THE GLOBAL STANDARD
FOR LIVESTOCK DATA

Network. Guidelines. Certification.

FRAME OF ACTIVITY : ICAR MILK ANALYSES SUB-COMMITTEE (MA SC)

# ICAR - Proficiency Testing Scheme Raw cow milk

## “Routine” Methods

ORGANISER: ICAR, VIA SAVOIA 78, I-00198 ROME, ITALY

THE GLOBAL STANDARD FOR LIVESTOCK DATA

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# 1.Introduction

Dear Participant,

Thank you for participating in the ICAR Proficiency Test (PT) March 2017 !

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" 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 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.

D) In this table is reported if some of your results are outlier for Grubbs or Cochran test

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 E the results of your $Z\text{-Score}_{PT}$ (standard deviation calculated on this proficiency test) and the $Z\text{-Score}_{FIX}$ (standard deviation of the reference method) are summarized. If you have obtained all the $-2<Z\text{-Score results}<+2$ 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 F 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.

H) The control charts have been created using your data obtained with the Z-Score$_{PT}$ and Z-Score$_{FIX}$. 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.

For the BHB we have not established a fix standard deviation so we are providing you only the ZS for the PT

Your Control Charts have been updated with data of the previous ICAR PT if you have participated.

The sample preparation and statistical elaboration have been done by ICAR Sub- contractor Actalia, accreditated 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 and urea.

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.

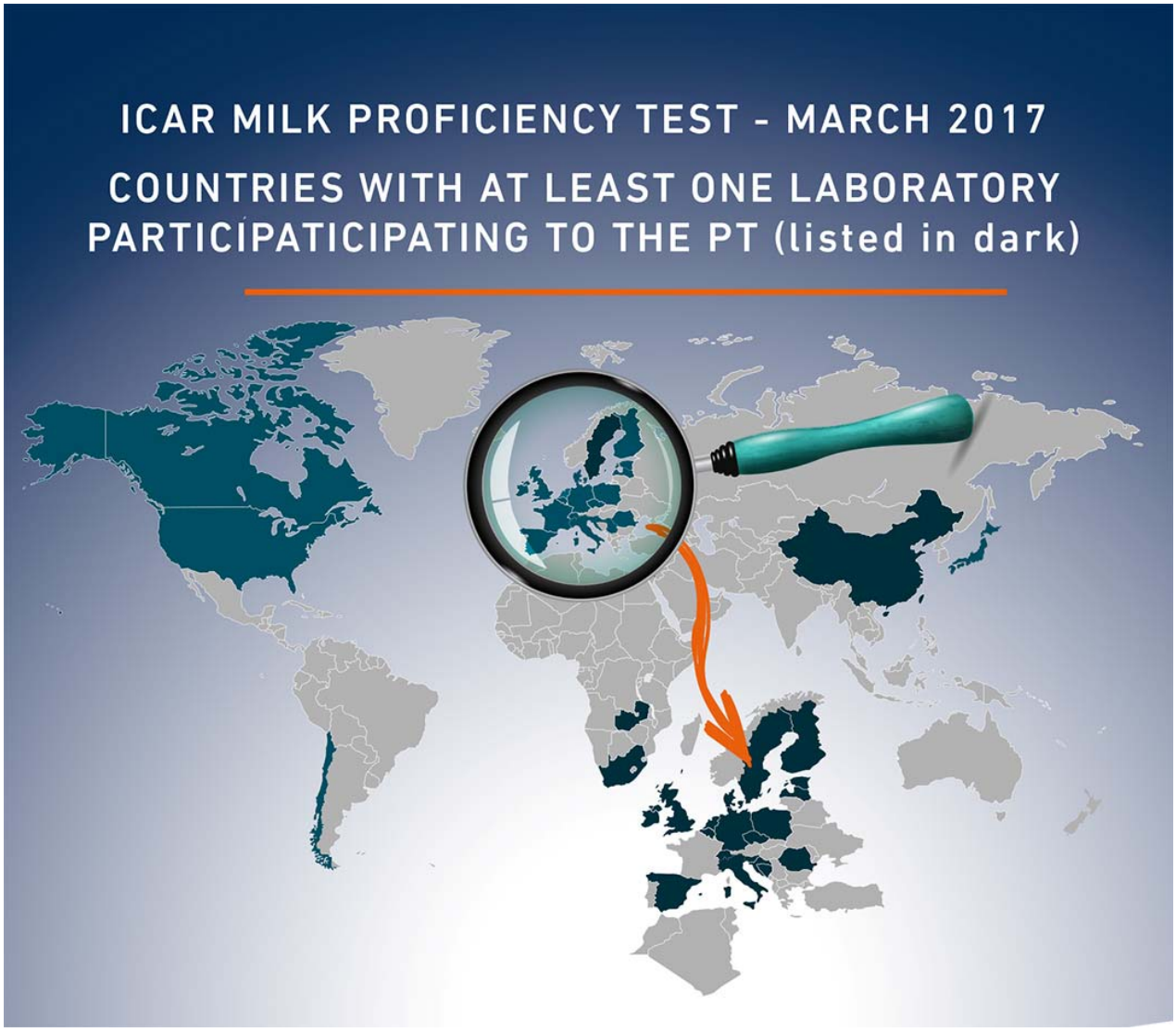
For each parameter the SR=standard deviation of reproducibility has been calculated after the outlier elimination. If you have participated, and your results are in the repeatability limits, you can use this value for the calculation of your uncertainty of measurement.

