. Your active participation in the ICAR PTs and in the Milk Analyses meetings is encouraging. We welcome any and all feedback/comments you may have on this activity, as it will help us continuously improve and to ultimately provide you a better service.

Kind Regards,

ICAR Secretariat



**ICAR - International Committee  
for Animal Recording**  
Via Savoia 78, ac. A, int.3,  
00191 Roma, Italy

## **ICAR Proficiency Test (PT) Chemical “Reference” methods – March 2016**

### **2. Your Performance Analysis and Control Charts**



## ICAR Proficiency Test (PT) Chemical "Reference" methods – March 2016

|                        |                        |  |  |  |  |
|------------------------|------------------------|--|--|--|--|
| <b>Laboratory Name</b> | <b>Laboratory Name</b> |  |  |  |  |
|------------------------|------------------------|--|--|--|--|

| A  | <b>Your participation Codes</b> |                              |                              |                           |                          |
|--|---------------------------------|------------------------------|------------------------------|---------------------------|--------------------------|
|  | <b>Fat<sub>ref</sub></b>        | <b>Protein<sub>ref</sub></b> | <b>Lactose<sub>ref</sub></b> | <b>Urea<sub>ref</sub></b> | <b>SCC<sub>alt</sub></b> |
| <b>Subscription</b>                          | Yes                             | Yes                          | Yes                          | Yes                       | Yes                      |
| <b>Participation Codes</b>                   |                                 |                              |                              |                           |                          |
| <b>Are all the sample results received ?</b> | Yes                             | Yes                          | Yes                          | Yes                       | Yes                      |

| B | <b>Data received on time</b> |  |  |          |            |
|---|------------------------------|--|--|----------|------------|
|   |                              |  |  | Yes      | No         |
|   |                              |  |  | Deadline | 21.03.2016 |

| C | <b>Have you sent the data with the correct units of measurements ?</b> |  |                              |                           |                          |
|---|--|--|------------------------------|---------------------------|--------------------------|
|   | <b>Fat<sub>ref</sub></b>   | <b>Protein<sup>*</sup><sub>ref</sub></b> | <b>Lactose<sub>ref</sub></b> | <b>Urea<sub>ref</sub></b> | <b>SCC<sub>alt</sub></b> |
|   | g/100g   | nitrogen g/100g                          | g/100g                       | mg/dl                     | SCC*1000/ml              |
|   |  |  |                              |                           | Yes                      |

\*It was requested to report the value in total nitrogen

| D | <b>Outliers</b>          |                              |                              |                           |                          |
|---|--------------------------|------------------------------|------------------------------|---------------------------|--------------------------|
|   | <b>Fat<sub>ref</sub></b> | <b>Protein<sub>ref</sub></b> | <b>Lactose<sub>ref</sub></b> | <b>Urea<sub>ref</sub></b> | <b>SCC<sub>alt</sub></b> |
|   | a/100g<br>sample         | a/100g<br>sample             | a/100g<br>sample             | mg/dl<br>sample           | SCC*1000/ml<br>sample    |

| E | <b>Repeatability</b>               |  |  |   |  |
|---|------------------------------------|--|--|---|--|
|   | <b>Your "r" performance</b>        |  |  | <b>Limits</b>   |  |
|   | <b>Fat<sub>ref</sub></b><br>a/100g | <b>Protein<sub>ref</sub></b><br>a/100g | <b>Lactose<sub>ref</sub></b><br>a/100g | <b>Urea<sub>ref</sub></b><br>mg/dl  | <b>SCC<sub>alt</sub></b><br>SCC*1000/ml          |
|   |                                    |  |  | ISO 1211 IDF 1D ISO 8968 / IDF 20 ISO 22662 IDF 198 ISO IDF 195 ISO 13366-2 IDF 148-2 |  |
|   |                                    |  |  | 0,043 g/100g 0,038 g/100g 0,06 g/100g 1,52 mg/dl                                      | Level r  |
|   |                                    |  |  |   | 150 25<br>300 42<br>450 51<br>750 64<br>1500 126 |

If the repeatability is smaller than the limit the cell is in green if there is a sample with a "r" bigger than the limit the cell is in red. Please check table II in correspondence of the parameter and your lab code.

| F | <b>Z-Score</b>           |                              |                              |  |                          |
|---|--------------------------|------------------------------|------------------------------|--|--------------------------|
|   | <b>Your Z-Score PT</b>   |                              |                              | <b>Interpretation Z-Score</b>                                |                          |
|   | <b>Fat<sub>ref</sub></b> | <b>Protein<sub>ref</sub></b> | <b>Lactose<sub>ref</sub></b> | <b>Urea<sub>ref</sub></b>                                    | <b>SCC<sub>alt</sub></b> |
|   |                          |                              |                              | -2<Z-Score<-2 -3<Z-Score<-2 2<Z-Score<3 Z-Score<-3 Z-Score>3 |                          |
|   |                          |                              |                              | Good Moderate Moderate Poor Poor                             |                          |

Your FIX Z-Score

|                          |                              |                              |                           |                          |
|--------------------------|------------------------------|------------------------------|---------------------------|--------------------------|
| <b>Fat<sub>ref</sub></b> | <b>Protein<sub>ref</sub></b> | <b>Lactose<sub>ref</sub></b> | <b>Urea<sub>ref</sub></b> | <b>SCC<sub>alt</sub></b> |
|--------------------------|------------------------------|------------------------------|---------------------------|--------------------------|

If there is a sample with a "z-score" in the yellow or red area please check table VI and VII in correspondence of your lab code.

| G | <b>Ranking of your lab</b>                                  |                              |                              |   |                          |
|---|---|------------------------------|------------------------------|---|--------------------------|
|   | <b>Mean difference and standard deviation of difference</b> |                              |                              | <b>Indicative Limits defined in the ICAR MA SC</b>                                    |                          |
|   | <b>Fat<sub>ref</sub></b>                                    | <b>Protein<sub>ref</sub></b> | <b>Lactose<sub>ref</sub></b> | <b>Urea<sub>ref</sub></b>   | <b>SCC<sub>alt</sub></b> |
|   |   |                              |                              | g/100g g/100g g/100g mg/dl  | SCC*1000/ml              |
|   |   |                              |                              | ISO 1211 IDF 1D ISO 8968 / IDF 20 ISO 22662 IDF 198 ISO IDF 195 ISO 13366-2 IDF 148-2 |                          |
|   |   |                              |                              | d=0,02 d=0,025 d=0,10 d=2,5 d=35  |                          |
|   |   |                              |                              | sd=0,03 sd=0,020 sd=0,10 sd=1,5 sd=35   |                          |

If d and sd are in the limit (see Table 1 and Figure 1) the cell are in green

**Legenda:**

|  |  |
|--|--|
|  | positive performance for all the sample                              |
|  | same sample showed a performance out of range or there are miss data |
|  | The parameter was not analyzed                                       |

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

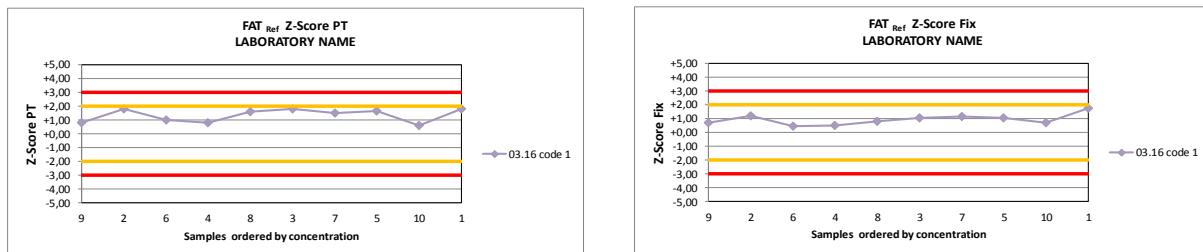
\*For this round the somatic cell ranking is not considered to evaluate the performance of the lab.

## ICAR Proficiency Test (PT) Chemical "Reference" methods – March 2016

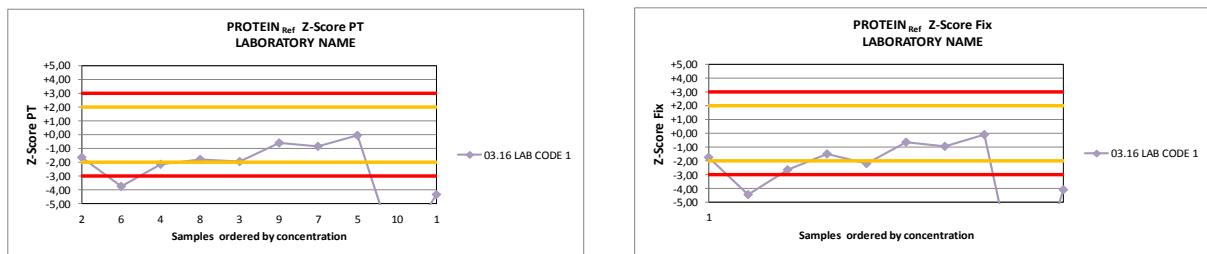
H

### Control Charts

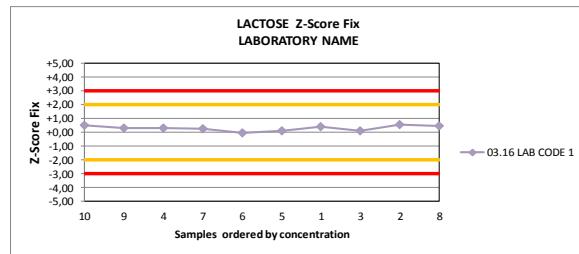
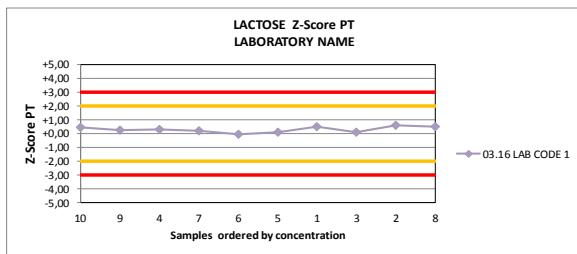
#### Control Charts Fat



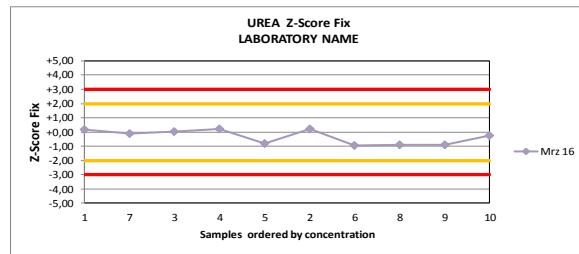
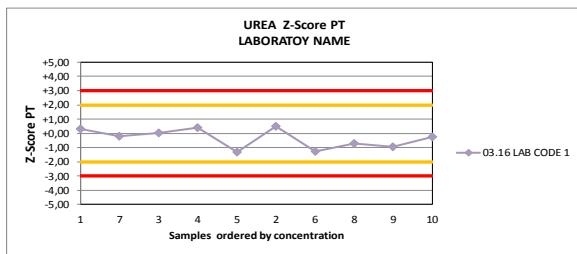
#### Control Charts Protein



## **ICAR Proficiency Test (PT) Chemical "Reference" methods – March 2016 Control Charts Lactose**

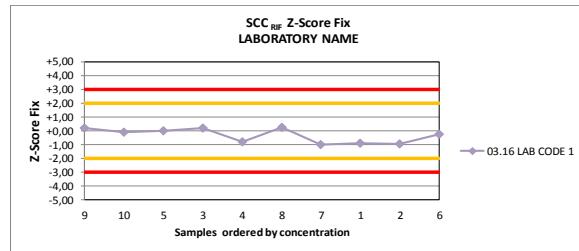
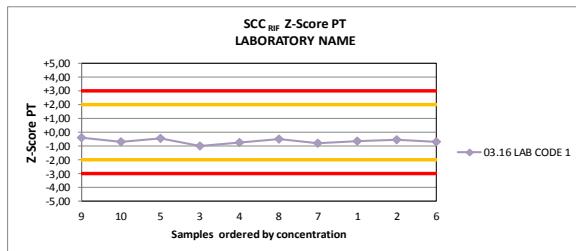


## **Control Charts Urea**



**ICAR Proficiency Test (PT) Chemical "Reference" methods – March 2016**

**Control Charts SCC**



## **ICAR Proficiency Test (PT) Chemical “Reference” methods – March 2016**

### **3. ICAR Statistical elaboration**



**ICAR**  
**PROFICIENCY TESTING SCHEME**

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**March 2016**

**Raw Milk**

**Determination of FAT CONTENT**

**Röse Gottlieb method**

Sending date of statistical treatment :      19<sup>th</sup> april 2016

|                     |  |
|---------------------|--|
| Frame of activity : | ICAR Milk Analyses Sub Committee (MA SC)   |
| Contact :           | Gavin Scott                                  gavin@milktest.co.nz                |
| ICAR Staff          | Silvia Orlandini                                  pt@icar.org    silvia@icar.org |



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**Table I : Ranking of the laboratories**      Units : g / 100 g

| Nb | %   | N° | d       | Sd    | D     | Method |
|----|-----|----|---------|-------|-------|--------|
| 1  | 7   | 10 | - 0,002 | 0,004 | 0,004 | A      |
| 2  | 13  | 9  | + 0,003 | 0,004 | 0,005 | A      |
| 3  | 20  | 2  | + 0,001 | 0,005 | 0,005 | A      |
| 4  | 27  | 13 | - 0,005 | 0,003 | 0,006 | A      |
| 5  | 33  | 14 | - 0,004 | 0,005 | 0,006 | A      |
| 6  | 40  | 3  | - 0,006 | 0,003 | 0,007 | A      |
| 7  | 47  | 11 | + 0,004 | 0,006 | 0,007 | A      |
| 8  | 53  | 15 | - 0,004 | 0,012 | 0,012 | A      |
| 9  | 60  | 7  | - 0,010 | 0,013 | 0,017 | A      |
| 10 | 67  | 12 | - 0,001 | 0,019 | 0,019 | B      |
| 11 | 73  | 1  | + 0,019 | 0,008 | 0,020 | A      |
| 12 | 80  | 5  | + 0,007 | 0,020 | 0,021 | A      |
| 13 | 87  | 6  | - 0,001 | 0,025 | 0,025 | B      |
| 14 | 93  | 4  | - 0,033 | 0,012 | 0,035 | A      |
| 15 | 100 | 8  | - 0,036 | 0,156 | 0,160 | A      |

The table should be studied in parallel with figure 1 where the laboratories are located according to an acceptability area (or target) the limits of which are :

+/- 0,020 g / 100 g for  $\bar{d}$  and 0,030 g / 100 g for Sd

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 13 laboratories using the reference method ISO 1211 / IDF 1, after outliers discarding using Grubbs test at 5% risk level,

A ISO 1211 IDF 1D Röse Gottlieb Method

B ISO 2446 / IDF 226 Gerber method

(NC : OUT of RANKING because of insufficient data number)

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

(d et Sd : mean and standard deviation of the differences (laboratory -reference))

(D : Euclidian distance to YX-axis origin = SQUARE ROOT.(d<sup>2</sup> + Sd<sup>2</sup>))

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.

Repeatability standard deviation of this ICAR proficiency test (after Cochran elimination at 5 %)

S<sub>r<sub>PT</sub></sub> 0,005

Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)

S<sub>R<sub>PT</sub></sub> 0,015

**Table II : REPEATABILITY - Absolute difference between replicates in g / 100 g**

| Sample lab<br>Code | 1       | 2     | 3     | 4       | 5       | 6     | 7       | 8     | 9     | 10    | Sr    | NL  |
|--------------------|---------|-------|-------|---------|---------|-------|---------|-------|-------|-------|-------|-----|
| 1                  | 0,022 * | 0,002 | 0,011 | 0,011   | 0,005   | 0,002 | 0,008   | 0,013 | 0,010 | 0,004 | 0,007 | 20  |
| 2                  | 0,001   | 0,004 | 0,004 | 0,005   | 0,001   | 0,000 | 0,003   | 0,004 | 0,001 | 0,002 | 0,002 | 20  |
| 3                  | 0,004   | 0,002 | 0,008 | 0,030 * | 0,000   | 0,001 | 0,009   | 0,004 | 0,004 | 0,013 | 0,008 | 20  |
| 4                  | 0,003   | 0,009 | 0,022 | 0,014   | 0,003   | 0,006 | 0,001   | 0,022 | 0,020 | 0,007 | 0,009 | 20  |
| 5                  | 0,000   | 0,020 | 0,000 | 0,000   | 0,000   | 0,000 | 0,000   | 0,010 | 0,020 | 0,000 | 0,007 | 20  |
| 6                  | 0,000   | 0,000 | 0,010 | 0,000   | 0,029 * | 0,000 | 0,029 * | 0,019 | 0,000 | 0,000 | 0,010 | 20  |
| 7                  | 0,004   | 0,005 | 0,014 | 0,006   | 0,012   | 0,007 | 0,004   | 0,016 | 0,010 | 0,024 | 0,008 | 20  |
| 8                  | 0,009   | 0,001 | 0,002 | 0,002   | 0,001   | 0,007 | 1,006 * | 0,001 | 0,008 | 0,018 | 0,225 | 20  |
| 9                  | 0,006   | 0,005 | 0,006 | 0,005   | 0,002   | 0,000 | 0,006   | 0,003 | 0,003 | 0,007 | 0,003 | 20  |
| 10                 | 0,007   | 0,011 | 0,006 | 0,008   | 0,011   | 0,003 | 0,007   | 0,003 | 0,005 | 0,000 | 0,005 | 20  |
| 11                 | 0,012   | 0,004 | 0,003 | 0,005   | 0,000   | 0,004 | 0,007   | 0,003 | 0,004 | 0,000 | 0,004 | 20  |
| 12                 | 0,000   | 0,000 | 0,000 | 0,000   | 0,000   | 0,000 | 0,000   | 0,000 | 0,000 | 0,000 | 0,000 | 20  |
| 13                 | 0,002   | 0,010 | 0,004 | 0,008   | 0,012   | 0,006 | 0,009   | 0,003 | 0,006 | 0,005 | 0,005 | 20  |
| 14                 | 0,002   | 0,008 | 0,004 | 0,003   | 0,001   | 0,002 | 0,002   | 0,001 | 0,005 | 0,005 | 0,003 | 20  |
| 15                 | 0,006   | 0,003 | 0,006 | 0,003   | 0,001   | 0,001 | 0,001   | 0,004 | 0,006 | 0,008 | 0,003 | 20  |
| Sr                 | 0,005   | 0,005 | 0,006 | 0,007   | 0,007   | 0,003 | 0,184   | 0,007 | 0,006 | 0,007 |       | 300 |
| NE                 | 30      | 30    | 30    | 30      | 30      | 30    | 30      | 30    | 30    | 30    |       |     |
| L                  | 0,014   | 0,020 | 0,023 | 0,017   | 0,015   | 0,010 | 0,014   | 0,026 | 0,024 | 0,025 |       |     |

Sr : repeatability standard deviation of each laboratory limit 0,016 g/100g

NL : number of measurements per laboratory

L : Limit for difference between duplicates according Cochran test at 5% level.