ICAR would like to see, in the next years, this 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 ICAR PT 2016 with at least one parameter is reported below and upload on ICAR website

http://www.icar.org/index.php/certifications/milk-analysis-laboratories-certifications/rresults-of-the-milk-laboratories-proficiency-test-march-2017




| Laboratory | Country |
|---|---|
| Eastern Lab services | USA |
| Qlip B.V., | The Netherlands |
| Teagasc Food research Center | Ireland |
| Milchprüfring Baden-Württemberg e.V. | Germany |
| Laboratorio Standard Latte | Italy |
| Central Milk Lab ICBA | Israel |
| Cattle Information Service (CIS) | England |
| Eurofins Steins Laboratory A/S | Denmark |
| Valacta - Centre d'Expertise en Production Laitière du Québec | Canada |
| Estonian Livestock Performance Recording Ltd | Estonia |

| Laboratory | Country |
|---|---|
| Laboratorium Oceny Mleka KCHZ Laboratorium Referecyjne z/s w Parzniewie | Poland |
| Japan Dairy Technical Association | Japan |
| PFHBiPM Region Oceny Bydgoszcz z/s w Minikowie | Poland |
| Laboratorija za ispitivanje kvaliteta mleka, Poljoprivredni fakultet Novi Sad, | Serbia |
| University of Ljubljana Biotechnical faculty department of Animal Science Institute of Dairy Science and Probiotics | Slovenia |
| Federazione Latterie Alto Adige Soc. Agr. Coop. | Italy |
| Actalia | Finland |
| Direction de l' Amelioration Genetique DirectiondeAmeliorationGenetique | Tunisia |
| Laborator pro rozbor mléka Bustehrad, Ceskomoravská spolecnost chovatelú a.s. | Czech Rep. |
| Merieux Nutriscience South Africa | South Africa |
| Valio | Finland |
| Central Milk Testing Lab | Canada |
| Pacific Milk Analysis | Canada |
| Horizon Lab Ltd | Canada |
| Croatian Agricultural Agency, Central Laboratory for Milk Quality Control | Croatia |
| Delaval international AB | Sweden |
| COOPRINSEM | Chile |
| Merieux NutriScience | South Africa |
| Holstein-Ro Breeders Association Laboratory | Romania |
| Eurofins Steins Laboratory A/B | Sweden |
| Shangai dairy breeding center Co.Ltd | China |

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

THE GLOBAL STANDARD
FOR LIVESTOCK DATA

# Routine Methods

ICAR PT
September 2016

## Laboratory participation codes and Performance analyses

| Laboratory Name | Laboratory name |
|---|---|

**A — Your participation Codes**

| | Fat | Protein | Lactose | Urea |
|---|---|---|---|---|
| Subscription | Yes | Yes | | |
| Participation Codes | 13 | 13 | | |
| Are all the sample results received ? | Yes | Yes | | |

**B — Data results received on time**

| Yes | No | Deadline |
|---|---|---|
| | | 19.09.2016 |

**C — Have you sent the data with the correct units of measurements ?**

| Fat | Protein | Lactose | Urea |
|---|---|---|---|
| g/100g | g/100g | g/100g | mg/dl |
| Yes | Yes | | |

**D — Outliers**

| Fat | Protein | Lactose | Urea |
|---|---|---|---|
| Sample | Sample | Sample | Sample |
| g/100g | g/100g | g/100g | mg/dl |
| | | | |
| | | | |

**E — Repeatability**

Your "r" performance

| Fat | Protein | Lactose | Urea |
|---|---|---|---|
| g/100g | g/100g | g/100g | mg/dl |
| | | | |

If the repeatability in 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.

Limits

| Fat | Protein | Lactose | Urea |
|---|---|---|---|
| g/100g | g/100g | g/100g | mg/dl |
| ISO 9622 IDF 141 | | | ISO 14637 IDF 195 |
| 0,04 | 0,04 | 0,04 | 1,5 |

**F — Z-Score**

Your Z-Score PT

| Fat | Protein | Lactose | Urea |
|---|---|---|---|
| | | | |
| | | | |

Your FIX Z-Score

| Fat | Protein | Lactose | Urea |
|---|---|---|---|
| | | | |
| | | | |

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.

Interpretation Z-Score

| -2<Z-Score<2 | -3<Z-Score<-2 | 2<Z-Score<3 | Z-Score<-3 | Z-Score>3 |
|---|---|---|---|---|
| Good | Moderate | Moderate | Poor | Poor |

**G — Ranking of your lab**

Mean difference and standard deviation of difference

| Fat | Protein | Lactose | Urea |
|---|---|---|---|
| | | | |

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

Indicative Limits defined in the ICAR MA SC

| Fat | Protein | Lactose | Urea |
|---|---|---|---|
| g/100g | g/100g | g/100g | mg/dl |
| d=0,02 | d=0,025 | d=0,10 | d=2,5 |
| sd=0,03 | sd=0,020 | sd=0,10 | sd=1,5 |

Note : Limits are only indicative and so far do not constitute standard values; they indicate what is normally reachhable by labs for their self evaluation

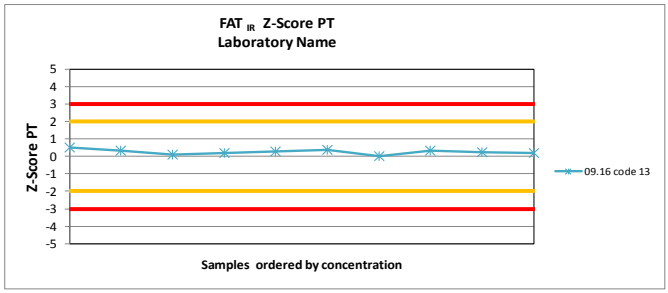
**Legenda:**

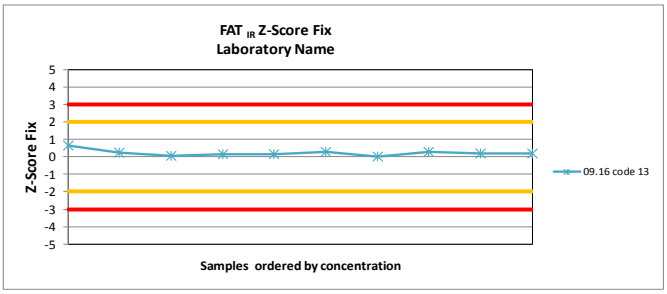
- positive performance for all the sample
- same sample showed a performance out of range or there are miss data
- The parameter was not analyzed

H

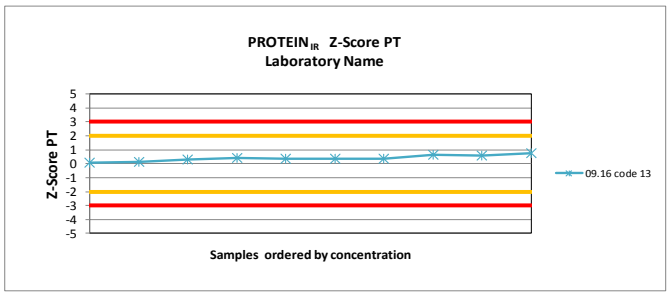
# Control Charts

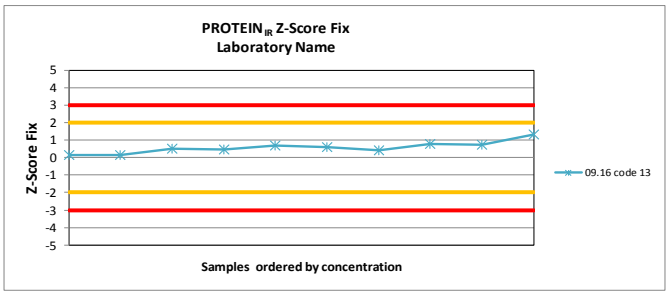
## Control Charts Fat




## Control Charts Protein



# ICAR
# PROFICIENCY TESTING SCHEME

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## March 2017

## Raw Milk

## Determination of FAT CONTENT
### Routine method

Sending date of statistical treatment : 21th April 2017


| | | |
|---|---|---|
| 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 |

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

| Nb | % | N° | d | Sd | D | Method |
|---|---|---|---|---|---|---|
| 1 | 5 | 1 | + 0,003 | 0,006 | 0,007 | IR |
| 2 | 10 | 14 | - 0,007 | 0,013 | 0,015 | IR |
| 3 | 14 | 15 | + 0,010 | 0,012 | 0,016 | IR |
| 4 | 19 | 8 | + 0,016 | 0,009 | 0,019 | IR |
| 5 | 24 | 5 | - 0,005 | 0,018 | 0,019 | IR |
| 6 | 29 | 16 | + 0,020 | 0,008 | 0,022 | IR |
| 7 | 33 | 6 | - 0,014 | 0,018 | 0,023 | IR |
| 8 | 38 | 17 | - 0,009 | 0,025 | 0,027 | IR |
| 9 | 43 | 4 | - 0,025 | 0,015 | 0,029 | IR |
| 10 | 48 | 2 | + 0,018 | 0,028 | 0,033 | Other* |
| 11 | 52 | 21 | + 0,038 | 0,011 | 0,039 | IR |
| 12 | 57 | 3 | + 0,038 | 0,013 | 0,040 | IR |
| 13 | 62 | 11 | + 0,010 | 0,046 | 0,047 | IR |
| 14 | 67 | 12 | + 0,045 | 0,028 | 0,053 | IR |
| 15 | 71 | 20 | - 0,052 | 0,015 | 0,054 | IR |
| 16 | 76 | 10 | - 0,016 | 0,053 | 0,055 | IR |
| 17 | 81 | 7 | + 0,031 | 0,048 | 0,057 | IR |
| 18 | 86 | 13 | - 0,055 | 0,024 | 0,060 | IR |
| 19 | 90 | 9 | - 0,032 | 0,072 | 0,079 | IR |
| 20 | 95 | 18 | + 0,059 | 0,122 | 0,135 | IR |
| 21 | 100 | 19 | - 0,084 | 0,137 | 0,161 | IR |