SE : repeatability standard deviation per sample

NE : number of measurements per sample

\*: discarded data using the test of Cochran at 5 %

\*\*: missing data

r : limit of repeatability, absolute difference between two replicates=0,043 according ISO 1211 IDF 1D 2010

**Table III :** Means of the replicates in g / 100 g

| Sample Lab<br>Code | 1     | 2     | 3     | 4     | 5       | 6       | 7       | 8     | 9     | 10    |
|--------------------|-------|-------|-------|-------|---------|---------|---------|-------|-------|-------|
| 1                  | 4,775 | 2,169 | 3,518 | 2,840 | 4,191   | 2,443   | 3,859   | 3,131 | 1,504 | 4,540 |
| 2                  | 4,739 | 2,140 | 3,495 | 2,831 | 4,170   | 2,431   | 3,844   | 3,115 | 1,491 | 4,537 |
| 3                  | 4,735 | 2,143 | 3,487 | 2,824 | 4,163   | 2,427   | 3,823   | 3,106 | 1,487 | 4,522 |
| 4                  | 4,708 | 2,119 | 3,468 | 2,795 | 4,114 * | 2,390 * | 3,799   | 3,105 | 1,464 | 4,490 |
| 5                  | 4,740 | 2,160 | 3,510 | 2,850 | 4,170   | 2,450   | 3,850   | 3,125 | 1,510 | 4,480 |
| 6                  | 4,786 | 2,136 | 3,500 | 2,816 | 4,151   | 2,427   | 3,821   | 3,117 | 1,456 | 4,563 |
| 7                  | 4,735 | 2,157 | 3,498 | 2,830 | 4,137   | 2,418   | 3,813   | 3,093 | 1,481 | 4,514 |
| 8                  | 4,756 | 2,156 | 3,509 | 2,837 | 4,175   | 2,450   | 3,355 * | 3,131 | 1,505 | 4,550 |
| 9                  | 4,748 | 2,151 | 3,496 | 2,830 | 4,171   | 2,433   | 3,844   | 3,116 | 1,493 | 4,533 |
| 10                 | 4,740 | 2,143 | 3,489 | 2,831 | 4,171   | 2,429   | 3,837   | 3,109 | 1,485 | 4,528 |
| 11                 | 4,751 | 2,141 | 3,503 | 2,836 | 4,182   | 2,439   | 3,841   | 3,111 | 1,484 | 4,531 |
| 12                 | 4,757 | 2,136 | 3,495 | 2,816 | 4,175   | 2,427   | 3,835   | 3,107 | 1,456 | 4,563 |
| 13                 | 4,739 | 2,140 | 3,495 | 2,821 | 4,160   | 2,426   | 3,833   | 3,108 | 1,484 | 4,525 |
| 14                 | 4,730 | 2,142 | 3,495 | 2,829 | 4,167   | 2,431   | 3,838   | 3,112 | 1,476 | 4,520 |
| 15                 | 4,724 | 2,125 | 3,485 | 2,817 | 4,163   | 2,429   | 3,838   | 3,123 | 1,504 | 4,533 |
| M                  | 4,744 | 2,144 | 3,496 | 2,827 | 4,167   | 2,433   | 3,834   | 3,114 | 1,485 | 4,528 |
| REF.               | 4,740 | 2,145 | 3,496 | 2,829 | 4,169   | 2,434   | 3,836   | 3,114 | 1,490 | 4,526 |
| SD                 | 0,019 | 0,013 | 0,012 | 0,013 | 0,013   | 0,009   | 0,016   | 0,010 | 0,017 | 0,023 |

M = mean per sample

REF. = reference values

SD = standard deviation per sample

\*: discarded data using the test of Grubbs at 5 %

**REF** : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 13 laboratories using the reference method ISO 1211 / IDF 1, after outliers discarding using Grubbs test at 5 % risk level.

**Table IV :** Outlier identification

| Sample                    | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10    |
|---------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| <b>Outliers</b>           | 1     |       |       | 3     | 6     |       | 6;8   |       |       |       |
| <b>Outlier<br/>Grubbs</b> |       |       |       |       | 4     | 4     | 8     |       |       |       |
| <b>sr</b>                 | 0,004 | 0,005 | 0,006 | 0,005 | 0,004 | 0,002 | 0,004 | 0,007 | 0,006 | 0,007 |
| <b>SR</b>                 | 0,018 | 0,014 | 0,013 | 0,014 | 0,013 | 0,010 | 0,016 | 0,011 | 0,018 | 0,023 |

**Table V :** ACCURACY - differences (laboratory - reference) in g / 100 g

| Sample lab code | 1       | 2       | 3       | 4       | 5       | 6       | 7       | 8       | 9       | 10      | d       | Sd <sub>lab</sub> | t    |
|-----------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|-------------------|------|
| 1               | + 0,035 | + 0,024 | + 0,021 | + 0,010 | + 0,021 | + 0,009 | + 0,023 | + 0,016 | + 0,014 | + 0,014 | + 0,019 | 0,008             | 7,64 |
| 2               | - 0,001 | - 0,005 | - 0,001 | + 0,001 | + 0,000 | - 0,003 | + 0,008 | + 0,001 | + 0,000 | + 0,011 | + 0,001 | 0,005             | 0,75 |
| 3               | - 0,005 | - 0,002 | - 0,009 | - 0,005 | - 0,006 | - 0,007 | - 0,013 | - 0,008 | - 0,003 | - 0,004 | - 0,006 | 0,003             | 6,07 |
| 4               | - 0,032 | - 0,027 | - 0,028 | - 0,034 | - 0,056 | - 0,044 | - 0,037 | - 0,009 | - 0,026 | - 0,036 | - 0,033 | 0,012             | 8,58 |
| 5               | + 0,000 | + 0,015 | + 0,014 | + 0,021 | + 0,001 | + 0,016 | + 0,014 | + 0,011 | + 0,020 | - 0,046 | + 0,007 | 0,020             | 1,06 |
| 6               | + 0,046 | - 0,009 | + 0,004 | - 0,013 | - 0,019 | - 0,007 | - 0,015 | + 0,002 | - 0,034 | + 0,037 | - 0,001 | 0,025             | 0,10 |
| 7               | - 0,005 | + 0,011 | + 0,002 | + 0,001 | - 0,032 | - 0,016 | - 0,023 | - 0,021 | - 0,009 | - 0,012 | - 0,010 | 0,013             | 2,51 |
| 8               | + 0,016 | + 0,010 | + 0,013 | + 0,008 | + 0,005 | + 0,016 | - 0,481 | + 0,016 | + 0,015 | + 0,024 | - 0,036 | 0,156             | 0,72 |
| 9               | + 0,008 | + 0,005 | - 0,000 | + 0,000 | + 0,002 | - 0,001 | + 0,008 | + 0,001 | + 0,002 | + 0,007 | + 0,003 | 0,004             | 2,98 |
| 10              | - 0,000 | - 0,003 | - 0,007 | + 0,002 | + 0,001 | - 0,005 | + 0,001 | - 0,006 | - 0,006 | + 0,002 | - 0,002 | 0,004             | 1,77 |
| 11              | + 0,011 | - 0,004 | + 0,006 | + 0,006 | + 0,013 | + 0,005 | + 0,005 | - 0,004 | - 0,006 | + 0,005 | + 0,004 | 0,006             | 1,86 |
| 12              | + 0,017 | - 0,009 | - 0,001 | - 0,013 | + 0,006 | - 0,007 | - 0,001 | - 0,007 | - 0,034 | + 0,037 | - 0,001 | 0,019             | 0,20 |
| 13              | - 0,001 | - 0,005 | - 0,001 | - 0,008 | - 0,009 | - 0,008 | - 0,003 | - 0,007 | - 0,006 | - 0,001 | - 0,005 | 0,003             | 5,03 |
| 14              | - 0,010 | - 0,003 | - 0,002 | - 0,001 | - 0,002 | - 0,002 | + 0,002 | - 0,003 | - 0,014 | - 0,006 | - 0,004 | 0,005             | 2,70 |
| 15              | - 0,016 | - 0,021 | - 0,011 | - 0,013 | - 0,007 | - 0,005 | + 0,002 | + 0,009 | + 0,014 | + 0,007 | - 0,004 | 0,012             | 1,11 |
| d               | + 0,004 | - 0,001 | - 0,000 | - 0,003 | - 0,002 | - 0,001 | - 0,002 | - 0,001 | - 0,005 | + 0,003 | - 0,005 | 0,042             |      |
| Sd              | 0,019   | 0,013   | 0,012   | 0,013   | 0,013   | 0,009   | 0,016   | 0,010   | 0,017   | 0,023   | 0,015   |                   |      |

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

Upper limits :  $\bar{d} = +/- 0,02 \text{ g / 100 g}$  Sd = 0,03 g / 100g

**ISO 1211 (2010) : Precision of the method :** Sr = 0,016 g / 100 g  
SR = 0,020 g / 100 g

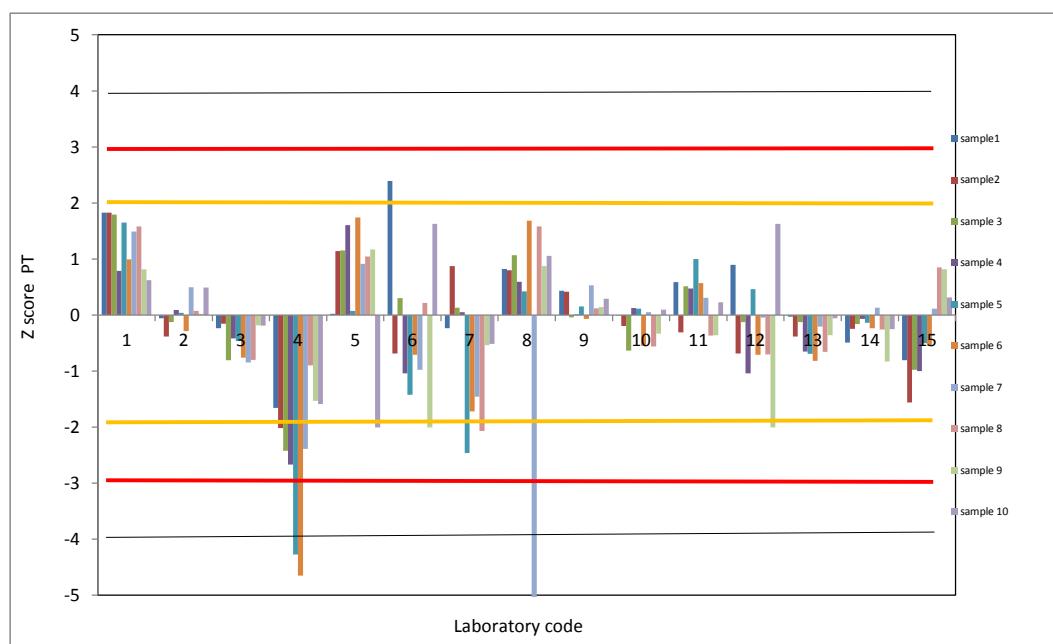
**Table VI :** Zscore of the different laboratories for each sample.  
ZS calculated on the PT standard deviation

| Sample Lab<br>Code \<br>Lab Code | 1     | 2     | 3     | 4     | 5     | 6     | 7      | 8     | 9     | 10    |
|----------------------------------|-------|-------|-------|-------|-------|-------|--------|-------|-------|-------|
| 1                                | +1,82 | +1,82 | +1,79 | +0,79 | +1,65 | +0,99 | +1,49  | +1,58 | +0,82 | +0,62 |
| 2                                | -0,06 | -0,38 | -0,12 | +0,09 | +0,04 | -0,28 | +0,50  | +0,07 | +0,02 | +0,49 |
| 3                                | -0,24 | -0,15 | -0,81 | -0,42 | -0,46 | -0,76 | -0,85  | -0,80 | -0,18 | -0,19 |
| 4                                | -1,65 | -2,02 | -2,42 | -2,67 | -4,27 | -4,65 | -2,39  | -0,90 | -1,53 | -1,59 |
| 5                                | +0,02 | +1,14 | +1,15 | +1,60 | +0,08 | +1,74 | +0,92  | +1,05 | +1,17 | -2,01 |
| 6                                | +2,39 | -0,69 | +0,30 | -1,04 | -1,42 | -0,71 | -0,98  | +0,22 | -2,00 | +1,63 |
| 7                                | -0,24 | +0,87 | +0,13 | +0,05 | -2,46 | -1,72 | -1,46  | -2,07 | -0,54 | -0,52 |
| 8                                | +0,82 | +0,80 | +1,07 | +0,59 | +0,42 | +1,69 | -30,82 | +1,58 | +0,87 | +1,06 |
| 9                                | +0,43 | +0,42 | -0,04 | +0,01 | +0,15 | -0,07 | +0,53  | +0,12 | +0,14 | +0,29 |
| 10                               | -0,01 | -0,19 | -0,64 | +0,13 | +0,11 | -0,55 | +0,05  | -0,56 | -0,33 | +0,10 |
| 11                               | +0,59 | -0,31 | +0,51 | +0,48 | +1,00 | +0,57 | +0,31  | -0,36 | -0,36 | +0,23 |
| 12                               | +0,90 | -0,69 | -0,12 | -1,04 | +0,46 | -0,71 | -0,05  | -0,70 | -2,00 | +1,63 |
| 13                               | -0,03 | -0,38 | -0,12 | -0,65 | -0,69 | -0,82 | -0,21  | -0,66 | -0,36 | -0,06 |
| 14                               | -0,49 | -0,25 | -0,16 | -0,07 | -0,14 | -0,24 | +0,13  | -0,26 | -0,83 | -0,25 |
| 15                               | -0,80 | -1,56 | -0,98 | -1,00 | -0,50 | -0,55 | +0,11  | +0,85 | +0,82 | +0,31 |

In yellow the values bigger or smaller than 2/-2

In red the values bigger or smaller than 3/-3

**Figure 2 :** Zscore of the different laboratories for each sample. ZS calculated on the PT standard deviation



**Table VII :** Zscore of the different laboratories for each sample.  
ZS calculated on the standard deviation of reproducibility of the method

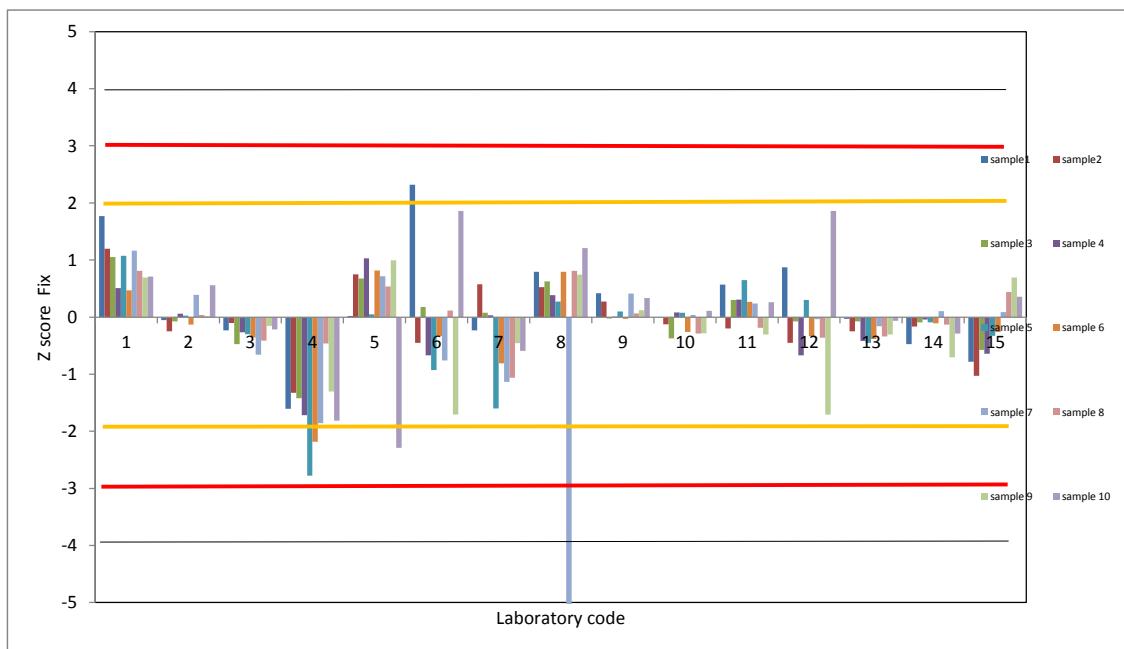
| Sample Lab code \ 1 | 1     | 2     | 3     | 4     | 5     | 6     | 7      | 8     | 9     | 10    |
|---------------------|-------|-------|-------|-------|-------|-------|--------|-------|-------|-------|
| 1                   | +1,77 | +1,20 | +1,05 | +0,51 | +1,07 | +0,47 | +1,16  | +0,81 | +0,69 | +0,71 |
| 2                   | -0,05 | -0,25 | -0,07 | +0,06 | +0,02 | -0,13 | +0,39  | +0,04 | +0,02 | +0,56 |
| 3                   | -0,23 | -0,10 | -0,47 | -0,27 | -0,30 | -0,36 | -0,66  | -0,41 | -0,16 | -0,22 |
| 4                   | -1,60 | -1,33 | -1,42 | -1,72 | -2,78 | -2,18 | -1,86  | -0,46 | -1,31 | -1,82 |
| 5                   | +0,02 | +0,75 | +0,68 | +1,03 | +0,05 | +0,82 | +0,71  | +0,54 | +0,99 | -2,29 |
| 6                   | +2,32 | -0,45 | +0,18 | -0,67 | -0,93 | -0,33 | -0,76  | +0,11 | -1,71 | +1,86 |
| 7                   | -0,23 | +0,57 | +0,08 | +0,03 | -1,60 | -0,81 | -1,14  | -1,06 | -0,46 | -0,59 |
| 8                   | +0,80 | +0,52 | +0,63 | +0,38 | +0,27 | +0,79 | -24,04 | +0,81 | +0,74 | +1,21 |
| 9                   | +0,42 | +0,27 | -0,02 | +0,01 | +0,10 | -0,03 | +0,41  | +0,06 | +0,12 | +0,33 |
| 10                  | -0,00 | -0,13 | -0,37 | +0,08 | +0,07 | -0,26 | +0,04  | -0,29 | -0,28 | +0,11 |
| 11                  | +0,57 | -0,20 | +0,30 | +0,31 | +0,65 | +0,27 | +0,24  | -0,19 | -0,31 | +0,26 |
| 12                  | +0,87 | -0,45 | -0,07 | -0,67 | +0,30 | -0,33 | -0,04  | -0,36 | -1,71 | +1,86 |
| 13                  | -0,03 | -0,25 | -0,07 | -0,42 | -0,45 | -0,38 | -0,16  | -0,34 | -0,31 | -0,07 |
| 14                  | -0,48 | -0,16 | -0,09 | -0,04 | -0,09 | -0,11 | +0,10  | -0,13 | -0,70 | -0,29 |
| 15                  | -0,78 | -1,03 | -0,57 | -0,64 | -0,33 | -0,26 | +0,09  | +0,44 | +0,69 | +0,36 |