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 20 sets of results send by 20 laboratories using routine method ISO 9622|IDF 141, after outlier discarging using Grubbs test at 5 % risk level

Other* : ASU L 01.00-78 IR-mesurement

(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.($d^2$ + $Sd^2$))

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.

| | | |
|---|---|---|
| eatability standard deviation of this ICAR proficiency test (after Cochran elimination at 5 %) | $Sr_{PT}$ | 0,005 |
| oducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) | $SR_{PT}$ | 0,040 |

**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 | 0,010 | 0,010 | 0,000 | 0,010 | 0,000 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,006 | 20 |
| 2 | 0,010 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,002 | 20 |
| 3 | 0,010 | 0,000 | 0,020 * | 0,020 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,009 | 20 |
| 4 | 0,010 | 0,010 | 0,000 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,010 | 0,004 | 20 |
| 5 | 0,000 | 0,000 | 0,000 | 0,010 | 0,000 | 0,010 | 0,010 | 0,000 | 0,000 | 0,000 | 0,004 | 20 |
| 6 | 0,010 | 0,000 | 0,010 | 0,010 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,000 | 0,004 | 20 |
| 7 | 0,002 | 0,017 | 0,005 | 0,005 | 0,020 * | 0,010 | 0,010 | 0,010 | 0,012 | 0,014 | 0,008 | 20 |
| 8 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,000 | 0,002 | 20 |
| 9 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,010 | 0,000 | 0,003 | 20 |
| 10 | 0,010 | 0,020 | 0,010 | 0,010 | 0,010 | 0,020 | 0,010 | 0,010 | 0,010 | 0,010 | 0,009 | 20 |
| 11 | 0,010 | 0,010 | 0,000 | 0,000 | 0,010 | 0,020 | 0,010 | 0,000 | 0,010 | 0,010 | 0,007 | 20 |
| 12 | 0,030 * | 0,000 | 0,010 | 0,010 | 0,000 | 0,010 | 0,000 | 0,000 | 0,010 | 0,000 | 0,008 | 20 |
| 13 | 0,000 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,010 | 0,004 | 20 |
| 14 | 0,010 | 0,010 | 0,005 | 0,005 | 0,000 | 0,005 | 0,000 | 0,005 | 0,005 | 0,005 | 0,004 | 20 |
| 15 | 0,010 | 0,000 | 0,000 | 0,010 | 0,010 | 0,000 | 0,030 * | 0,010 | 0,000 | 0,000 | 0,008 | 20 |
| 16 | 0,010 | 0,010 | 0,010 | 0,000 | 0,000 | 0,010 | 0,000 | 0,010 | 0,000 | 0,000 | 0,005 | 20 |
| 17 | 0,010 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,010 | 0,010 | 0,000 | 0,000 | 0,004 | 20 |
| 18 | 0,010 | 0,000 | 0,000 | 0,010 | 0,010 | 0,000 | 0,010 | 0,000 | 0,000 | 0,010 | 0,005 | 20 |
| 19 | 0,040 * | 0,010 | 0,000 | 0,010 | 0,000 | 0,050 * | 0,020 | 0,000 | 0,010 | 0,030 * | 0,017 | 20 |
| 20 | 0,000 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,003 | 20 |
| 21 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 20 |
| Sr | 0,009 | 0,006 | 0,004 | 0,006 | 0,005 | 0,010 | 0,007 | 0,004 | 0,005 | 0,007 | | 420 |
| NE | 42 | 42 | 42 | 42 | 42 | 42 | 42 | 42 | 42 | 42 | | |
| L | 0,022 | 0,022 | 0,013 | 0,023 | 0,014 | 0,027 | 0,023 | 0,017 | 0,018 | 0,019 | | |

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 betwen two replicates=0,040 according ISO 9622|IDF 141

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

| Sample / Lab code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 4,805 | 4,195 | 3,510 | 2,805 | 2,120 | 4,535 | 3,855 | 3,105 | 2,425 | 1,455 |
| 2 | 4,845 | 4,240 | 3,540 | 2,810 | 2,110 | 4,580 | 3,880 | 3,120 | 2,410 | 1,430 |
| 3 | 4,825 | 4,220 | 3,540 | 2,840 | 2,175 | 4,565 | 3,875 | 3,135 | 2,475 | 1,515 |
| 4 | 4,765 | 4,165 | 3,490 | 2,790 | 2,100 | 4,475 | 3,810 | 3,090 | 2,410 | 1,435 |
| 5 | 4,780 | 4,180 | 3,500 | 2,805 | 2,140 | 4,505 | 3,815 | 3,100 | 2,430 | 1,480 |
| 6 | 4,755 | 4,170 | 3,495 | 2,805 | 2,130 | 4,495 | 3,830 | 3,100 | 2,420 | 1,440 |
| 7 | 4,929 | 4,222 | 3,518 | 2,810 | 2,120 | 4,645 | 3,845 | 3,135 | 2,404 | 1,465 |
| 8 | 4,810 | 4,200 | 3,520 | 2,820 | 2,150 | 4,545 | 3,850 | 3,120 | 2,440 | 1,490 |
| 9 | 4,750 | 4,140 | 3,440 | 2,740 | 2,050 | 4,545 | 3,850 | 3,250 * | 2,345 | 1,350 |
| 10 | 4,815 | 4,200 | 3,515 | 2,805 | 2,115 | 4,370 | 3,845 | 3,105 | 2,415 | 1,435 |
| 11 | 4,775 | 4,175 | 3,500 | 2,800 | 2,125 | 4,660 | 3,815 | 3,090 | 2,455 | 1,485 |
| 12 | 4,865 | 4,230 | 3,565 | 2,775 | 2,170 | 4,605 | 3,900 | 3,150 | 2,445 | 1,530 |
| 13 | 4,710 | 4,120 | 3,450 | 2,755 | 2,090 | 4,450 | 3,785 | 3,050 | 2,380 | 1,445 |
| 14 | 4,815 | 4,195 | 3,503 | 2,788 | 2,110 | 4,548 | 3,820 | 3,088 | 2,408 | 1,438 |
| 15 | 4,835 | 4,200 | 3,520 | 2,825 | 2,125 | 4,550 | 3,845 | 3,105 | 2,420 | 1,460 |
| 16 | 4,825 | 4,205 | 3,525 | 2,820 | 2,140 | 4,565 | 3,860 | 3,115 | 2,440 | 1,490 |
| 17 | 4,795 | 4,200 | 3,540 | 2,790 | 2,100 | 4,540 | 3,845 | 3,095 | 2,400 | 1,390 |
| 18 | 5,205 * | 4,190 | 3,560 | 2,815 | 2,145 | 4,560 | 3,835 | 3,130 | 2,440 | 1,495 |
| 19 | 4,750 | 4,225 | 3,540 | 2,745 | 2,140 | 4,415 | 3,790 | 3,120 | 2,095 * | 1,125 * |
| 20 | 4,730 | 4,120 | 3,460 | 2,755 | 2,080 | 4,470 | 3,785 | 3,050 | 2,380 | 1,430 |
| 21 | 4,860 | 4,230 | 3,550 | 2,830 | 2,150 | 4,590 | 3,870 | 3,140 | 2,450 | 1,490 |
| M | 4,802 | 4,192 | 3,513 | 2,797 | 2,123 | 4,534 | 3,838 | 3,107 | 2,420 | 1,457 |
| REF. | 4,800 | 4,189 | 3,512 | 2,796 | 2,124 | 4,532 | 3,836 | 3,106 | 2,420 | 1,459 |
| SD | 0,052 | 0,034 | 0,034 | 0,028 | 0,030 | 0,071 | 0,031 | 0,026 | 0,030 | 0,042 |

M = mean per sample
SD = standard deviation per sample
REF. = reference values
*: 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 20 laboratories using the Routine method ISO 9622|IDF 141 , after outliers discarging using Grubbs test at 5 % risk level.

**Table IV :** Outlier identification

| **Sample** | **1** | **2** | **3** | **4** | **5** | **6** | **7** | **8** | **9** | **10** |
|---|---|---|---|---|---|---|---|---|---|---|
| **Outliers Cochran** | 12; 19 | | 3 | | 7 | 19 | 15 | | | 19 |
| **Outlier Grubbs** | 18 | | | | | | | 9 | 19 | 19 |
| **sr** | 0,006 | 0,006 | 0,003 | 0,006 | 0,004 | 0,007 | 0,006 | 0,004 | 0,004 | 0,005 |
| **SR** | 0,052 | 0,034 | 0,034 | 0,028 | 0,030 | 0,067 | 0,032 | 0,027 | 0,030 | 0,042 |