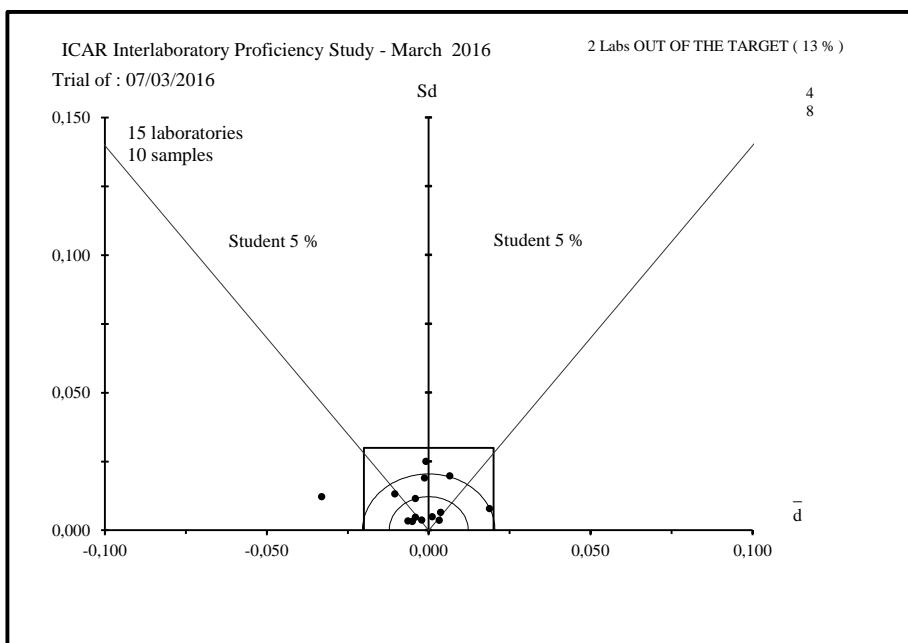
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=0,02

In yellow the values bigger or smaller than 2/-2

In red the values bigger or smaller than 3/-3

**Figure 3 :**  
Zscore of the different laboratories for each sample. ZS calculated on the standard deviation of reproducibility of the method





**Figure 1 :** ACCURACY - Evaluation of the individual performances (to see table I).

#### LIST OF THE PARTICIPANTS ICAR

ICAR PROFICIENCY TEST  
RAW MILK  
FAT CONTENT Röse Göttlieb  
March 2016

| Name   | City                 | Country    |
|--|----------------------|------------|
| AIA-LAB. STANDARD LATTE                      | MACARESE (ROMA)      | ITALY      |
| CENTRAL MILK LABORATORY - ICBA               | CAESAREA             | ISRAEL     |
| DEPARTEMENT QUALITE CRA-W                    | GEMBLOUX             | BELGIUM    |
| EASTERN LAB SERVICES                         | MEDINA               | USA        |
| ESTONIAN LIVESTOCK PERFORMANCE RECORDING LTD | TARTU                | ESTONIA    |
| ILVO - T.V.                                  | MELLE                | BELGIUM    |
| JAPAN DAIRY TECHNICAL ASSOCIATION            | TOKYO                | JAPAN      |
| LAB AGROAL DE SANTANDER                      | SANTANDER            | SPAIN      |
| LOM KCHZ LABORATORIM REFERENCYJNE            | PRUSZKOW             | POLAND     |
| MILCHPRUFRING BADEN-WURTTEMBERG E.V.         | KIRCHEIM / TECK      | GERMANY    |
| QLIP N.V.                                    | CM ZUTPHEN           | NETHERLAND |
| TEAGASC FOOD RESEARCH CENTER                 | FERMOY CO-CORK       | IRELAND    |
| VALACTA                                      | STE ANNE DE BELLEVUE | CANADA     |



**ICAR  
PROFICIENCY TESTING SCHEME**

-----

**March 2016**

**Raw Milk**

**Determination of CRUDE PROTEIN CONTENT  
KJELDAHL Method**

Sending date of statistical treatment : 19<sup>th</sup> April 2016

|                     |   |
|---------------------|---|
| Frame of activity : | ICAR Milk Analyses Sub Committee (MA SC)        |
| Contact :           | Gavin Scott<br>gavin@milktest.co.nz             |
| ICAR Staff          | Silvia Orlandini<br>pt@icar.org silvia@icar.org |



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**Table I : Ranking of the laboratories**      Units : g / 100 g

| Nb | %   | N° | d       | Sd    | D     |
|----|-----|----|---------|-------|-------|
| 1  | 7   | 9  | - 0,000 | 0,007 | 0,007 |
| 2  | 13  | 2  | + 0,001 | 0,008 | 0,008 |
| 3  | 20  | 6  | - 0,001 | 0,012 | 0,012 |
| 4  | 27  | 15 | - 0,004 | 0,011 | 0,012 |
| 5  | 33  | 4  | - 0,014 | 0,005 | 0,015 |
| 6  | 40  | 12 | - 0,014 | 0,007 | 0,015 |
| 7  | 47  | 10 | + 0,014 | 0,007 | 0,016 |
| 8  | 53  | 8  | - 0,016 | 0,006 | 0,017 |
| 9  | 60  | 11 | + 0,015 | 0,007 | 0,017 |
| 10 | 67  | 14 | + 0,013 | 0,017 | 0,021 |
| 11 | 73  | 13 | + 0,019 | 0,011 | 0,022 |
| 12 | 80  | 3  | + 0,022 | 0,010 | 0,025 |
| 13 | 87  | 5  | + 0,020 | 0,029 | 0,035 |
| 14 | 93  | 7  | - 0,037 | 0,013 | 0,039 |
| 15 | 100 | 1  | - 0,057 | 0,069 | 0,089 |

The table should be studied in parallel with figure 1 where the laboratories are located according to an acceptability area (or target) the limits of which are :

+/- 0,025 g / 100 g for d and 0,020 g / 100 g for Sd

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 15 laboratories using the reference method (ISO 8968 / IDF 20), after outlier discarding using Grubbs test at 5 % risk level

(NC : OUT of RANKING because of insufficient data number)

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

(d et Sd : mean and standard deviation of the differences (laboratory -reference))

(D : Euclidian distance to YX-axis origin = SQUARE ROOT.(d<sup>2</sup> + Sd<sup>2</sup>))

**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.

Repeatability standard deviation of this ICAR proficiency test (after Cochran elimination at 5 %)

S<sub>r</sub><sub>PT</sub> 0,009

Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)

S<sub>R</sub><sub>PT</sub> 0,021

**Table II : REPEATABILITY - Absolute difference between replicates in g / 100 g**

| Sample Lab code | 1       | 2       | 3       | 4     | 5     | 6       | 7     | 8     | 9     | 10      | Sr    | NL  |
|-----------------|---------|---------|---------|-------|-------|---------|-------|-------|-------|---------|-------|-----|
| 1               | 0,001   | 0,001   | 0,001   | 0,001 | 0,001 | 0,000   | 0,000 | 0,001 | 0,001 | 0,450 * | 0,101 | 20  |
| 2               | 0,002   | 0,001   | 0,013   | 0,002 | 0,001 | 0,011   | 0,013 | 0,003 | 0,003 | 0,030   | 0,008 | 20  |
| 3               | 0,038 * | 0,006   | 0,006   | 0,013 | 0,019 | 0,006   | 0,006 | 0,006 | 0,006 | 0,000   | 0,011 | 20  |
| 4               | 0,007   | 0,008   | 0,010   | 0,001 | 0,008 | 0,001   | 0,001 | 0,001 | 0,011 | 0,006   | 0,005 | 20  |
| 5               | 0,014   | 0,012   | 0,073 * | 0,004 | 0,034 | 0,043 * | 0,007 | 0,015 | 0,024 | 0,035   | 0,023 | 20  |
| 6               | 0,010   | 0,006   | 0,023   | 0,010 | 0,044 | 0,006   | 0,016 | 0,001 | 0,037 | 0,017   | 0,015 | 20  |
| 7               | 0,017   | 0,006   | 0,025   | 0,003 | 0,011 | 0,010   | 0,014 | 0,019 | 0,012 | 0,010   | 0,010 | 20  |
| 8               | 0,027   | 0,029 * | 0,029   | 0,009 | 0,014 | 0,014   | 0,004 | 0,004 | 0,008 | 0,009   | 0,012 | 20  |
| 9               | 0,008   | 0,002   | 0,003   | 0,001 | 0,001 | 0,001   | 0,003 | 0,002 | 0,014 | 0,000   | 0,004 | 20  |
| 10              | 0,000   | 0,006   | 0,004   | 0,004 | 0,003 | 0,005   | 0,003 | 0,001 | 0,012 | 0,001   | 0,004 | 20  |
| 11              | 0,001   | 0,002   | 0,005   | 0,008 | 0,011 | 0,012   | 0,009 | 0,014 | 0,011 | 0,011   | 0,007 | 20  |
| 12              | 0,005   | 0,003   | 0,007   | 0,010 | 0,006 | 0,000   | 0,001 | 0,001 | 0,003 | 0,007   | 0,004 | 20  |
| 13              | 0,005   | 0,005   | 0,018   | 0,003 | 0,010 | 0,000   | 0,023 | 0,021 | 0,018 | 0,005   | 0,009 | 20  |
| 14              | 0,013   | 0,010   | 0,011   | 0,003 | 0,001 | 0,013   | 0,013 | 0,008 | 0,006 | 0,040   | 0,011 | 20  |
| 15              | 0,003   | 0,003   | 0,015   | 0,001 | 0,004 | 0,029 * | 0,016 | 0,001 | 0,002 | 0,004   | 0,008 | 20  |
| Sr              | 0,010   | 0,007   | 0,017   | 0,004 | 0,012 | 0,011   | 0,008 | 0,007 | 0,010 | 0,083   |       | 300 |
| NE              | 30      | 30      | 30      | 30    | 30    | 30      | 30    | 30    | 30    | 30      |       |     |
| L               | 0,028   | 0,016   | 0,039   | 0,016 | 0,044 | 0,021   | 0,029 | 0,025 | 0,039 | 0,047   |       |     |

Sr : repeatability standard deviation of each laboratory limit 0,014 g /100g

NL : number of measurements per laboratory

L : Limit for difference between duplicates according Cochran test at 5% level.

SE : repeatability standard deviation per sample

NE : number of measurements per sample

\*: discarded data using the test of Cochran at 5 %

\*\* : missing data

r : limit of repeatability, absolute difference between two replicates=0,038 according ISO 8968 / IDF 20

**Table III :** Means of the replicates in g / 100 g

| Sample Lab code | 1       | 2     | 3     | 4     | 5     | 6       | 7     | 8       | 9     | 10      |
|-----------------|---------|-------|-------|-------|-------|---------|-------|---------|-------|---------|
| 1               | 3,652 * | 2,678 | 3,210 | 2,843 | 3,534 | 2,721 * | 3,415 | 3,054   | 3,253 | 3,390 * |
| 2               | 3,735   | 2,712 | 3,252 | 2,899 | 3,542 | 2,793   | 3,438 | 3,070   | 3,267 | 3,618   |
| 3               | 3,726   | 2,734 | 3,270 | 2,928 | 3,570 | 2,823   | 3,448 | 3,097   | 3,283 | 3,656   |
| 4               | 3,714   | 2,691 | 3,244 | 2,881 | 3,521 | 2,783   | 3,417 | 3,056   | 3,251 | 3,613   |
| 5               | 3,734   | 2,739 | 3,257 | 2,880 | 3,555 | 2,852   | 3,444 | 3,159 * | 3,244 | 3,651   |
| 6               | 3,724   | 2,724 | 3,249 | 2,900 | 3,508 | 2,802   | 3,423 | 3,080   | 3,260 | 3,636   |
| 7               | 3,687   | 2,675 | 3,204 | 2,859 | 3,494 | 2,765   | 3,387 | 3,074   | 3,220 | 3,577   |
| 8               | 3,711   | 2,699 | 3,238 | 2,868 | 3,513 | 2,785   | 3,416 | 3,073   | 3,248 | 3,603   |
| 9               | 3,735   | 2,704 | 3,249 | 2,890 | 3,526 | 2,796   | 3,439 | 3,072   | 3,275 | 3,623   |
| 10              | 3,744   | 2,718 | 3,255 | 2,919 | 3,543 | 2,806   | 3,454 | 3,093   | 3,277 | 3,642   |
| 11              | 3,740   | 2,723 | 3,268 | 2,895 | 3,558 | 2,814   | 3,457 | 3,083   | 3,283 | 3,646   |
| 12              | 3,710   | 2,704 | 3,231 | 2,881 | 3,520 | 2,789   | 3,411 | 3,076   | 3,252 | 3,602   |
| 13              | 3,749   | 2,723 | 3,276 | 2,902 | 3,560 | 2,818   | 3,461 | 3,100   | 3,293 | 3,624   |
| 14              | 3,727   | 2,707 | 3,253 | 2,899 | 3,544 | 2,807   | 3,440 | 3,098   | 3,275 | 3,687   |
| 15              | 3,706   | 2,696 | 3,245 | 2,901 | 3,529 | 2,788   | 3,417 | 3,091   | 3,265 | 3,635   |
| M               | 3,724   | 2,708 | 3,247 | 2,890 | 3,534 | 2,802   | 3,431 | 3,080   | 3,263 | 3,629   |
| REF.            | 3,725   | 2,709 | 3,248 | 2,890 | 3,535 | 2,800   | 3,432 | 3,080   | 3,264 | 3,629   |
| SD              | 0,017   | 0,019 | 0,020 | 0,022 | 0,021 | 0,021   | 0,021 | 0,015   | 0,019 | 0,027   |

M = mean per sample

REF. = reference values

SD = standard deviation per sample

\*: discarded data using the test of Grubbs 5 %

**REF** : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528,  
of 15 laboratories using the reference method ISO 8968 / IDF 20, after outliers discarding using Grubbs test at 5 % risk level.

**Table IV :** Outlier identification

| Sample                  | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10    |
|-------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| <b>Outliers Cochran</b> | 3     | 8     | 5     |       |       | 5;15  |       |       |       | 1     |
| <b>Outlier Grubbs</b>   | 1     |       |       |       |       | 1     |       | 5     |       | 1     |
| <b>sr</b>               | 0,008 | 0,004 | 0,011 | 0,004 | 0,012 | 0,006 | 0,008 | 0,006 | 0,010 | 0,013 |
| <b>SR</b>               | 0,019 | 0,020 | 0,022 | 0,022 | 0,023 | 0,017 | 0,021 | 0,016 | 0,020 | 0,029 |

**Table V :** ACCURACY - differences (laboratory - reference) in g / 100 g

| Sample Lab<br>code | 1       | 2       | 3       | 4       | 5       | 6       | 7       | 8       | 9       | 10      | d       | Sd <sub>lab</sub> | t    |
|--------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|-------------------|------|
| 1                  | - 0,074 | - 0,031 | - 0,039 | - 0,048 | - 0,001 | - 0,079 | - 0,017 | - 0,026 | - 0,011 | - 0,239 | - 0,057 | 0,069             | 2,60 |
| 2                  | + 0,010 | + 0,004 | + 0,003 | + 0,009 | + 0,007 | - 0,007 | + 0,006 | - 0,010 | + 0,003 | - 0,011 | + 0,001 | 0,008             | 0,53 |
| 3                  | + 0,001 | + 0,025 | + 0,021 | + 0,038 | + 0,035 | + 0,023 | + 0,016 | + 0,018 | + 0,019 | + 0,027 | + 0,022 | 0,010             | 6,75 |
| 4                  | - 0,012 | - 0,018 | - 0,004 | - 0,010 | - 0,014 | - 0,018 | - 0,016 | - 0,024 | - 0,013 | - 0,016 | - 0,014 | 0,005             | 8,57 |
| 5                  | + 0,008 | + 0,030 | + 0,008 | - 0,011 | + 0,020 | + 0,052 | + 0,012 | + 0,079 | - 0,020 | + 0,022 | + 0,020 | 0,029             | 2,22 |
| 6                  | - 0,001 | + 0,015 | + 0,001 | + 0,010 | - 0,027 | + 0,002 | - 0,009 | + 0,000 | - 0,004 | + 0,007 | - 0,001 | 0,012             | 0,20 |
| 7                  | - 0,038 | - 0,033 | - 0,045 | - 0,032 | - 0,040 | - 0,036 | - 0,045 | - 0,006 | - 0,044 | - 0,052 | - 0,037 | 0,013             | 9,30 |
| 8                  | - 0,014 | - 0,010 | - 0,010 | - 0,022 | - 0,022 | - 0,015 | - 0,016 | - 0,007 | - 0,016 | - 0,026 | - 0,016 | 0,006             | 8,17 |
| 9                  | + 0,010 | - 0,004 | + 0,000 | - 0,001 | - 0,009 | - 0,004 | + 0,007 | - 0,008 | + 0,011 | - 0,006 | - 0,000 | 0,007             | 0,16 |
| 10                 | + 0,019 | + 0,010 | + 0,007 | + 0,029 | + 0,008 | + 0,006 | + 0,022 | + 0,013 | + 0,013 | + 0,013 | + 0,014 | 0,007             | 5,91 |
| 11                 | + 0,015 | + 0,014 | + 0,020 | + 0,004 | + 0,024 | + 0,014 | + 0,025 | + 0,003 | + 0,019 | + 0,017 | + 0,015 | 0,007             | 6,72 |
| 12                 | - 0,015 | - 0,005 | - 0,018 | - 0,009 | - 0,015 | - 0,011 | - 0,021 | - 0,004 | - 0,012 | - 0,027 | - 0,014 | 0,007             | 6,04 |
| 13                 | + 0,024 | + 0,014 | + 0,028 | + 0,011 | + 0,026 | + 0,018 | + 0,029 | + 0,020 | + 0,029 | - 0,005 | + 0,019 | 0,011             | 5,84 |
| 14                 | + 0,001 | - 0,001 | + 0,004 | + 0,009 | + 0,010 | + 0,006 | + 0,008 | + 0,018 | + 0,012 | + 0,058 | + 0,013 | 0,017             | 2,35 |
| 15                 | - 0,020 | - 0,012 | - 0,004 | + 0,011 | - 0,006 | - 0,013 | - 0,015 | + 0,012 | + 0,001 | + 0,006 | - 0,004 | 0,011             | 1,14 |
| d                  | - 0,001 | - 0,000 | - 0,002 | - 0,001 | - 0,000 | + 0,001 | - 0,001 | - 0,000 | - 0,001 | + 0,000 | - 0,003 | 0,030             |      |
| Sd                 | 0,017   | 0,019   | 0,020   | 0,022   | 0,021   | 0,021   | 0,021   | 0,015   | 0,019   | 0,027   | 0,021   |                   |      |

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

Upper limits :  $\bar{d} = +/- 0,025 \text{ g / 100 g}$       Sd = 0,020 g / 100 g