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

| Sample / Lab Code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | d | $Sd_{lab}$ | t |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | + 0,005 | + 0,006 | - 0,002 | + 0,009 | - 0,004 | + 0,003 | + 0,019 | - 0,001 | + 0,005 | - 0,004 | + 0,004 | 0,007 | 1,65 |
| 2 | + 0,045 | + 0,051 | + 0,028 | + 0,014 | - 0,014 | + 0,048 | + 0,044 | + 0,014 | - 0,010 | - 0,029 | + 0,019 | 0,029 | 2,09 |
| 3 | + 0,025 | + 0,031 | + 0,028 | + 0,044 | + 0,051 | + 0,033 | + 0,039 | + 0,029 | + 0,055 | + 0,056 | + 0,039 | 0,012 | 10,55 |
| 4 | - 0,035 | - 0,024 | - 0,022 | - 0,006 | - 0,024 | - 0,057 | - 0,026 | - 0,016 | - 0,010 | - 0,024 | - 0,024 | 0,014 | 5,50 |
| 5 | - 0,020 | - 0,009 | - 0,012 | + 0,009 | + 0,016 | - 0,027 | - 0,021 | - 0,006 | + 0,010 | + 0,021 | - 0,004 | 0,017 | 0,72 |
| 6 | - 0,045 | - 0,019 | - 0,017 | + 0,009 | + 0,006 | - 0,037 | - 0,006 | - 0,006 | - 0,000 | - 0,019 | - 0,013 | 0,018 | 2,41 |
| 7 | + 0,129 | + 0,032 | + 0,006 | + 0,014 | - 0,004 | + 0,113 | + 0,009 | + 0,029 | - 0,016 | + 0,006 | + 0,032 | 0,049 | 2,04 |
| 8 | + 0,010 | + 0,011 | + 0,008 | + 0,024 | + 0,026 | + 0,013 | + 0,014 | + 0,014 | + 0,020 | + 0,031 | + 0,017 | 0,008 | 6,97 |
| 9 | - 0,050 | - 0,049 | - 0,072 | - 0,056 | - 0,074 | + 0,013 | + 0,014 | + 0,144 | - 0,075 | - 0,109 | - 0,031 | 0,072 | 1,37 |
| 10 | + 0,015 | + 0,011 | + 0,003 | + 0,009 | - 0,009 | - 0,162 | + 0,009 | - 0,001 | - 0,005 | - 0,024 | - 0,015 | 0,053 | 0,92 |
| 11 | - 0,025 | - 0,014 | - 0,012 | + 0,004 | + 0,001 | + 0,128 | - 0,021 | - 0,016 | + 0,035 | + 0,026 | + 0,011 | 0,046 | 0,73 |
| 12 | + 0,065 | + 0,041 | + 0,053 | - 0,021 | + 0,046 | + 0,073 | + 0,064 | + 0,044 | + 0,025 | + 0,071 | + 0,046 | 0,028 | 5,21 |
| 13 | - 0,090 | - 0,069 | - 0,062 | - 0,041 | - 0,034 | - 0,082 | - 0,051 | - 0,056 | - 0,040 | - 0,014 | - 0,054 | 0,023 | 7,43 |
| 14 | + 0,015 | + 0,006 | - 0,009 | - 0,008 | - 0,014 | + 0,016 | - 0,016 | - 0,019 | - 0,013 | - 0,021 | - 0,006 | 0,014 | 1,46 |
| 15 | + 0,035 | + 0,011 | + 0,008 | + 0,029 | + 0,001 | + 0,018 | + 0,009 | - 0,001 | - 0,000 | + 0,001 | + 0,011 | 0,013 | 2,77 |
| 16 | + 0,025 | + 0,016 | + 0,013 | + 0,024 | + 0,016 | + 0,033 | + 0,024 | + 0,009 | + 0,020 | + 0,031 | + 0,021 | 0,008 | 8,47 |
| 17 | - 0,005 | + 0,011 | + 0,028 | - 0,006 | - 0,024 | + 0,008 | + 0,009 | - 0,011 | - 0,020 | - 0,069 | - 0,008 | 0,027 | 0,94 |
| 18 | + 0,405 | + 0,001 | + 0,048 | + 0,019 | + 0,021 | + 0,028 | - 0,001 | + 0,024 | + 0,020 | + 0,036 | + 0,060 | 0,122 | 1,56 |
| 19 | - 0,050 | + 0,036 | + 0,028 | - 0,051 | + 0,016 | - 0,117 | - 0,046 | + 0,014 | - 0,325 | - 0,334 | - 0,083 | 0,138 | 1,90 |
| 20 | - 0,070 | - 0,069 | - 0,052 | - 0,041 | - 0,044 | - 0,062 | - 0,051 | - 0,056 | - 0,040 | - 0,029 | - 0,051 | 0,013 | 12,24 |
| 21 | + 0,060 | + 0,041 | + 0,038 | + 0,034 | + 0,026 | + 0,058 | + 0,034 | + 0,034 | + 0,030 | + 0,031 | + 0,039 | 0,012 | 10,50 |
| d | + 0,002 | + 0,002 | + 0,001 | + 0,001 | - 0,001 | + 0,002 | + 0,002 | + 0,001 | - 0,001 | - 0,001 | + 0,000 | 0,059 | |
| Sd | 0,052 | 0,034 | 0,034 | 0,028 | 0,030 | 0,071 | 0,031 | 0,026 | 0,030 | 0,042 | 0,040 | | |

d = mean of differences  Sd = standard deviation of differences  t = Student test - comparison to 0

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

**ISO 9622|IDF 141 : Precision of the method :**  Sr = 0.014 g / 100 g
SR = 0,04 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 | +0,10 | +0,17 | -0,06 | +0,33 | -0,13 | +0,05 | +0,60 | -0,05 | +0,16 | -0,09 |
| 2 | +0,87 | +1,50 | +0,83 | +0,50 | -0,46 | +0,68 | +1,40 | +0,51 | -0,34 | -0,68 |
| 3 | +0,49 | +0,91 | +0,83 | +1,57 | +1,73 | +0,47 | +1,24 | +1,08 | +1,84 | +1,33 |
| 4 | -0,66 | -0,71 | -0,65 | -0,21 | -0,80 | -0,80 | -0,84 | -0,62 | -0,34 | -0,56 |
| 5 | -0,38 | -0,27 | -0,35 | +0,33 | +0,55 | -0,38 | -0,68 | -0,24 | +0,33 | +0,50 |
| 6 | -0,86 | -0,56 | -0,50 | +0,33 | +0,21 | -0,52 | -0,20 | -0,24 | -0,00 | -0,45 |
| 7 | +2,48 | +0,95 | +0,16 | +0,48 | -0,13 | +1,60 | +0,28 | +1,08 | -0,54 | +0,15 |
| 8 | +0,20 | +0,32 | +0,24 | +0,86 | +0,89 | +0,19 | +0,44 | +0,51 | +0,67 | +0,74 |
| 9 | -0,95 | -1,44 | -2,13 | -1,98 | -2,49 | +0,19 | +0,44 | +5,42 | -2,51 | -2,57 |
| 10 | +0,29 | +0,32 | +0,09 | +0,33 | -0,30 | -2,29 | +0,28 | -0,05 | -0,17 | -0,56 |
| 11 | -0,47 | -0,41 | -0,35 | +0,15 | +0,04 | +1,82 | -0,68 | -0,62 | +1,17 | +0,62 |
| 12 | +1,25 | +1,20 | +1,57 | -0,74 | +1,56 | +1,04 | +2,05 | +1,65 | +0,83 | +1,68 |
| 13 | -1,72 | -2,03 | -1,83 | -1,45 | -1,14 | -1,16 | -1,64 | -2,13 | -1,34 | -0,33 |
| 14 | +0,29 | +0,17 | -0,28 | -0,30 | -0,46 | +0,22 | -0,52 | -0,72 | -0,42 | -0,50 |
| 15 | +0,68 | +0,32 | +0,24 | +1,04 | +0,04 | +0,26 | +0,28 | -0,05 | -0,00 | +0,03 |
| 16 | +0,49 | +0,47 | +0,38 | +0,86 | +0,55 | +0,47 | +0,76 | +0,32 | +0,67 | +0,74 |
| 17 | -0,09 | +0,32 | +0,83 | -0,21 | -0,80 | +0,12 | +0,28 | -0,43 | -0,67 | -1,63 |
| 18 | +7,77 | +0,03 | +1,42 | +0,68 | +0,72 | +0,40 | -0,04 | +0,89 | +0,67 | +0,86 |
| 19 | -0,95 | +1,06 | +0,83 | -1,81 | +0,55 | -1,65 | -1,48 | +0,51 | -10,88 | -7,89 |
| 20 | -1,34 | -2,03 | -1,54 | -1,45 | -1,48 | -0,87 | -1,64 | -2,13 | -1,34 | -0,68 |
| 21 | +1,16 | +1,20 | +1,12 | +1,21 | +0,89 | +0,83 | +1,08 | +1,27 | +1,00 | +0,74 |