**ISO 8968 / IDF 20 : Precision of the method :**      Sr = 0,014 g / 100 g  
SR = 0,018 g / 100 g

**Table VI :** Zscore of the different laboratories for each sample.  
ZS calculated on the PT standard deviation

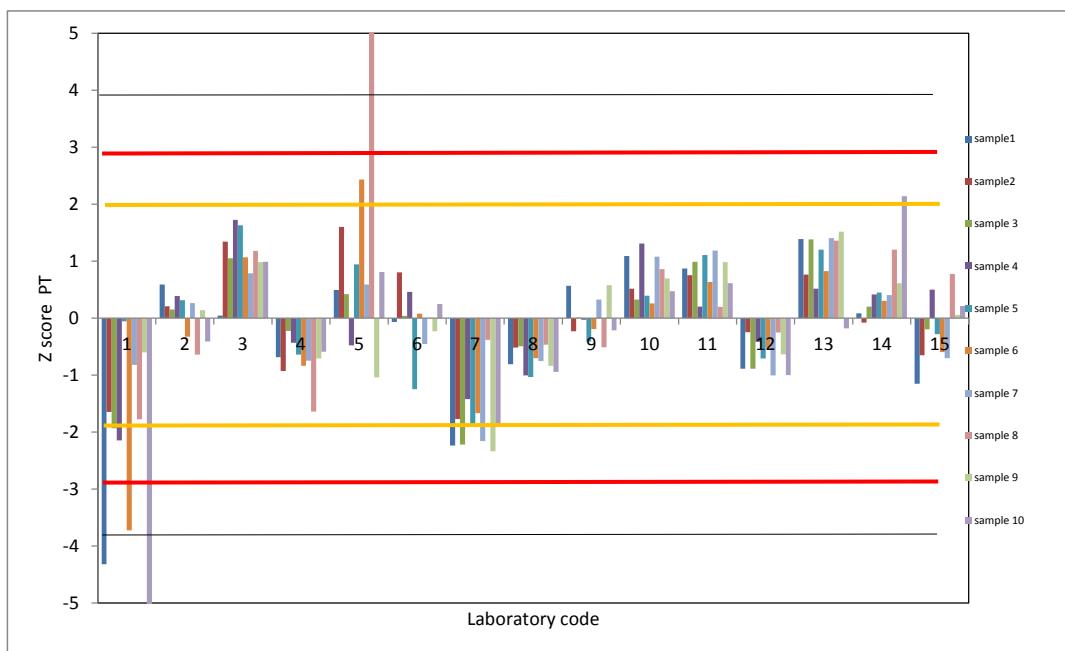
| Sample Lab code | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10    |
|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 1               | -4,32 | -1,65 | -1,93 | -2,15 | -0,06 | -3,72 | -0,82 | -1,77 | -0,60 | -8,80 |
| 2               | +0,59 | +0,21 | +0,15 | +0,39 | +0,32 | -0,32 | +0,27 | -0,64 | +0,14 | -0,41 |
| 3               | +0,04 | +1,34 | +1,05 | +1,72 | +1,63 | +1,07 | +0,79 | +1,18 | +0,98 | +0,99 |
| 4               | -0,68 | -0,93 | -0,22 | -0,44 | -0,64 | -0,84 | -0,75 | -1,64 | -0,71 | -0,59 |
| 5               | +0,49 | +1,60 | +0,42 | -0,48 | +0,94 | +2,43 | +0,59 | +5,29 | -1,04 | +0,81 |
| 6               | -0,07 | +0,80 | +0,04 | +0,46 | -1,25 | +0,08 | -0,46 | +0,00 | -0,23 | +0,25 |
| 7               | -2,24 | -1,77 | -2,22 | -1,42 | -1,89 | -1,67 | -2,16 | -0,38 | -2,34 | -1,90 |
| 8               | -0,81 | -0,51 | -0,50 | -1,01 | -1,03 | -0,70 | -0,75 | -0,47 | -0,84 | -0,94 |
| 9               | +0,57 | -0,23 | +0,01 | -0,03 | -0,41 | -0,19 | +0,33 | -0,51 | +0,58 | -0,21 |
| 10              | +1,09 | +0,51 | +0,33 | +1,31 | +0,39 | +0,26 | +1,08 | +0,86 | +0,70 | +0,47 |
| 11              | +0,87 | +0,75 | +0,99 | +0,20 | +1,11 | +0,63 | +1,19 | +0,20 | +0,98 | +0,61 |
| 12              | -0,89 | -0,25 | -0,89 | -0,42 | -0,71 | -0,52 | -1,01 | -0,25 | -0,64 | -1,00 |
| 13              | +1,39 | +0,76 | +1,38 | +0,52 | +1,20 | +0,82 | +1,40 | +1,36 | +1,52 | -0,18 |
| 14              | +0,08 | -0,08 | +0,20 | +0,42 | +0,45 | +0,30 | +0,40 | +1,20 | +0,61 | +2,14 |
| 15              | -1,15 | -0,65 | -0,20 | +0,50 | -0,28 | -0,59 | -0,70 | +0,77 | +0,06 | +0,21 |

In yellow the values bigger or smaller than 2/-2

In red the values bigger or smaller than 3/-3

**Figure 2 :**

Zscore of the different laboratories for each sample. ZS calculated on the PT standard deviation



**Table VII :** Zscore of the different laboratories for each sample.  
ZS calculated on the standard deviation of reproducibility of the method

| Sample Lab code | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10     |
|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|--------|
| 1               | -4,09 | -1,72 | -2,17 | -2,65 | -0,07 | -4,41 | -0,95 | -1,47 | -0,63 | -13,28 |
| 2               | +0,56 | +0,22 | +0,17 | +0,48 | +0,38 | -0,38 | +0,31 | -0,53 | +0,15 | -0,62  |
| 3               | +0,04 | +1,41 | +1,18 | +2,13 | +1,94 | +1,26 | +0,91 | +0,98 | +1,03 | +1,49  |
| 4               | -0,65 | -0,97 | -0,25 | -0,54 | -0,76 | -0,99 | -0,86 | -1,36 | -0,74 | -0,89  |
| 5               | +0,47 | +1,67 | +0,47 | -0,59 | +1,12 | +2,88 | +0,68 | +4,38 | -1,09 | +1,22  |
| 6               | -0,06 | +0,84 | +0,05 | +0,57 | -1,48 | +0,09 | -0,53 | +0,00 | -0,24 | +0,37  |
| 7               | -2,12 | -1,85 | -2,49 | -1,76 | -2,25 | -1,98 | -2,49 | -0,32 | -2,46 | -2,87  |
| 8               | -0,77 | -0,54 | -0,56 | -1,24 | -1,23 | -0,83 | -0,87 | -0,39 | -0,88 | -1,42  |
| 9               | +0,54 | -0,24 | +0,01 | -0,04 | -0,49 | -0,22 | +0,38 | -0,42 | +0,61 | -0,32  |
| 10              | +1,03 | +0,54 | +0,37 | +1,61 | +0,47 | +0,31 | +1,25 | +0,71 | +0,73 | +0,71  |
| 11              | +0,82 | +0,79 | +1,11 | +0,25 | +1,32 | +0,75 | +1,37 | +0,16 | +1,03 | +0,92  |
| 12              | -0,84 | -0,26 | -1,00 | -0,52 | -0,85 | -0,61 | -1,16 | -0,21 | -0,67 | -1,51  |
| 13              | +1,31 | +0,80 | +1,55 | +0,64 | +1,43 | +0,97 | +1,62 | +1,13 | +1,59 | -0,27  |
| 14              | +0,08 | -0,08 | +0,22 | +0,51 | +0,54 | +0,36 | +0,47 | +0,99 | +0,64 | +3,22  |
| 15              | -1,09 | -0,68 | -0,22 | +0,62 | -0,33 | -0,70 | -0,81 | +0,64 | +0,06 | +0,32  |

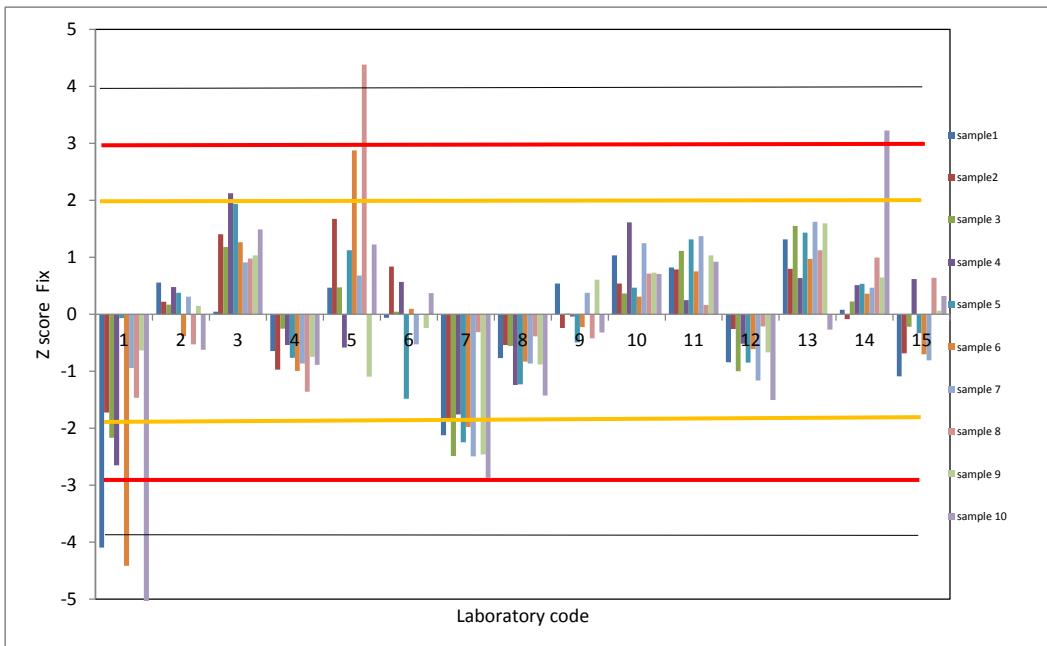
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=0,018

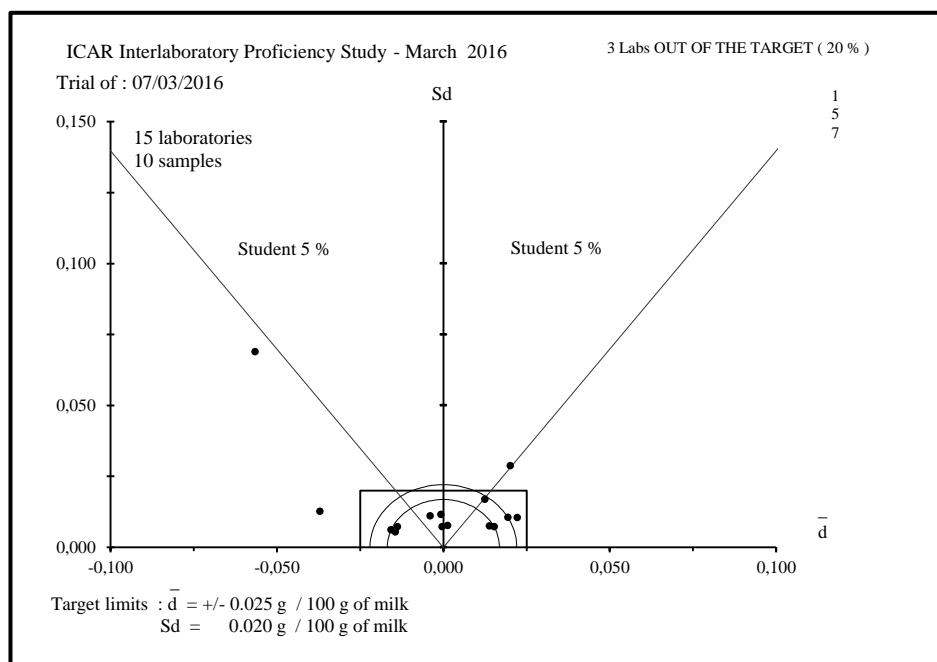
In yellow the values bigger or smaller than 2/-2

In red the values bigger or smaller than 3/-3

**Figure 3 :**

Zscore of the different laboratories for each sample. ZS calculated on the standard deviation of reproducibility of the method





**Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).**

LIST OF THE PARTICIPANTS ICAR

ICAR PROFICIENCY TEST  
RAW MILK  
NITROGEN CONTENT  
March 2016

| Name   | City                 | Country    |
|--|----------------------|------------|
| ACTALIA-Cecalait                             | POLIGNY              | FRANCE     |
| AIA-LAB. STANDARD LATTE                      | MACCARESE (ROMA)     | ITALY      |
| CENTRAL MILK LABORATORY - ICBA               | CAESAREA             | ISRAEL     |
| DEPARTEMENT QUALITE CRA-W                    | GEMBLOUX             | BELGIUM    |
| EASTERN LAB SERVICES                         | MEDINA               | USA        |
| ESTONIAN LIVESTOCK PERFORMANCE RECORDING LTD | TARTU                | ESTONIA    |
| ILVO - T.V.                                  | MELLE                | BELGIUM    |
| JAPAN DAIRY TECHNICAL ASSOCIATION            | TOKYO                | JAPAN      |
| LAB AGROAL DE SANTANDER                      | SANTANDER            | SPAIN      |
| LOM KCHZ LABORATORIM REFERENCYJNE            | PRUSZKOW             | POLAND     |
| MILCHPRUFRING BADEN-WURTTEMBERG E.V.         | KIRCHEIM / TECK      | GERMANY    |
| QLIP N.V.                                    | CM ZUTPHEN           | NETHERLAND |
| TEAGASC FOOD RESEARCH CENTER                 | FERMOY CO-CORK       | IRELAND    |
| VALACTA                                      | STE ANNE DE BELLEVUE | CANADA     |



**ICAR  
PROFICIENCY TESTING SCHEME**

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**March 2016**

**Raw Milk**

**Determination of LACTOSE CONTENT**

Sending date of statistical treatment : 19<sup>th</sup> april 2016

|                     |   |
|---------------------|---|
| Frame of activity : | ICAR Milk Analyses Sub Committee (MA SC)  |
| Contact :           | Gavin Scott<br>Silvia Orlandini   |
| ICAR Staff          | <a href="mailto:gavin@milktest.co.nz">gavin@milktest.co.nz</a><br><a href="mailto:pt@icar.org">pt@icar.org</a> <a href="mailto:silvia@icar.org">silvia@icar.org</a> |

**Table I : Ranking of the laboratories**      Units : g / 100 g

| Nb | %   | N° | d       | Sd    | D     | Method                   |
|----|-----|----|---------|-------|-------|--------------------------|
| 1  | 8   | 2  | - 0,009 | 0,012 | 0,015 | Polarimeter              |
| 2  | 17  | 1  | + 0,013 | 0,009 | 0,016 | Internal chemical method |
| 3  | 25  | 7  | + 0,008 | 0,021 | 0,023 | Gravimetric              |
| 4  | 33  | 6  | + 0,030 | 0,009 | 0,031 | HPLC                     |
| 5  | 42  | 5  | + 0,032 | 0,018 | 0,037 | HPLC                     |
| 6  | 50  | 10 | - 0,035 | 0,016 | 0,038 | Infra red                |
| 7  | 58  | 9  | - 0,036 | 0,019 | 0,040 | Lane-Eynon               |
| 8  | 67  | 8  | + 0,005 | 0,041 | 0,041 | diff.pH                  |
| 9  | 75  | 12 | + 0,048 | 0,034 | 0,059 | HPLC                     |
| 10 | 83  | 4  | - 0,044 | 0,041 | 0,060 | diff.pH                  |
| 11 | 92  | 3  | - 0,071 | 0,024 | 0,075 | Enzymatic                |
| 12 | 100 | 11 | + 0,100 | 0,055 | 0,114 | Continuous flow          |

The table should be studied in parallel with figure 1 where the laboratories are located according to an acceptability area (or target) the limits of which are :

—  
+/- 0.100 g / 100 g for d and 0.100 g / 100g for Sd

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 12 laboratories , after outliers discarding using Grubbs test at 5 % risk level.

(NC : OUT of RANKING because of insufficient data number)

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

(d et Sd : mean and standard deviation of the differences (laboratory -reference))

(D : Euclidian distance to YX-axis origin = SQUARE ROOT.(d<sup>2</sup> + Sd<sup>2</sup>))

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.

Repeatability standard deviation of this ICAR proficiency test (after Cochran elimination at 5 %)

S<sub>r<sub>PT</sub></sub> 0,010

Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)

S<sub>R<sub>PT</sub></sub> 0,047

**Table II : REPEATABILITY - Absolute difference between replicates in g / 100 g**

| Sample Lab<br>code | 1       | 2       | 3       | 4     | 5       | 6     | 7       | 8       | 9       | 10      | Sr    | NL  |
|--------------------|---------|---------|---------|-------|---------|-------|---------|---------|---------|---------|-------|-----|
| 1                  | 0,001   | 0,004   | 0,008   | 0,002 | 0,006   | 0,003 | 0,003   | 0,004   | 0,003   | 0,004   | 0,003 | 20  |
| 2                  | 0,000   | 0,010   | 0,000   | 0,010 | 0,000   | 0,010 | 0,010   | 0,000   | 0,000   | 0,000   | 0,004 | 20  |
| 3                  | 0,018   | 0,000   | 0,018   | 0,019 | 0,018   | 0,018 | 0,019   | 0,000   | 0,000   | 0,000   | 0,010 | 20  |
| 4                  | 0,014   | 0,004   | 0,016   | 0,012 | 0,011   | 0,001 | 0,003   | 0,006   | 0,017   | 0,004   | 0,007 | 20  |
| 5                  | 0,015   | 0,009   | 0,025   | 0,011 | 0,016   | 0,015 | 0,003   | 0,009   | 0,024   | 0,036 * | 0,013 | 20  |
| 6                  | 0,008   | 0,005   | 0,003   | 0,004 | 0,006   | 0,001 | 0,008   | 0,002   | 0,012   | 0,013   | 0,005 | 20  |
| 7                  | 0,001   | 0,006   | 0,002   | 0,000 | 0,001   | 0,000 | 0,010   | 0,002   | 0,010   | 0,007   | 0,004 | 20  |
| 8                  | 0,028   | 0,013   | 0,006   | 0,022 | 0,079 * | 0,052 | 0,043 * | 0,047 * | 0,061 * | 0,001   | 0,030 | 20  |
| 9                  | 0,002   | 0,003   | 0,019   | 0,000 | 0,005   | 0,002 | 0,004   | 0,005   | 0,006   | 0,003   | 0,005 | 20  |
| 10                 | 0,000   | 0,000   | 0,000   | 0,000 | 0,000   | 0,020 | 0,000   | 0,000   | 0,000   | 0,000   | 0,004 | 20  |
| 11                 | 0,060 * | 0,030 * | 0,090 * | 0,030 | 0,010   | 0,030 | 0,010   | 0,030 * | 0,030   | 0,020   | 0,029 | 20  |
| 12                 | 0,008   | 0,004   | 0,007   | 0,005 | 0,001   | 0,021 | 0,007   | 0,006   | 0,032   | 0,020   | 0,010 | 20  |
| Sr                 | 0,015   | 0,008   | 0,020   | 0,009 | 0,017   | 0,015 | 0,011   | 0,012   | 0,017   | 0,010   |       | 240 |
| NE                 | 24      | 24      | 24      | 24    | 24      | 24    | 24      | 24      | 24      | 24      |       |     |
| L                  | 0,031   | 0,016   | 0,031   | 0,034 | 0,023   | 0,053 | 0,022   | 0,011   | 0,042   | 0,025   |       |     |

Sr : repeatability standard deviation of each laboratory limit 0,022 g/100g

NL : number of measurements per laboratory

L : Limit for difference between duplicates according Cochran test at 5% level.

SE : repeatability standard deviation per sample

NE : number of measurements per sample

\*: discarded data using the test of Cochran at 5 %

\*\*: missing data

r : limit of repeatability, absolute difference between two replicates=0,061 according ISO 22662 / IDF 198

**Table III :** Means of the replicates in g / 100 g

| Sample Lab code | 1       | 2       | 3     | 4     | 5       | 6     | 7     | 8     | 9     | 10    |
|-----------------|---------|---------|-------|-------|---------|-------|-------|-------|-------|-------|
| 1               | 4,910   | 5,051   | 4,975 | 4,776 | 4,841   | 4,820 | 4,784 | 5,110 | 4,742 | 4,665 |
| 2               | 4,880   | 5,025   | 4,970 | 4,755 | 4,840   | 4,795 | 4,765 | 5,090 | 4,720 | 4,610 |
| 3               | 4,848   | 4,960   | 4,870 | 4,662 | 4,765   | 4,758 | 4,695 | 4,990 | 4,671 | 4,607 |
| 4               | 4,817   | 4,997   | 4,946 | 4,755 | 4,845   | 4,826 | 4,725 | 5,031 | 4,631 | 4,530 |
| 5               | 4,951   | 5,090   | 5,010 | 4,796 | 4,854   | 4,847 | 4,793 | 5,120 | 4,739 | 4,664 |
| 6               | 4,916   | 5,053   | 4,988 | 4,790 | 4,862   | 4,843 | 4,808 | 5,131 | 4,771 | 4,674 |
| 7               | 4,914   | 5,064   | 4,982 | 4,746 | 4,862   | 4,800 | 4,771 | 5,121 | 4,731 | 4,632 |
| 8               | 4,873   | 4,983   | 4,941 | 4,762 | 4,855   | 4,917 | 4,746 | 5,131 | 4,744 | 4,638 |
| 9               | 4,875   | 4,993   | 4,955 | 4,738 | 4,789   | 4,751 | 4,760 | 5,038 | 4,689 | 4,596 |
| 10              | 4,870   | 5,010   | 4,940 | 4,710 | 4,800   | 4,770 | 4,720 | 5,080 | 4,680 | 4,610 |
| 11              | 5,060 * | 5,205 * | 5,075 | 4,835 | 5,005 * | 4,865 | 4,865 | 5,115 | 4,795 | 4,720 |
| 12              | 4,925   | 5,061   | 5,001 | 4,797 | 4,876   | 4,889 | 4,847 | 5,072 | 4,830 | 4,726 |
| M               | 4,889   | 5,026   | 4,971 | 4,760 | 4,835   | 4,823 | 4,773 | 5,086 | 4,728 | 4,639 |
| REF.            | 4,890   | 5,026   | 4,971 | 4,762 | 4,837   | 4,822 | 4,772 | 5,088 | 4,728 | 4,641 |
| SD              | 0,038   | 0,041   | 0,049 | 0,045 | 0,035   | 0,052 | 0,050 | 0,045 | 0,056 | 0,055 |

M = mean per sample

REF. = reference values

SD = standard deviation per sample

\*: discarded data using the test of Grubbs 5 %

**REF** : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528,  
of 12 laboratories, after outliers discarding using Grubbs test at 5 % risk level.

**Table IV :** Outlier identification

| Sample                  | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10    |
|-------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| <b>Outliers Cochran</b> | 11    | 11    | 11    |       | 8     |       | 8     | 8;11  | 8     | 5     |
| <b>Outlier Grubbs</b>   | 11    | 11    |       |       | 11    |       |       |       |       |       |
| <b>sr</b>               | 0,009 | 0,005 | 0,009 | 0,009 | 0,006 | 0,015 | 0,006 | 0,003 | 0,012 | 0,007 |
| <b>SR</b>               | 0,039 | 0,041 | 0,039 | 0,046 | 0,037 | 0,053 | 0,052 | 0,046 | 0,059 | 0,057 |

**Table V :** ACCURACY - differences (laboratory - reference) in g / 100 g

| Sample Lab<br>code | 1       | 2       | 3       | 4       | 5       | 6       | 7       | 8       | 9       | 10      | d       | Sd <sub>lab</sub> | t     |
|--------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|-------------------|-------|
| 1                  | + 0,020 | + 0,025 | + 0,004 | + 0,014 | + 0,004 | - 0,003 | + 0,011 | + 0,022 | + 0,013 | + 0,024 | + 0,013 | 0,009             | 4,53  |
| 2                  | - 0,010 | - 0,001 | - 0,001 | - 0,007 | + 0,003 | - 0,027 | - 0,007 | + 0,002 | - 0,008 | - 0,031 | - 0,009 | 0,012             | 2,36  |
| 3                  | - 0,042 | - 0,066 | - 0,101 | - 0,101 | - 0,072 | - 0,064 | - 0,078 | - 0,098 | - 0,057 | - 0,034 | - 0,071 | 0,024             | 9,55  |
| 4                  | - 0,073 | - 0,029 | - 0,025 | - 0,007 | + 0,008 | + 0,003 | - 0,048 | - 0,057 | - 0,098 | - 0,111 | - 0,044 | 0,041             | 3,35  |
| 5                  | + 0,061 | + 0,064 | + 0,039 | + 0,033 | + 0,017 | + 0,024 | + 0,020 | + 0,031 | + 0,011 | + 0,023 | + 0,032 | 0,018             | 5,78  |
| 6                  | + 0,026 | + 0,027 | + 0,017 | + 0,028 | + 0,025 | + 0,020 | + 0,036 | + 0,043 | + 0,043 | + 0,032 | + 0,030 | 0,009             | 10,66 |
| 7                  | + 0,024 | + 0,038 | + 0,011 | - 0,016 | + 0,025 | - 0,022 | - 0,001 | + 0,033 | + 0,003 | - 0,010 | + 0,008 | 0,021             | 1,26  |
| 8                  | - 0,017 | - 0,043 | - 0,030 | - 0,000 | + 0,018 | + 0,095 | - 0,027 | + 0,042 | + 0,015 | - 0,004 | + 0,005 | 0,041             | 0,38  |
| 9                  | - 0,015 | - 0,033 | - 0,016 | - 0,024 | - 0,048 | - 0,071 | - 0,012 | - 0,051 | - 0,039 | - 0,046 | - 0,036 | 0,019             | 5,91  |
| 10                 | - 0,020 | - 0,016 | - 0,031 | - 0,052 | - 0,037 | - 0,052 | - 0,052 | - 0,008 | - 0,048 | - 0,031 | - 0,035 | 0,016             | 6,70  |
| 11                 | + 0,170 | + 0,179 | + 0,104 | + 0,073 | + 0,168 | + 0,043 | + 0,093 | + 0,027 | + 0,067 | + 0,079 | + 0,100 | 0,055             | 5,80  |
| 12                 | + 0,035 | + 0,035 | + 0,030 | + 0,034 | + 0,039 | + 0,066 | + 0,074 | - 0,016 | + 0,102 | + 0,085 | + 0,048 | 0,034             | 4,52  |
| d                  | - 0,001 | - 0,000 | + 0,000 | - 0,002 | - 0,002 | + 0,001 | + 0,001 | - 0,002 | + 0,000 | - 0,002 | + 0,004 | 0,052             |       |
| Sd                 | 0,038   | 0,041   | 0,049   | 0,045   | 0,035   | 0,052   | 0,050   | 0,045   | 0,056   | 0,055   | 0,047   |                   |       |

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

Upper limits :  $\bar{d} = +/- 0.100 \text{ g} / 100\text{g}$  Sd = 0.100 g / 100g**ISO 22662 / IDF 198 : Precision of the method :**

Sr = 0.022 g / 100 g

SR = 0.047 g / 100 g

**Table VI :** Zscore of the different laboratories for each sample.  
ZS calculated on the PT standard deviation

| Sample Lab code | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10    |
|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 1               | +0,51 | +0,62 | +0,09 | +0,30 | +0,12 | -0,06 | +0,22 | +0,48 | +0,24 | +0,43 |
| 2               | -0,26 | -0,02 | -0,01 | -0,16 | +0,09 | -0,53 | -0,14 | +0,04 | -0,15 | -0,57 |
| 3               | -1,10 | -1,62 | -2,05 | -2,24 | -2,05 | -1,25 | -1,54 | -2,16 | -1,03 | -0,62 |
| 4               | -1,91 | -0,71 | -0,50 | -0,16 | +0,22 | +0,06 | -0,95 | -1,26 | -1,76 | -2,03 |
| 5               | +1,59 | +1,57 | +0,79 | +0,73 | +0,49 | +0,47 | +0,40 | +0,69 | +0,20 | +0,41 |
| 6               | +0,68 | +0,65 | +0,34 | +0,61 | +0,72 | +0,39 | +0,71 | +0,95 | +0,77 | +0,59 |
| 7               | +0,62 | +0,94 | +0,23 | -0,36 | +0,71 | -0,43 | -0,02 | +0,73 | +0,05 | -0,18 |
| 8               | -0,44 | -1,07 | -0,60 | -0,01 | +0,51 | +1,83 | -0,53 | +0,93 | +0,28 | -0,07 |
| 9               | -0,39 | -0,82 | -0,33 | -0,54 | -1,38 | -1,38 | -0,24 | -1,12 | -0,70 | -0,83 |
| 10              | -0,52 | -0,39 | -0,62 | -1,16 | -1,05 | -1,01 | -1,04 | -0,18 | -0,87 | -0,57 |
| 11              | +4,45 | +4,41 | +2,13 | +1,61 | +4,81 | +0,82 | +1,84 | +0,59 | +1,20 | +1,43 |
| 12              | +0,92 | +0,86 | +0,61 | +0,76 | +1,11 | +1,28 | +1,48 | -0,36 | +1,83 | +1,54 |

In yellow the values bigger or smaller than 2/-2

In red the values bigger or smaller than 3/-3

**Figure 2 :**

Zscore of the different laboratories for each sample. ZS calculated on the PT standard deviation



**Table VII :** Zscore of the different laboratories for each sample.  
ZS calculated on the standard deviation of reproducibility of the method

| Sample Lab code | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10    |
|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 1               | +0,42 | +0,53 | +0,09 | +0,29 | +0,09 | -0,06 | +0,24 | +0,47 | +0,29 | +0,51 |
| 2               | -0,21 | -0,02 | -0,01 | -0,16 | +0,07 | -0,58 | -0,15 | +0,04 | -0,17 | -0,66 |
| 3               | -0,89 | -1,40 | -2,14 | -2,15 | -1,53 | -1,37 | -1,65 | -2,09 | -1,21 | -0,73 |
| 4               | -1,55 | -0,62 | -0,52 | -0,16 | +0,16 | +0,06 | -1,01 | -1,22 | -2,08 | -2,37 |
| 5               | +1,29 | +1,35 | +0,83 | +0,70 | +0,37 | +0,51 | +0,43 | +0,67 | +0,23 | +0,48 |
| 6               | +0,56 | +0,57 | +0,36 | +0,59 | +0,54 | +0,43 | +0,76 | +0,91 | +0,91 | +0,69 |
| 7               | +0,50 | +0,81 | +0,24 | -0,35 | +0,53 | -0,48 | -0,03 | +0,70 | +0,06 | -0,21 |
| 8               | -0,36 | -0,92 | -0,63 | -0,01 | +0,38 | +2,01 | -0,57 | +0,90 | +0,33 | -0,08 |
| 9               | -0,32 | -0,71 | -0,34 | -0,52 | -1,03 | -1,52 | -0,26 | -1,08 | -0,83 | -0,97 |
| 10              | -0,42 | -0,34 | -0,65 | -1,11 | -0,78 | -1,12 | -1,11 | -0,17 | -1,02 | -0,66 |
| 11              | +3,62 | +3,81 | +2,22 | +1,54 | +3,58 | +0,91 | +1,97 | +0,57 | +1,42 | +1,68 |
| 12              | +0,75 | +0,75 | +0,64 | +0,73 | +0,82 | +1,41 | +1,58 | -0,34 | +2,17 | +1,80 |

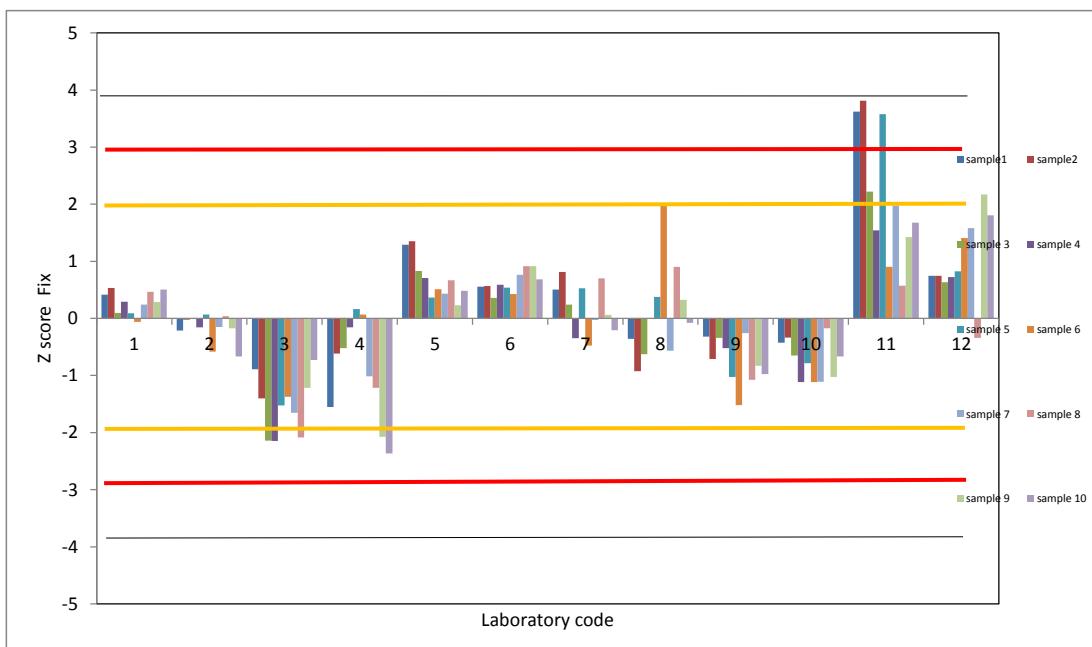
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=0,047

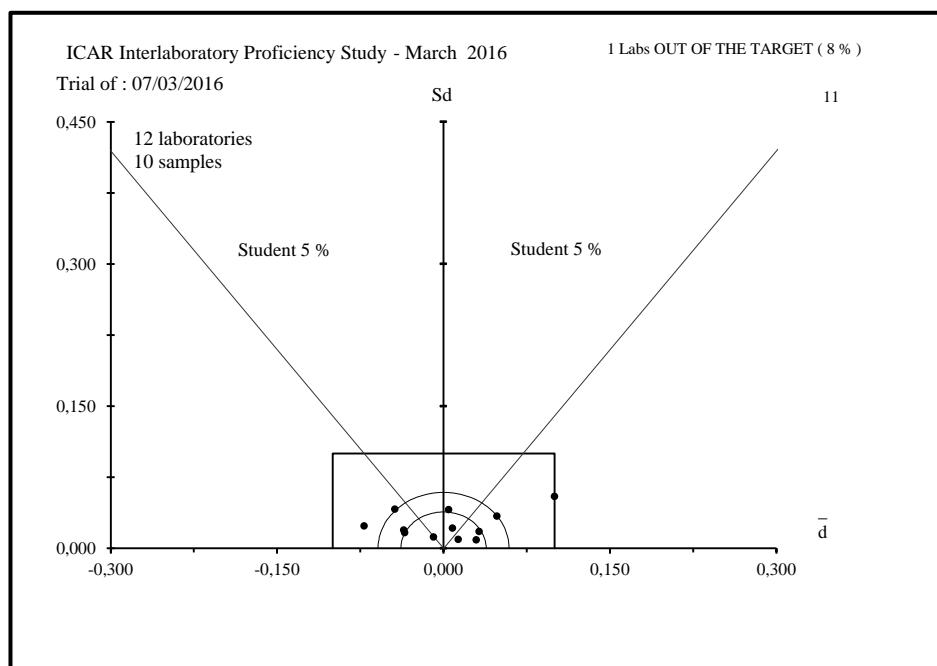
In yellow the values bigger or smaller than 2/-2

In red the values bigger or smaller than 3/-3

**Figure 3 :**

Zscore of the different laboratories for each sample. ZS calculated on the standard deviation of reproducibility of the method





**Figure 1 :** ACCURACY - Evaluation of the individual performances (to see table I).

#### LIST OF THE PARTICIPANTS ICAR

ICAR PROFICIENCY TEST  
RAW MILK  
LACTOSE CONTENT  
March 2016

| Name   | City                 | Country      |
|--|----------------------|--------------|
| AIA-LAB. STANDARD LATTE                      | MACARESE (ROMA)      | ITALY        |
| DEPARTEMENT QUALITE CRA-W                    | GEMBLOUX             | BELGIUM      |
| ESTONIAN LIVESTOCK PERFORMANCE RECORDING LTD | TARTU                | ESTONIA      |
| ILVO - T.V.                                  | MELLE                | BELGIUM      |
| JAPAN DAIRY TECHNICAL ASSOCIATION            | TOKYO                | JAPAN        |
| LAB AGROAL DE SANTANDER                      | SANTANDER            | SPAIN        |
| LACTOLAB                                     | IRENE                | SOUTH AFRICA |
| LOM KCHZ LABORATORIM REFERENCYJNE            | PRUSZKOW             | POLAND       |
| MILCHPRUFRING BADEN-WURTTEMBERG E.V.         | KIRCHEIM / TECK      | GERMANY      |
| QLIP N.V.                                    | CM ZUTPHEN           | NETHERLAND   |
| TEAGASC FOOD RESEARCH CENTER                 | FERMOY CO-CORK       | IRELAND      |
| VALACTA                                      | STE ANNE DE BELLEVUE | CANADA       |



**ICAR**  
**PROFICIENCY TESTING SCHEME**

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**March 2016**

**Raw Milk**

**Determination of UREA CONTENT**

Sending date of statistical treatment :      19<sup>th</sup> april 2016

|                     |  |
|---------------------|--|
| Frame of activity : | ICAR Milk Analyses Sub Committee (MA SC)                                 |
| Contact :           | Gavin Scott                          gavin@milktest.co.nz                |
| ICAR Staff          | Silvia Orlandini                          pt@icar.org    silvia@icar.org |



ACCRÉDITATION  
N° 1-2473  
PORTÉE  
DISPONIBLE SUR  
[WWW.COFRAC.FR](http://WWW.COFRAC.FR)

**Table I : Ranking of the laboratories**Units : mg / 100 g

| Nb | %   | N° | d      | Sd   | D    | Method          |
|----|-----|----|--------|------|------|-----------------|
| 1  | 9   | 3  | - 0,10 | 0,50 | 0,51 | ChemSpec        |
| 2  | 18  | 7  | - 0,40 | 0,33 | 0,52 | enzymatic       |
| 3  | 27  | 9  | - 0,30 | 0,45 | 0,54 | diff.pH         |
| 4  | 36  | 11 | - 0,31 | 0,65 | 0,72 | diff.pH         |
| 5  | 45  | 8  | + 0,32 | 0,83 | 0,89 | diff.pH         |
| 6  | 55  | 1  | - 0,59 | 0,92 | 1,09 | diff.pH         |
| 7  | 64  | 10 | - 0,92 | 0,79 | 1,21 | Continuous flow |
| 8  | 73  | 2  | + 0,89 | 1,54 | 1,78 | diff.pH         |
| 9  | 82  | 6  | + 1,59 | 0,85 | 1,81 | Continuous flow |
| 10 | 91  | 5  | - 1,89 | 1,06 | 2,17 | diff.pH         |
| 11 | 100 | 4  | + 1,96 | 1,24 | 2,32 | diff.pH         |

The table should be studied in parallel with figure 1 where the laboratories are located according to an acceptability area (or target) the limits of which are :

+/- 2,50 mg / 100 g for  $\bar{d}$  and 1,50 mg / 100 g for Sd

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 7 laboratories using reference method (ISO 14637/IDF 195), after outlier discarding using Grubbs test at 5% risk level

(NC : OUT of RANKING because of insufficient data number)

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

(d et Sd : mean and standard deviation of the differences (laboratory -reference))

(D : Euclidian distance to YX-axis origin = SQUARE ROOT.(d<sup>2</sup> + Sd<sup>2</sup>))

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.

Repeatability standard deviation of this ICAR proficiency test (after Cochran elimination at 5 %)

S<sub>r<sub>PT</sub></sub> 0,39

Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)

S<sub>R<sub>PT</sub></sub> 1,32

**Table II : REPEATABILITY - Absolute difference between replicates in mg / 100 g**

| Sample lab<br>Code | 1    | 2    | 3    | 4    | 5    | 6    | 7    | 8      | 9    | 10     | Sr   | NL  |
|--------------------|------|------|------|------|------|------|------|--------|------|--------|------|-----|
| 1                  | 0,00 | 0,21 | 0,21 | 0,21 | 0,00 | 0,21 | 0,00 | 0,21   | 0,21 | 0,21   | 0,13 | 20  |
| 2                  | 0,10 | 0,00 | 0,20 | 0,30 | 0,30 | 1,00 | 0,00 | 1,90 * | 0,50 | 0,00   | 0,50 | 20  |
| 3                  | 0,30 | 0,20 | 0,00 | 0,20 | 0,10 | 0,30 | 0,10 | 0,30   | 0,30 | 0,10   | 0,15 | 20  |
| 4                  | 0,50 | 1,70 | 0,10 | 0,40 | 0,40 | 0,00 | 0,10 | 0,70   | 0,80 | 0,00   | 0,48 | 20  |
| 5                  | 0,20 | 0,10 | 0,40 | 0,00 | 0,20 | 0,40 | 0,50 | 0,30   | 0,80 | 0,50   | 0,29 | 20  |
| 6                  | 0,10 | 0,40 | 0,70 | 0,50 | 2,00 | 1,30 | 0,60 | 0,40   | 0,30 | 1,00 * | 0,64 | 20  |
| 7                  | 0,10 | 0,20 | 0,00 | 0,40 | 1,10 | 1,00 | 0,70 | 0,20   | 0,50 | 0,30   | 0,41 | 20  |
| 8                  | 0,00 | 0,90 | 0,30 | 0,20 | 0,40 | 0,40 | 0,80 | 0,70   | 0,10 | 0,10   | 0,35 | 20  |
| 9                  | 0,10 | 0,20 | 0,60 | 0,20 | 1,10 | 0,20 | 0,70 | 0,20   | 0,60 | 0,40   | 0,37 | 20  |
| 10                 | 0,29 | 1,07 | 0,58 | 0,10 | 0,39 | 0,78 | 0,19 | 0,19   | 0,10 | 0,19   | 0,35 | 20  |
| 11                 | 0,00 | 0,00 | 0,30 | 0,50 | 1,10 | 1,00 | 0,40 | 0,90   | 1,40 | 0,30   | 0,53 | 20  |
| Sr                 | 0,15 | 0,49 | 0,27 | 0,22 | 0,61 | 0,51 | 0,33 | 0,52   | 0,44 | 0,28   |      | 220 |
| NE                 | 22   | 22   | 22   | 22   | 22   | 22   | 22   | 22     | 22   | 22     |      |     |
| L                  | 0,54 | 1,72 | 0,96 | 0,79 | 2,17 | 1,82 | 1,18 | 1,18   | 1,57 | 0,65   |      |     |

Sr : repeatability standard deviation of each laboratory limit 0,54 mg/100g

NL : number of measurements per laboratory

L : Limit for difference between duplicates according Cochran test at 5% level.

SE : repeatability standard deviation per sample

NE : number of measurements per sample

\*: discarded data using the test of Cochran at 5 %

\*\* : missing data

r : limit of repeatability, absolute difference between two replicates=1,50 according ISO 14637 / IDF 195

**Table III :** Means of the replicates in mg / 100 g

| Sample lab<br>Code | 1     | 2       | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10    |
|--------------------|-------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| 1                  | 19,47 | 42,48   | 29,00 | 33,49 | 36,17 | 45,48 | 23,97 | 50,40 | 54,68 | 60,88 |
| 2                  | 19,45 | 42,00   | 28,90 | 33,15 | 38,25 | 48,30 | 25,10 | 57,15 | 56,95 | 61,60 |
| 3                  | 17,85 | 42,50   | 28,60 | 33,00 | 37,25 | 47,45 | 24,15 | 52,15 | 56,65 | 61,35 |
| 4                  | 19,45 | 43,05   | 29,95 | 34,40 | 39,40 | 49,90 | 25,35 | 54,75 | 60,30 | 65,00 |
| 5                  | 18,70 | 41,25   | 27,80 | 31,70 | 35,80 | 44,90 | 22,85 | 48,75 | 53,50 | 57,75 |
| 6                  | 20,55 | 45,80 * | 30,85 | 34,95 | 39,10 | 47,85 | 25,20 | 53,70 | 57,35 | 62,50 |
| 7                  | 18,65 | 42,00   | 27,80 | 32,80 | 37,15 | 47,20 | 23,55 | 51,60 | 56,05 | 61,15 |
| 8                  | 18,50 | 42,05   | 29,45 | 34,10 | 38,20 | 48,30 | 23,80 | 50,95 | 57,95 | 61,85 |
| 9                  | 18,35 | 41,30   | 28,70 | 32,60 | 37,95 | 46,80 | 23,85 | 51,50 | 56,00 | 61,90 |
| 10                 | 17,14 | 40,15   | 27,57 | 32,57 | 37,18 | 46,41 | 22,72 | 50,97 | 56,75 | 61,26 |
| 11                 | 20,10 | 42,30   | 28,85 | 32,45 | 37,45 | 46,90 | 24,20 | 51,15 | 55,20 | 60,25 |
| M                  | 18,93 | 41,91   | 28,86 | 33,20 | 37,63 | 47,23 | 24,07 | 52,10 | 56,49 | 61,41 |
| REF.               | 19,15 | 42,06   | 28,96 | 33,13 | 37,60 | 47,23 | 24,16 | 52,01 | 56,34 | 61,30 |
| SD                 | 0,99  | 0,82    | 0,98  | 0,96  | 1,10  | 1,39  | 0,88  | 2,31  | 1,79  | 1,72  |

M = mean per sample

REF. = reference values

SD = standard deviation per sample

\*: discarded data using the test of Grubbs 5 %

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528,

of 7 laboratories using the reference method ISO 14637 / IDF 195, after outliers discarding using Grubbs test 5% risk level

**Table IV :** Outlier identification

| Sample          | 1    | 2    | 3    | 4    | 5    | 6    | 7    | 8    | 9    | 10   |
|-----------------|------|------|------|------|------|------|------|------|------|------|
| <b>Outliers</b> |      |      |      |      |      |      |      |      |      |      |
| <b>Cochran</b>  |      |      |      |      |      |      |      | 2    |      | 6    |
| <b>Outlier</b>  |      |      |      |      |      |      |      |      |      |      |
| <b>Grubbs</b>   |      | 6    |      |      |      |      |      |      |      |      |
| <b>sr</b>       | 0,15 | 0,50 | 0,27 | 0,22 | 0,61 | 0,51 | 0,33 | 0,34 | 0,44 | 0,19 |
| <b>SR</b>       | 1,00 | 0,89 | 0,99 | 0,97 | 1,19 | 1,44 | 0,91 | 1,69 | 1,82 | 1,78 |

**Table V :** ACCURACY - differences (laboratory - reference) in mg / 100 g

| Sample lab<br>code | 1      | 2      | 3      | 4      | 5      | 6      | 7      | 8      | 9      | 10     | d      | Sd <sub>lab</sub> | t    |
|--------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|-------------------|------|
| 1                  | + 0,33 | + 0,42 | + 0,04 | + 0,36 | - 1,44 | - 1,75 | - 0,19 | - 1,61 | - 1,66 | - 0,41 | - 0,59 | 0,92              | 2,04 |
| 2                  | + 0,30 | - 0,06 | - 0,06 | + 0,02 | + 0,65 | + 1,08 | + 0,94 | + 5,14 | + 0,61 | + 0,30 | + 0,89 | 1,54              | 1,83 |
| 3                  | - 1,30 | + 0,44 | - 0,36 | - 0,13 | - 0,35 | + 0,23 | - 0,01 | + 0,14 | + 0,31 | + 0,05 | - 0,10 | 0,50              | 0,62 |
| 4                  | + 0,30 | + 0,99 | + 0,99 | + 1,27 | + 1,80 | + 2,68 | + 1,19 | + 2,74 | + 3,96 | + 3,70 | + 1,96 | 1,24              | 5,00 |
| 5                  | - 0,45 | - 0,81 | - 1,16 | - 1,43 | - 1,80 | - 2,33 | - 1,31 | - 3,26 | - 2,84 | - 3,55 | - 1,89 | 1,06              | 5,66 |
| 6                  | + 1,40 | + 3,74 | + 1,89 | + 1,82 | + 1,50 | + 0,63 | + 1,04 | + 1,69 | + 1,01 | + 1,20 | + 1,59 | 0,85              | 5,91 |
| 7                  | - 0,50 | - 0,06 | - 1,16 | - 0,33 | - 0,45 | - 0,02 | - 0,61 | - 0,41 | - 0,29 | - 0,15 | - 0,40 | 0,33              | 3,83 |
| 8                  | - 0,65 | - 0,01 | + 0,49 | + 0,97 | + 0,60 | + 1,08 | - 0,36 | - 1,06 | + 1,61 | + 0,55 | + 0,32 | 0,83              | 1,22 |
| 9                  | - 0,80 | - 0,76 | - 0,26 | - 0,53 | + 0,35 | - 0,43 | - 0,31 | - 0,51 | - 0,34 | + 0,60 | - 0,30 | 0,45              | 2,10 |
| 10                 | - 2,01 | - 1,92 | - 1,39 | - 0,55 | - 0,42 | - 0,82 | - 1,44 | - 1,04 | + 0,41 | - 0,03 | - 0,92 | 0,79              | 3,68 |
| 11                 | + 0,95 | + 0,24 | - 0,11 | - 0,68 | - 0,15 | - 0,33 | + 0,04 | - 0,86 | - 1,14 | - 1,05 | - 0,31 | 0,65              | 1,51 |
| d                  | - 0,22 | - 0,15 | - 0,10 | + 0,07 | + 0,03 | + 0,00 | - 0,09 | + 0,09 | + 0,15 | + 0,11 | + 0,02 | 1,37              |      |
| Sd                 | 0,99   | 0,82   | 0,98   | 0,96   | 1,10   | 1,39   | 0,88   | 2,31   | 1,79   | 1,72   | 1,38   |                   |      |

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

Upper limits :  $\bar{d} = +/- 2,50 \text{ mg / 100 g}$  Sd = 1,50 mg / 100 g**ISO 14637 / IDF 195 : Precision of the method :** Sr = 0,54 mg / 100 g

SR = 1,81 mg / 100 g

**Table VI :** Zscore of the different laboratories for each sample.  
ZS calculated on the PT standard deviation

| Sample Lab<br>Code \<br>Lab Code | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10    |
|----------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 1                                | +0,33 | +0,51 | +0,04 | +0,38 | -1,30 | -1,26 | -0,22 | -0,70 | -0,93 | -0,24 |
| 2                                | +0,31 | -0,07 | -0,06 | +0,02 | +0,59 | +0,77 | +1,07 | +2,23 | +0,34 | +0,18 |
| 3                                | -1,30 | +0,53 | -0,37 | -0,13 | -0,32 | +0,16 | -0,01 | +0,06 | +0,17 | +0,03 |
| 4                                | +0,31 | +1,20 | +1,02 | +1,33 | +1,63 | +1,92 | +1,35 | +1,19 | +2,21 | +2,15 |
| 5                                | -0,45 | -0,99 | -1,19 | -1,49 | -1,63 | -1,67 | -1,49 | -1,41 | -1,59 | -2,06 |
| 6                                | +1,41 | +4,55 | +1,94 | +1,90 | +1,36 | +0,45 | +1,18 | +0,73 | +0,57 | +0,70 |
| 7                                | -0,50 | -0,07 | -1,19 | -0,34 | -0,41 | -0,02 | -0,69 | -0,18 | -0,16 | -0,09 |
| 8                                | -0,65 | -0,01 | +0,50 | +1,02 | +0,54 | +0,77 | -0,41 | -0,46 | +0,90 | +0,32 |
| 9                                | -0,80 | -0,93 | -0,27 | -0,55 | +0,31 | -0,31 | -0,35 | -0,22 | -0,19 | +0,35 |
| 10                               | -2,02 | -2,33 | -1,42 | -0,58 | -0,38 | -0,59 | -1,64 | -0,45 | +0,23 | -0,02 |
| 11                               | +0,96 | +0,29 | -0,11 | -0,71 | -0,14 | -0,23 | +0,05 | -0,37 | -0,64 | -0,61 |

In yellow the values bigger or smaller than 2/-2

In red the values bigger or smaller than 3/-3

**Figure 2 :** Zscore of the different laboratories for each sample. ZS calculated on the PT standard deviation



**Table VII :** Zscore of the different laboratories for each sample.  
ZS calculated on the standard deviation of reproducibility of the method

| Sample Lab code | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10    |
|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 1               | +0,18 | +0,23 | +0,02 | +0,20 | -0,79 | -0,97 | -0,11 | -0,89 | -0,92 | -0,23 |
| 2               | +0,17 | -0,03 | -0,03 | +0,01 | +0,36 | +0,59 | +0,52 | +2,84 | +0,34 | +0,17 |
| 3               | -0,72 | +0,24 | -0,20 | -0,07 | -0,19 | +0,12 | -0,01 | +0,08 | +0,17 | +0,03 |
| 4               | +0,17 | +0,55 | +0,55 | +0,70 | +0,99 | +1,48 | +0,66 | +1,51 | +2,19 | +2,05 |
| 5               | -0,25 | -0,45 | -0,64 | -0,79 | -1,00 | -1,28 | -0,72 | -1,80 | -1,57 | -1,96 |
| 6               | +0,78 | +2,07 | +1,04 | +1,01 | +0,83 | +0,35 | +0,57 | +0,93 | +0,56 | +0,66 |
| 7               | -0,27 | -0,03 | -0,64 | -0,18 | -0,25 | -0,01 | -0,34 | -0,23 | -0,16 | -0,08 |
| 8               | -0,36 | -0,01 | +0,27 | +0,54 | +0,33 | +0,59 | -0,20 | -0,59 | +0,89 | +0,31 |
| 9               | -0,44 | -0,42 | -0,14 | -0,29 | +0,19 | -0,23 | -0,17 | -0,28 | -0,19 | +0,33 |
| 10              | -1,11 | -1,06 | -0,77 | -0,31 | -0,23 | -0,45 | -0,80 | -0,57 | +0,23 | -0,02 |
| 11              | +0,53 | +0,13 | -0,06 | -0,37 | -0,08 | -0,18 | +0,02 | -0,48 | -0,63 | -0,58 |

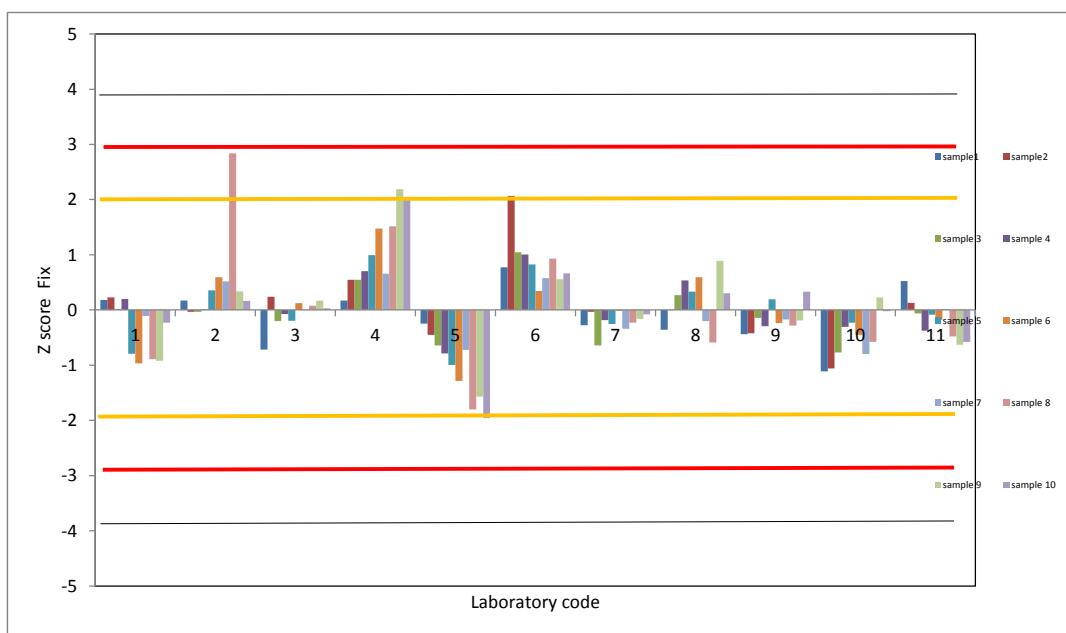
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=1,81

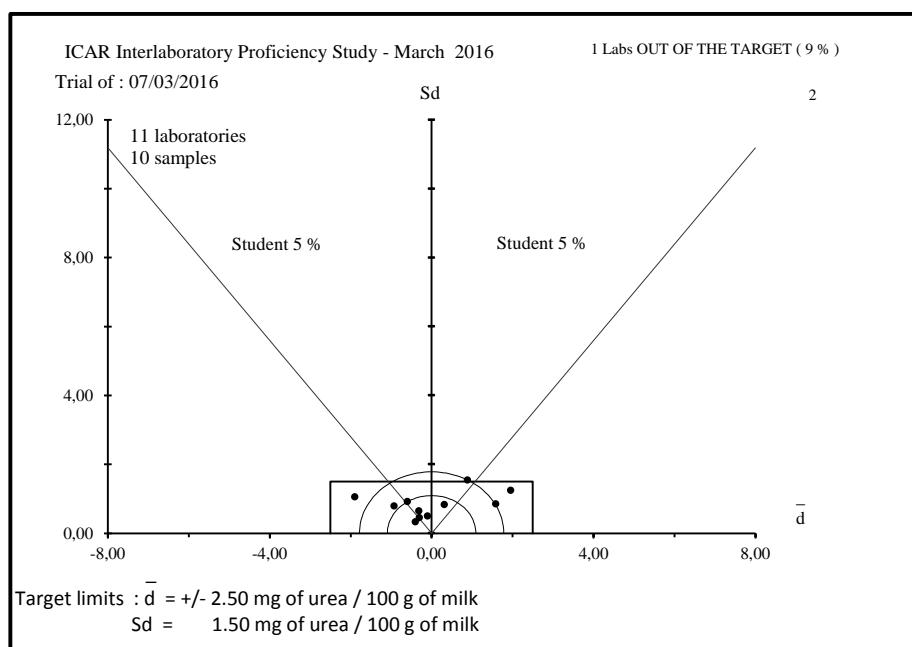
In yellow the values bigger or smaller than 2/-2

In red the values bigger or smaller than 3/-3

**Figure 3 :**

Zscore of the different laboratories for each sample. ZS calculated on the standard deviation of reproducibility of the method



**Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).**

## LIST OF THE PARTICIPANTS ICAR

ICAR PROFICIENCY TEST  
RAW MILK  
UREA CONTENT  
March 2016

| Name   | City                 | Country    |
|--|----------------------|------------|
| ACTALIA-Cecalait                             | POLIGNY              | FRANCE     |
| AIA-LAB. STANDARD LATTE                      | MACARESE (ROMA)      | ITALY      |
| CENTRAL MILK LABORATORY - ICBA               | CAESAREA             | ISRAEL     |
| DEPARTEMENT QUALITE CRA-W                    | GEMBLOUX             | BELGIUM    |
| EASTERN LAB SERVICES                         | MEDINA               | USA        |
| ESTONIAN LIVESTOCK PERFORMANCE RECORDING LTD | TARTU                | ESTONIA    |
| LOM KCHZ LABORATORIM REFERENCYJNE            | PRUSZKOW             | POLAND     |
| MILCHPRUFRING BADEN-WURTTEMBERG E.V.         | KIRCHEIM / TECK      | GERMANY    |
| QLIP N.V.                                    | CM ZUTPHEN           | NETHERLAND |
| VALACTA                                      | STE ANNE DE BELLEVUE | CANADA     |



**ICAR**  
**PROFICIENCY TESTING SCHEME**

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**March 2016**

**Raw Milk**

**Enumeration of SOMATIC CELLS**

Sending date of statistical treatment :      19<sup>th</sup> april 2016

|                     |  |
|---------------------|--|
| Frame of activity : | ICAR Milk Analyses Sub Committee (MA SC)               |
| Contact :           | Gavin Scott<br>Silvia Orlandini                        |
| ICAR Staff          | gavin@milktest.co.nz<br>pt@icar.org    silvia@icar.org |

**Table I : Ranking of the laboratories**      Units:  $10^3$  cells / ml

| Nb | %   | Nº | d     | Sd  | D   | Method |
|----|-----|----|-------|-----|-----|--------|
| 1  | 4   | 12 | + 8   | 11  | 13  | B      |
| 2  | 7   | 10 | + 10  | 15  | 18  | B      |
| 3  | 11  | 14 | - 2   | 28  | 28  | B      |
| 4  | 15  | 4  | - 15  | 38  | 41  | B      |
| 5  | 19  | 1  | - 39  | 31  | 50  | A      |
| 6  | 22  | 2  | - 41  | 32  | 52  | B      |
| 7  | 26  | 16 | + 42  | 32  | 53  | B      |
| 8  | 30  | 9  | + 42  | 34  | 54  | B      |
| 9  | 33  | 24 | - 38  | 38  | 54  | NC     |
| 10 | 37  | 5  | + 43  | 36  | 56  | B      |
| 11 | 41  | 20 | - 45  | 35  | 57  | B      |
| 12 | 44  | 19 | - 43  | 39  | 58  | B      |
| 13 | 48  | 11 | + 48  | 39  | 62  | B      |
| 14 | 52  | 18 | - 37  | 51  | 63  | B      |
| 15 | 56  | 17 | - 53  | 45  | 69  | B      |
| 16 | 59  | 8  | + 53  | 47  | 71  | B      |
| 17 | 63  | 13 | + 54  | 47  | 72  | B      |
| 18 | 67  | 21 | + 54  | 48  | 72  | B      |
| 19 | 70  | 25 | - 56  | 47  | 73  | B      |
| 20 | 74  | 26 | + 47  | 61  | 77  | A      |
| 21 | 78  | 15 | + 65  | 47  | 80  | A      |
| 22 | 81  | 7  | - 62  | 55  | 83  | B      |
| 23 | 85  | 27 | + 66  | 64  | 92  | B      |
| 24 | 89  | 22 | + 75  | 61  | 97  | B      |
| 25 | 93  | 23 | - 81  | 58  | 100 | B      |
| 26 | 96  | 3  | - 115 | 105 | 156 | B      |
| 27 | 100 | 6  | - 60  | 164 | 174 | A      |

The table should be studied in parallel with figure 1 where the laboratories are located according to an acceptability area (or target) the limits of which are :

$\pm 35.10^3$  cells / ml for d and  $35.10^3$  cells / ml for Sd

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 27 laboratories using reference method ISO 13366-1 IDF 148-1 and alternative method ISO 13366-2 IDF 148-2 after outlier discarding using Grubbs test at 5% risk level

A ISO 13366-1 IDF 148-1

B ISO 13366-2 IDF 148-2

(NC : OUT of RANKING because of insufficient data number)

(Nb : laboratory rank; % : relative rank)

(Nº : laboratory identification number)

(d et Sd : mean and standard deviation of the differences (laboratory -reference))

(D : Euclidian distance to YX-axis origin = SQUARE ROOT.(d<sup>2</sup> + Sd<sup>2</sup>))

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.

Repeatability standard deviation of this ICAR proficiency test (after Cochran elimination at 5 %)

Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)

S<sub>r<sub>PT</sub></sub> 14 2%

S<sub>R<sub>PT</sub></sub> 76 13%

**Table II : REPEATABILITY - Absolute difference between replicates in  $10^3$  cells / ml**

| Sample Lab<br>Code | 1   | 2   | 3  | 4     | 5  | 6   | 7  | 8  | 9  | 10 | Sr | NL  |
|--------------------|-----|-----|----|-------|----|-----|----|----|----|----|----|-----|
| 1                  | 15  | 30  | 10 | 10    | 0  | 20  | 30 | 0  | 5  | 5  | 12 | 20  |
| 2                  | 13  | 22  | 7  | 105 * | 1  | 5   | 13 | 2  | 5  | 5  | 24 | 20  |
| 3                  | 18  | 25  | 19 | 5     | 1  | 88  | 3  | 12 | 7  | 0  | 22 | 20  |
| 4                  | 3   | 7   | 1  | 1     | 5  | 4   | 4  | 17 | 2  | 5  | 5  | 20  |
| 5                  | 23  | 1   | 21 | 9     | 2  | 22  | 20 | 19 | 4  | 11 | 11 | 20  |
| 6                  | 78  | 50  | 25 | 31    | 18 | 26  | 15 | 38 | 3  | 1  | 25 | 20  |
| 7                  | 8   | 3   | 7  | 29    | 5  | 31  | 16 | 20 | 0  | 3  | 11 | 20  |
| 8                  | 12  | 6   | 13 | 8     | 17 | 26  | 20 | 29 | 13 | 9  | 12 | 20  |
| 9                  | 55  | 16  | 10 | 11    | 17 | 6   | 5  | 1  | 4  | 2  | 14 | 20  |
| 10                 | 1   | 55  | 3  | 14    | 3  | 5   | 22 | 14 | 9  | 2  | 14 | 20  |
| 11                 | 14  | 38  | 37 | 3     | 9  | 79  | 24 | 9  | 2  | 15 | 23 | 20  |
| 12                 | 7   | 28  | 12 | 15    | 16 | 72  | 38 | 3  | 4  | 12 | 20 | 20  |
| 13                 | 18  | 21  | 19 | 17    | 3  | 18  | 7  | 51 | 5  | 17 | 15 | 20  |
| 14                 | 25  | 4   | 3  | 17    | 2  | 9   | 27 | 3  | 3  | 4  | 9  | 20  |
| 15                 | 10  | 22  | 6  | 7     | 3  | 7   | 17 | 1  | 1  | 0  | 7  | 20  |
| 16                 | 8   | 18  | 2  | 3     | 4  | 6   | 31 | 9  | 2  | 1  | 9  | 20  |
| 17                 | 5   | 20  | 1  | 18    | 8  | 20  | 8  | 7  | 1  | 6  | 8  | 20  |
| 18                 | 33  | 11  | 6  | 9     | 1  | 1   | 9  | 33 | 2  | 11 | 11 | 20  |
| 19                 | 37  | 44  | 17 | 23    | 8  | 17  | 1  | 6  | 5  | 4  | 15 | 20  |
| 20                 | 5   | 6   | 7  | 12    | 22 | 0   | 7  | 3  | 9  | 10 | 7  | 20  |
| 21                 | 7   | 35  | 14 | 12    | 23 | 19  | 26 | 47 | 1  | 3  | 16 | 20  |
| 22                 | 80  | 26  | 26 | 4     | 4  | 10  | 6  | 0  | 6  | 9  | 20 | 20  |
| 23                 | 13  | 6   | 2  | **    | ** | 3   | 0  | 1  | 1  | ** | 4  | 14  |
| 24                 | 3   | 27  | 20 | 3     | 2  | 15  | 2  | 6  | 2  | 2  | 8  | 20  |
| 25                 | 22  | 26  | 5  | 1     | 4  | 56  | 3  | 10 | 0  | 4  | 15 | 20  |
| 26                 | 28  | 28  | 7  | 4     | 5  | 42  | 32 | 5  | 2  | 4  | 15 | 20  |
| 27                 | 10  | 14  | 1  | 10    | 8  | 4   | 9  | 4  | 4  | 1  | 5  | 20  |
| Sr                 | 20  | 18  | 10 | 17    | 7  | 23  | 13 | 14 | 3  | 5  |    | 534 |
| r                  | 126 | 126 | 42 | 50    | 42 | 126 | 63 | 63 | 25 | 25 |    |     |
| NE                 | 54  | 54  | 54 | 52    | 52 | 54  | 54 | 54 | 54 | 52 |    |     |
| L                  | 84  | 76  | 42 | 39    | 29 | 96  | 53 | 56 | 14 | 21 |    |     |

Sr : repeatability standard deviation of each laboratory limit : Cf up down

NL : number of measurements per laboratory

L : Limit for difference between duplicates according Cochran test at 5% level.

SE : repeatability standard deviation per sample

NE : number of measurements per sample

\*: discarded data using the test of Cochran at 5 %

\*\*: missing data

r : limit of repeatability, absolute difference between two replicates according ISO 13366-2 / IDF 148-2 : Cf up down

| Level $10^3$ / ml | Sr % | r   |
|-------------------|------|-----|
| 150               | 6    | 25  |
| 200               | 5    | 42  |
| 450               | 4    | 50  |
| 750               | 3    | 63  |
| 1500              | 3    | 126 |

**Table III :** Means of the replicates in  $10^3$  cells / ml

| Sample Lab<br>Code | 1    | 2    | 3   | 4   | 5   | 6    | 7   | 8   | 9    | 10   |
|--------------------|------|------|-----|-----|-----|------|-----|-----|------|------|
| 1                  | 893  | 1085 | 245 | 355 | 170 | 1280 | 655 | 500 | 48   | 83   |
| 2                  | 892  | 1089 | 249 | 308 | 171 | 1288 | 662 | 503 | 48   | 83   |
| 3                  | 769  | 924  | 227 | 328 | 149 | 1054 | 557 | 435 | 38   | 71   |
| 4                  | 882  | 1083 | 291 | 395 | 194 | 1329 | 707 | 500 | 62   | 108  |
| 5                  | 1057 | 1224 | 296 | 423 | 196 | 1481 | 748 | 562 | 47   | 98   |
| 6                  | 675  | 812  | 196 | 316 | 140 | 1619 | 781 | 474 | 18 * | 74   |
| 7                  | 872  | 1029 | 251 | 347 | 158 | 1201 | 630 | 465 | 43   | 83   |
| 8                  | 1067 | 1267 | 304 | 418 | 196 | 1502 | 770 | 559 | 55   | 91   |
| 9                  | 1040 | 1237 | 293 | 416 | 183 | 1451 | 781 | 572 | 49   | 97   |
| 10                 | 1006 | 1164 | 280 | 406 | 175 | 1376 | 716 | 543 | 49   | 88   |
| 11                 | 1038 | 1249 | 313 | 417 | 191 | 1498 | 755 | 573 | 53   | 97   |
| 12                 | 971  | 1165 | 275 | 394 | 184 | 1410 | 704 | 534 | 46   | 96   |
| 13                 | 1077 | 1281 | 301 | 419 | 198 | 1489 | 761 | 571 | 55   | 94   |
| 14                 | 966  | 1134 | 287 | 385 | 175 | 1333 | 718 | 538 | 98 * | 45 * |
| 15                 | 1083 | 1259 | 307 | 429 | 212 | 1527 | 779 | 587 | 63   | 102  |
| 16                 | 1034 | 1242 | 296 | 425 | 194 | 1466 | 744 | 567 | 56   | 100  |
| 17                 | 858  | 1068 | 256 | 364 | 155 | 1244 | 633 | 464 | 44   | 85   |
| 18                 | 886  | 1079 | 264 | 370 | 169 | 1230 | 667 | 486 | 53   | 125  |
| 19                 | 874  | 1090 | 255 | 358 | 160 | 1253 | 650 | 500 | 47   | 81   |
| 20                 | 904  | 1103 | 255 | 360 | 159 | 1266 | 623 | 460 | 46   | 79   |
| 21                 | 1084 | 1266 | 277 | 428 | 197 | 1489 | 778 | 576 | 56   | 95   |
| 22                 | 1084 | 1278 | 289 | 457 | 204 | 1565 | 811 | 609 | 61   | 93   |
| 23                 | 874  | 1028 | 226 |     |     | 1196 | 627 | 483 | 45   |      |
| 24                 | 892  | 1080 | 278 | 339 | 164 | 1265 | 678 | 480 | 54   | 85   |
| 25                 | 891  | 1036 | 246 | 357 | 161 | 1238 | 617 | 463 | 43   | 87   |
| 26                 | 1113 | 1263 | 283 | 352 | 189 | 1505 | 744 | 556 | 49   | 117  |
| 27                 | 1090 | 1299 | 301 | 425 | 195 | 1552 | 789 | 574 | 47   | 92   |
| M                  | 958  | 1142 | 272 | 384 | 178 | 1374 | 707 | 523 | 50   | 92   |
| REF.               | 963  | 1150 | 273 | 384 | 178 | 1377 | 708 | 523 | 50   | 91   |
| SD                 | 112  | 121  | 29  | 40  | 19  | 143  | 68  | 49  | 6    | 12   |

M = mean per sample

REF. = reference values

SD = standard deviation per sample

\*: discarded data using the test of Grubbs 5 %

**REF** : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 27 laboratories using the reference method ISO 13366 / IDF 148-1 and alternative method ISO 13366-2 IDF 148-2, after outlier discarding using Grubbs test at 5% risk level

**Table IV :** Outlier identification

| Sample          | 1   | 2   | 3   | 4   | 5   | 6   | 7   | 8   | 9    | 10  |
|-----------------|-----|-----|-----|-----|-----|-----|-----|-----|------|-----|
| <b>Outliers</b> |     |     |     |     |     |     |     |     |      |     |
| <b>Cochran</b>  |     |     |     | 2   |     |     |     |     |      |     |
| <b>Outlier</b>  |     |     |     |     |     |     |     |     |      |     |
| <b>Grubbs</b>   |     |     |     |     |     |     |     |     | 6;14 | 14  |
| <b>sr</b>       | 20  | 18  | 10  | 10  | 7   | 23  | 13  | 14  | 3    | 5   |
| <b>SR</b>       | 113 | 122 | 30  | 39  | 20  | 144 | 68  | 50  | 7    | 13  |
| <b>sr %</b>     | 2%  | 2%  | 4%  | 2%  | 4%  | 2%  | 2%  | 3%  | 7%   | 6%  |
| <b>SR %</b>     | 12% | 11% | 11% | 10% | 11% | 10% | 10% | 10% | 14%  | 14% |

**Table V : ACCURACY - differences (laboratory - reference) in  $10^3$  cells / ml**

| Sample Lab code | 1     | 2     | 3    | 4    | 5    | 6     | 7     | 8    | 9    | 10   | d     | Sd <sub>lab</sub> | t    |
|-----------------|-------|-------|------|------|------|-------|-------|------|------|------|-------|-------------------|------|
| 1               | - 71  | - 65  | - 28 | - 29 | - 8  | - 97  | - 53  | - 23 | - 2  | - 8  | - 39  | 31                | 3,87 |
| 2               | - 72  | - 61  | - 25 | - 77 | - 8  | - 90  | - 47  | - 20 | - 2  | - 8  | - 41  | 32                | 4,01 |
| 3               | - 194 | - 226 | - 47 | - 57 | - 30 | - 323 | - 152 | - 88 | - 12 | - 20 | - 115 | 105               | 3,47 |
| 4               | - 82  | - 67  | + 17 | + 10 | + 15 | - 48  | - 1   | - 24 | + 12 | + 17 | - 15  | 38                | 1,26 |
| 5               | + 93  | + 74  | + 22 | + 38 | + 18 | + 104 | + 40  | + 38 | - 3  | + 7  | + 43  | 36                | 3,77 |
| 6               | - 288 | - 338 | - 78 | - 69 | - 38 | + 242 | + 72  | - 49 | - 32 | - 17 | - 60  | 164               | 1,15 |
| 7               | - 91  | - 121 | - 23 | - 38 | - 21 | - 177 | - 78  | - 58 | - 7  | - 8  | - 62  | 55                | 3,56 |
| 8               | + 104 | + 117 | + 30 | + 34 | + 17 | + 125 | + 62  | + 35 | + 5  | - 0  | + 53  | 47                | 3,58 |
| 9               | + 76  | + 87  | + 20 | + 31 | + 4  | + 74  | + 72  | + 48 | - 1  | + 6  | + 42  | 34                | 3,90 |
| 10              | + 42  | + 14  | + 6  | + 22 | - 4  | - 2   | + 8   | + 20 | - 1  | - 3  | + 10  | 15                | 2,20 |
| 11              | + 75  | + 99  | + 39 | + 32 | + 12 | + 120 | + 47  | + 49 | + 3  | + 6  | + 48  | 39                | 3,87 |
| 12              | + 7   | + 15  | + 2  | + 9  | + 6  | + 33  | - 4   | + 10 | - 4  | + 5  | + 8   | 11                | 2,36 |
| 13              | + 114 | + 131 | + 27 | + 34 | + 19 | + 112 | + 52  | + 47 | + 5  | + 3  | + 54  | 47                | 3,62 |
| 14              | + 2   | - 16  | + 13 | + 0  | - 3  | - 45  | + 9   | + 14 | + 48 | - 46 | - 2   | 28                | 0,25 |
| 15              | + 120 | + 109 | + 34 | + 44 | + 33 | + 149 | + 70  | + 63 | + 13 | + 11 | + 65  | 47                | 4,32 |
| 16              | + 71  | + 92  | + 23 | + 40 | + 16 | + 89  | + 35  | + 43 | + 6  | + 9  | + 42  | 32                | 4,22 |
| 17              | - 106 | - 82  | - 18 | - 20 | - 23 | - 133 | - 75  | - 60 | - 6  | - 6  | - 53  | 45                | 3,73 |
| 18              | - 78  | - 71  | - 9  | - 15 | - 10 | - 148 | - 42  | - 38 | + 3  | + 34 | - 37  | 51                | 2,30 |
| 19              | - 90  | - 60  | - 19 | - 27 | - 18 | - 125 | - 59  | - 23 | - 3  | - 10 | - 43  | 39                | 3,49 |
| 20              | - 60  | - 47  | - 19 | - 24 | - 19 | - 111 | - 86  | - 64 | - 4  | - 12 | - 45  | 35                | 4,01 |
| 21              | + 120 | + 116 | + 4  | + 44 | + 18 | + 111 | + 70  | + 52 | + 6  | + 4  | + 54  | 48                | 3,60 |
| 22              | + 121 | + 128 | + 16 | + 73 | + 26 | + 188 | + 103 | + 86 | + 11 | + 2  | + 75  | 61                | 3,87 |
| 23              | - 90  | - 122 | - 47 |      |      | - 182 | - 81  | - 41 | - 5  |      | - 81  | 58                | 3,68 |
| 24              | - 72  | - 70  | + 5  | - 46 | - 14 | - 113 | - 30  | - 43 | + 4  | - 6  | - 38  | 38                | 3,19 |
| 25              | - 72  | - 114 | - 28 | - 28 | - 17 | - 139 | - 92  | - 60 | - 7  | - 4  | - 56  | 47                | 3,76 |
| 26              | + 150 | + 113 | + 9  | - 32 | + 10 | + 128 | + 36  | + 32 | - 1  | + 26 | + 47  | 61                | 2,44 |
| 27              | + 127 | + 149 | + 27 | + 41 | + 17 | + 175 | + 80  | + 51 | - 3  | + 1  | + 66  | 64                | 3,30 |
| d               | - 5   | - 8   | - 2  | - 0  | - 0  | - 3   | - 2   | - 0  | + 0  | + 1  | - 2   | 75                |      |
| Sd              | 112   | 121   | 29   | 40   | 19   | 143   | 68    | 49   | 6    | 12   | 76    |                   |      |

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

Upper limits :  $\bar{d} = +/- 35 \cdot 10^3$  cells / ml   Sd =  $35 \cdot 10^3$  cells / ml**ISO 13366-2 / IDF 148-2 : Precision of the method :**

| Level SCC * $10^3$ /ml | Sr % | r   | SR % | R   |
|------------------------|------|-----|------|-----|
| 150                    | 6    | 25  | 9    | 38  |
| 200                    | 5    | 42  | 8    | 67  |
| 450                    | 4    | 50  | 7    | 88  |
| 750                    | 3    | 63  | 6    | 126 |
| 1500                   | 3    | 126 | 6    | 252 |

**Table VI :** Zscore of the different laboratories for each sample.

ZS calculated on the PT standard deviation

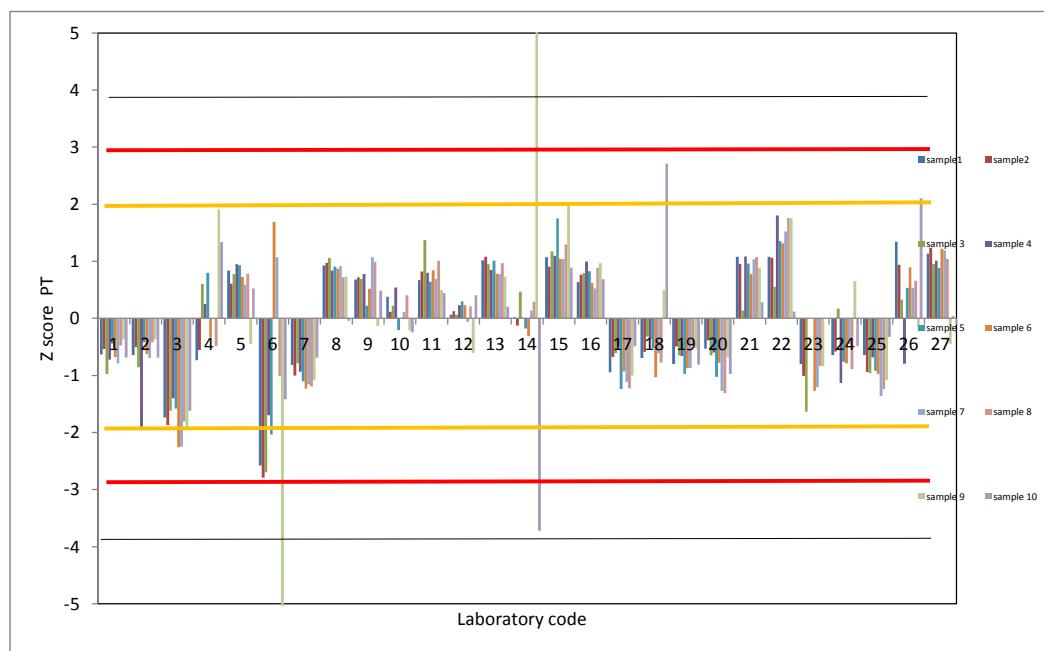
| Sample Lab code | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     | 10    |
|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 1               | -0,63 | -0,53 | -0,98 | -0,72 | -0,44 | -0,68 | -0,79 | -0,48 | -0,37 | -0,69 |
| 2               | -0,64 | -0,50 | -0,85 | -1,90 | -0,42 | -0,63 | -0,69 | -0,42 | -0,37 | -0,69 |
| 3               | -1,74 | -1,87 | -1,62 | -1,40 | -1,58 | -2,26 | -2,25 | -1,81 | -1,94 | -1,62 |
| 4               | -0,73 | -0,55 | +0,60 | +0,26 | +0,80 | -0,34 | -0,02 | -0,49 | +1,91 | +1,34 |
| 5               | +0,84 | +0,61 | +0,78 | +0,95 | +0,93 | +0,73 | +0,59 | +0,78 | -0,45 | +0,53 |
| 6               | -2,58 | -2,79 | -2,70 | -1,70 | -2,03 | +1,69 | +1,07 | -1,01 | -5,08 | -1,42 |
| 7               | -0,81 | -1,00 | -0,79 | -0,93 | -1,10 | -1,23 | -1,16 | -1,19 | -1,08 | -0,69 |
| 8               | +0,93 | +0,97 | +1,06 | +0,84 | +0,91 | +0,87 | +0,92 | +0,72 | +0,73 | -0,04 |
| 9               | +0,68 | +0,72 | +0,69 | +0,77 | +0,22 | +0,52 | +1,07 | +0,99 | -0,13 | +0,49 |
| 10              | +0,38 | +0,11 | +0,22 | +0,54 | -0,21 | -0,01 | +0,12 | +0,40 | -0,21 | -0,24 |
| 11              | +0,67 | +0,82 | +1,37 | +0,80 | +0,64 | +0,84 | +0,69 | +1,01 | +0,49 | +0,45 |
| 12              | +0,07 | +0,13 | +0,07 | +0,23 | +0,30 | +0,23 | -0,06 | +0,21 | -0,61 | +0,40 |
| 13              | +1,02 | +1,08 | +0,95 | +0,85 | +1,01 | +0,78 | +0,78 | +0,97 | +0,73 | +0,20 |
| 14              | +0,02 | -0,13 | +0,46 | +0,01 | -0,18 | -0,31 | +0,14 | +0,29 | +7,49 | -3,72 |
| 15              | +1,07 | +0,90 | +1,18 | +1,10 | +1,75 | +1,04 | +1,04 | +1,30 | +1,99 | +0,89 |
| 16              | +0,63 | +0,76 | +0,79 | +1,00 | +0,83 | +0,62 | +0,52 | +0,89 | +0,97 | +0,69 |
| 17              | -0,94 | -0,67 | -0,61 | -0,50 | -1,24 | -0,93 | -1,11 | -1,23 | -1,00 | -0,49 |
| 18              | -0,69 | -0,59 | -0,32 | -0,36 | -0,52 | -1,03 | -0,62 | -0,77 | +0,49 | +2,71 |
| 19              | -0,80 | -0,49 | -0,65 | -0,66 | -0,97 | -0,87 | -0,87 | -0,48 | -0,53 | -0,81 |
| 20              | -0,53 | -0,39 | -0,65 | -0,60 | -1,03 | -0,78 | -1,27 | -1,31 | -0,68 | -0,97 |
| 21              | +1,08 | +0,96 | +0,14 | +1,08 | +0,96 | +0,78 | +1,03 | +1,07 | +0,89 | +0,28 |
| 22              | +1,08 | +1,06 | +0,55 | +1,80 | +1,36 | +1,31 | +1,52 | +1,76 | +1,75 | +0,12 |
| 23              | -0,80 | -1,01 | -1,64 | -1,64 | -1,27 | -1,20 | -0,84 | -0,84 | -0,84 | -0,84 |
| 24              | -0,64 | -0,58 | +0,17 | -1,13 | -0,76 | -0,79 | -0,45 | -0,89 | +0,65 | -0,49 |
| 25              | -0,64 | -0,94 | -0,96 | -0,68 | -0,92 | -0,97 | -1,36 | -1,24 | -1,08 | -0,32 |
| 26              | +1,34 | +0,94 | +0,33 | -0,80 | +0,54 | +0,89 | +0,53 | +0,66 | -0,13 | +2,10 |
| 27              | +1,13 | +1,23 | +0,95 | +1,01 | +0,88 | +1,22 | +1,19 | +1,04 | -0,45 | +0,04 |

In yellow the values bigger or smaller than 2/-2

In red the values bigger or smaller than 3/-3

**Figure 2 :**

Zscore of the different laboratories for each sample. ZS calculated on the PT standard deviation



**Table VII :****Zscore of the different laboratories for each sample.****ZS calculated on the standard deviation of reproducibility of the method**

| Sample Lab code | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9      | 10    |
|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|--------|-------|
| 1               | -1,22 | -0,94 | -1,14 | -0,95 | -0,52 | -1,18 | -1,07 | -0,64 | -0,53  | -1,04 |
| 2               | -1,24 | -0,88 | -1,00 | -2,49 | -0,49 | -1,08 | -0,94 | -0,55 | -0,53  | -1,04 |
| 3               | -3,36 | -3,28 | -1,90 | -1,84 | -1,86 | -3,91 | -3,06 | -2,41 | -2,75  | -2,44 |
| 4               | -1,41 | -0,97 | +0,71 | +0,34 | +0,94 | -0,58 | -0,02 | -0,65 | +2,71  | +2,01 |
| 5               | +1,62 | +1,07 | +0,91 | +1,25 | +1,10 | +1,26 | +0,80 | +1,04 | -0,64  | +0,79 |
| 6               | -4,99 | -4,90 | -3,16 | -2,23 | -2,39 | +2,93 | +1,46 | -1,35 | -7,21  | -2,14 |
| 7               | -1,58 | -1,76 | -0,92 | -1,23 | -1,30 | -2,14 | -1,58 | -1,59 | -1,53  | -1,04 |
| 8               | +1,80 | +1,70 | +1,24 | +1,10 | +1,07 | +1,51 | +1,25 | +0,96 | +1,03  | -0,06 |
| 9               | +1,32 | +1,27 | +0,81 | +1,02 | +0,26 | +0,89 | +1,46 | +1,32 | -0,19  | +0,73 |
| 10              | +0,73 | +0,20 | +0,26 | +0,71 | -0,24 | -0,02 | +0,16 | +0,54 | -0,30  | -0,37 |
| 11              | +1,30 | +1,44 | +1,60 | +1,05 | +0,76 | +1,46 | +0,94 | +1,34 | +0,70  | +0,67 |
| 12              | +0,13 | +0,22 | +0,08 | +0,30 | +0,35 | +0,40 | -0,08 | +0,28 | -0,86  | +0,61 |
| 13              | +1,97 | +1,90 | +1,11 | +1,12 | +1,19 | +1,35 | +1,06 | +1,29 | +1,03  | +0,31 |
| 14              | +0,04 | -0,23 | +0,54 | +0,01 | -0,21 | -0,54 | +0,19 | +0,39 | +10,62 | -5,62 |
| 15              | +2,07 | +1,59 | +1,38 | +1,44 | +2,06 | +1,81 | +1,42 | +1,73 | +2,82  | +1,34 |
| 16              | +1,23 | +1,34 | +0,93 | +1,31 | +0,97 | +1,08 | +0,71 | +1,18 | +1,37  | +1,04 |
| 17              | -1,83 | -1,18 | -0,72 | -0,66 | -1,46 | -1,61 | -1,52 | -1,63 | -1,42  | -0,73 |
| 18              | -1,34 | -1,03 | -0,37 | -0,48 | -0,62 | -1,79 | -0,84 | -1,03 | +0,70  | +4,09 |
| 19              | -1,55 | -0,86 | -0,76 | -0,87 | -1,14 | -1,51 | -1,18 | -0,64 | -0,75  | -1,22 |
| 20              | -1,03 | -0,68 | -0,76 | -0,79 | -1,21 | -1,35 | -1,73 | -1,74 | -0,97  | -1,47 |
| 21              | +2,08 | +1,68 | +0,16 | +1,43 | +1,13 | +1,35 | +1,41 | +1,43 | +1,26  | +0,43 |
| 22              | +2,09 | +1,86 | +0,65 | +2,37 | +1,60 | +2,27 | +2,07 | +2,34 | +2,48  | +0,18 |
| 23              | -1,55 | -1,76 | -1,92 | -1,49 | -0,90 | -2,20 | -1,64 | -1,11 | -1,19  |       |
| 24              | -1,24 | -1,02 | +0,20 | -1,49 | -0,90 | -1,36 | -0,61 | -1,18 | +0,92  | -0,73 |
| 25              | -1,25 | -1,65 | -1,12 | -0,90 | -1,08 | -1,68 | -1,85 | -1,65 | -1,53  | -0,49 |
| 26              | +2,59 | +1,64 | +0,38 | -1,05 | +0,63 | +1,55 | +0,72 | +0,88 | -0,19  | +3,17 |
| 27              | +2,20 | +2,17 | +1,11 | +1,33 | +1,04 | +2,12 | +1,62 | +1,38 | -0,64  | +0,06 |

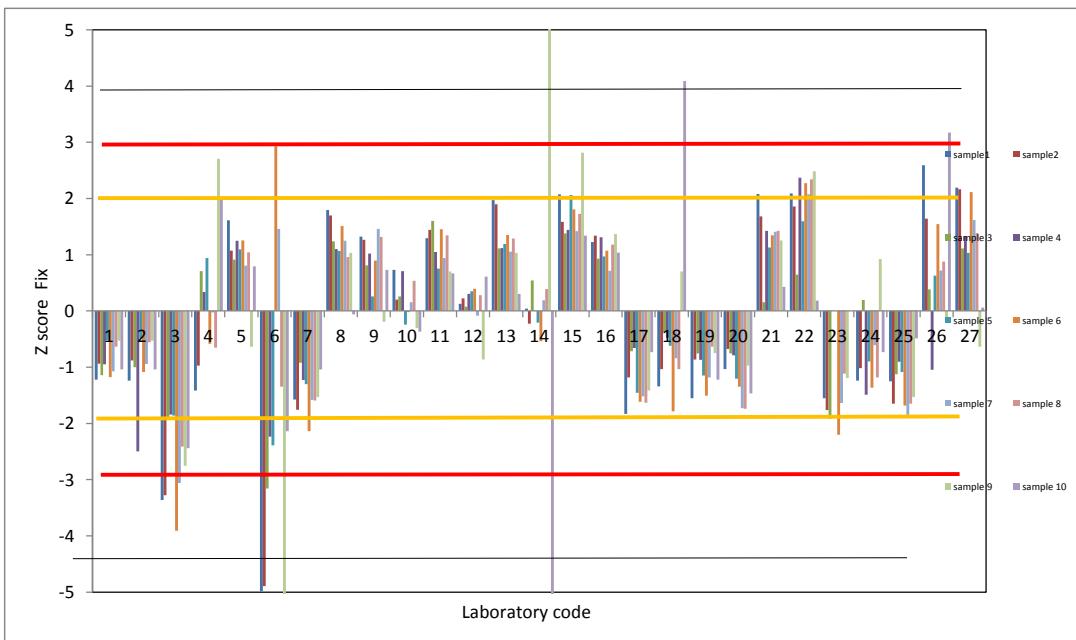
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR : Cf page 5/8

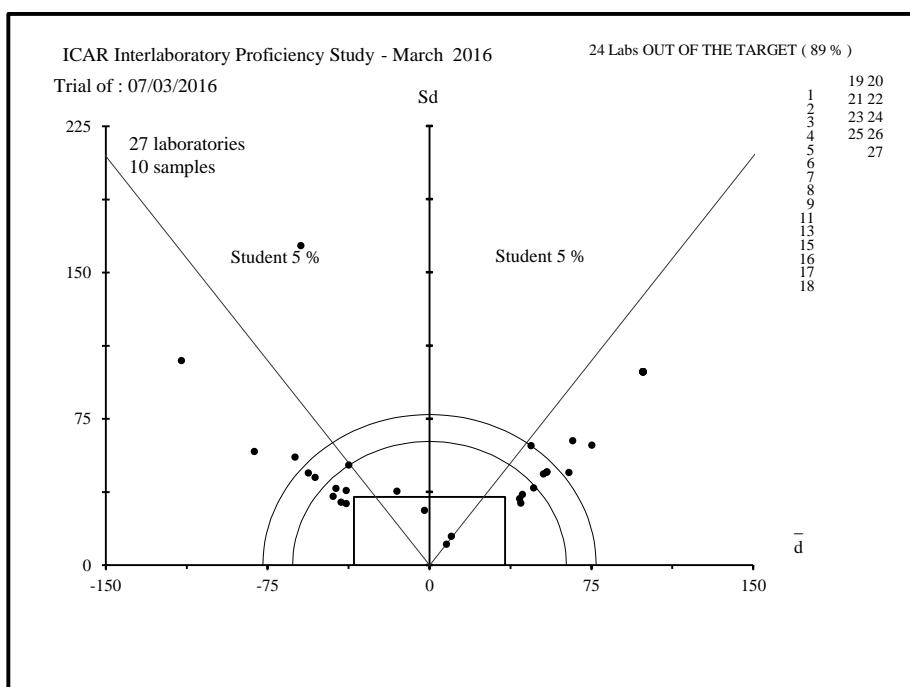
In yellow the values bigger or smaller than 2/-2

In red the values bigger or smaller than 3/-3

**Figure 3 :**

Zscore of the different laboratories for each sample. ZS calculated on the standard deviation of reproducibility of the method





**Figure 1 :** ACCURACY - Evaluation of the individual performances (to see table I).

LIST OF THE PARTICIPANTS ICAR  
ICAR PROFICIENCY TEST

RAW MILK

Enumeration of SOMATIC CELLS  
March 2016

FRANCE

| Name   | City                 | Country        |
|--|----------------------|----------------|
| ACTALIA-Cecalait                             | POLIGNY              | FRANCE         |
| ALLATTENYESZTESI TELJESITMENYVIZSGALO KFT    | GODOLLO              | HUNGARY        |
| CENTRAL MILK LABORATORY - ICBA               | CAESAREA             | ISRAEL         |
| EASTERN LAB SERVICES                         | MEDINA               | USA            |
| ESTONIAN LIVESTOCK PERFORMANCE RECORDING LTD | TARTU                | ESTONIA        |
| FEDERAZIONE LATTERIE ALTO ADIGE              | BOLZANO              | ITALY          |
| LAB. AGROALIMENTARIO DE SANTANDER            | SANTANDER            | SPAIN          |
| LACTOLAB                                     | IRENE                | SOUTH AFRICA   |
| LOM KCHZ LABORATORIM REFERENCYJNE            | PRUSZKOW             | POLAND         |
| MILCHPRUFRING BADEN-WURTTEMBERG E.V.         | KIRCHEIM / TECK      | GERMANY        |
| P.F.H.B. PROD. MLEKA - KOBIERNO              | KOBIERNO             | POLAND         |
| P.F.H.B. PROD. MLEKA - MINIKOWO              | MINIKOWO             | POLAND         |
| P.F.H.B. PROD. MLEKA - PRUSZKOW              | PRUSZKOW             | POLAND         |
| P.F.H.B. PROD. MLEKA - TYKOCIN               | TYKOCIN              | POLAND         |
| QLIP N.V.                                    | CM ZUTPHEN           | NETHERLAND     |
| SUISSE LAB AG                                | ZOLLIKOFEN           | SWITZERLAND    |
| THE CATTLE INFORMATION SERVICE               | TELFORD              | UNITED KINGDOM |
| UNIVERZITET U NOVOM SADU                     | NOVI SAD             | SERBIA         |
| VALACTA                                      | STE ANNE DE BELLEVUE | CANADA         |