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

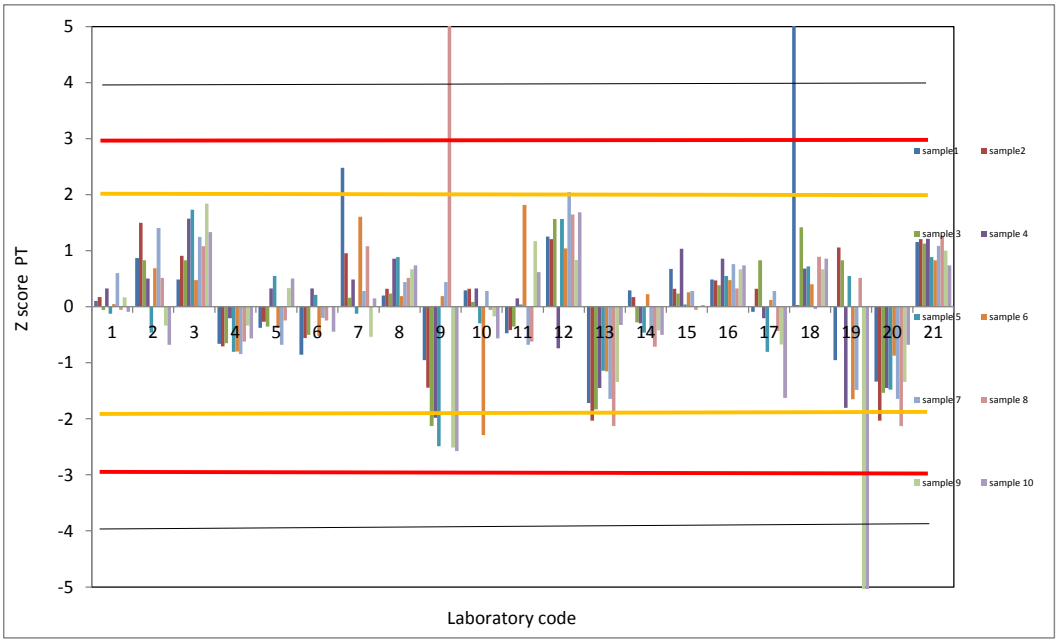


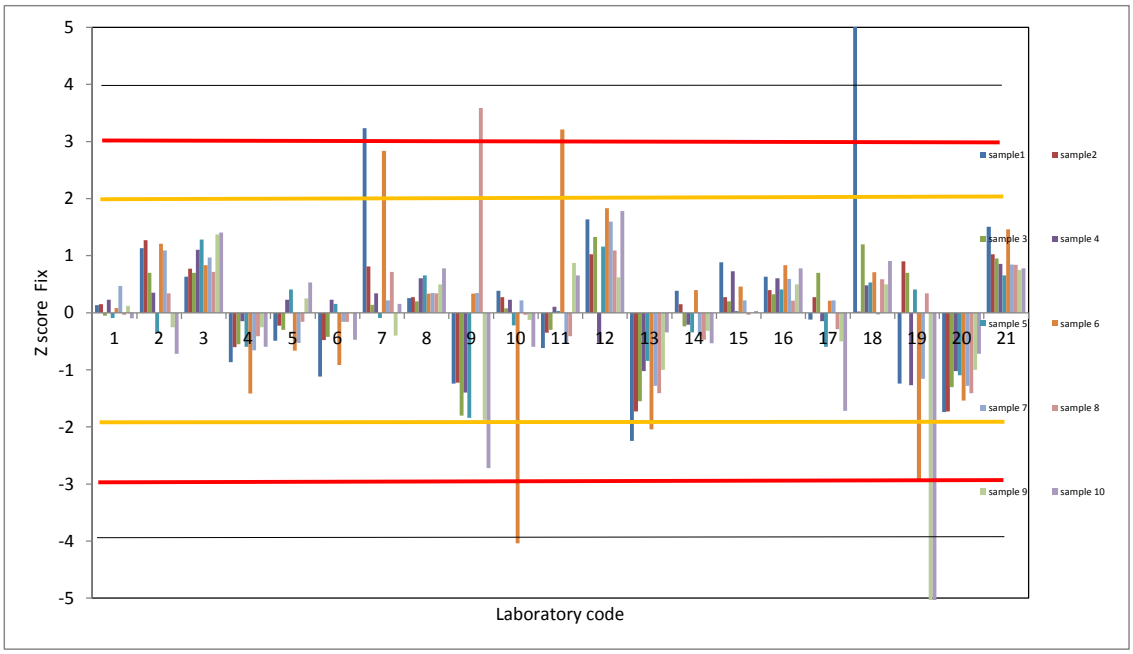
**Table VII :** **Zscore of the different laboratories for each sample.**
**ZS calculated on the standard deviation of reproducibility of the method**

| Sample / Lab code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | +0,13 | +0,15 | -0,05 | +0,23 | -0,09 | +0,08 | +0,47 | -0,04 | +0,12 | -0,10 |
| 2 | +1,13 | +1,27 | +0,70 | +0,35 | -0,34 | +1,21 | +1,09 | +0,34 | -0,25 | -0,72 |
| 3 | +0,63 | +0,77 | +0,70 | +1,10 | +1,28 | +0,83 | +0,97 | +0,71 | +1,37 | +1,40 |
| 4 | -0,87 | -0,60 | -0,55 | -0,15 | -0,59 | -1,42 | -0,66 | -0,41 | -0,25 | -0,60 |
| 5 | -0,49 | -0,23 | -0,30 | +0,23 | +0,41 | -0,67 | -0,53 | -0,16 | +0,25 | +0,53 |
| 6 | -1,12 | -0,48 | -0,42 | +0,23 | +0,16 | -0,92 | -0,16 | -0,16 | -0,00 | -0,47 |
| 7 | +3,23 | +0,81 | +0,14 | +0,34 | -0,09 | +2,83 | +0,22 | +0,71 | -0,40 | +0,15 |
| 8 | +0,26 | +0,27 | +0,20 | +0,60 | +0,66 | +0,33 | +0,34 | +0,34 | +0,50 | +0,78 |
| 9 | -1,24 | -1,23 | -1,80 | -1,40 | -1,84 | +0,33 | +0,34 | +3,59 | -1,88 | -2,72 |
| 10 | +0,38 | +0,27 | +0,08 | +0,23 | -0,22 | -4,04 | +0,22 | -0,04 | -0,13 | -0,60 |
| 11 | -0,62 | -0,35 | -0,30 | +0,10 | +0,03 | +3,21 | -0,53 | -0,41 | +0,87 | +0,65 |
| 12 | +1,63 | +1,02 | +1,33 | -0,52 | +1,16 | +1,83 | +1,59 | +1,09 | +0,62 | +1,78 |
| 13 | -2,24 | -1,73 | -1,55 | -1,02 | -0,84 | -2,04 | -1,28 | -1,41 | -1,00 | -0,35 |
| 14 | +0,38 | +0,15 | -0,24 | -0,21 | -0,34 | +0,40 | -0,41 | -0,47 | -0,31 | -0,53 |
| 15 | +0,88 | +0,27 | +0,20 | +0,73 | +0,03 | +0,46 | +0,22 | -0,04 | -0,00 | +0,03 |
| 16 | +0,63 | +0,40 | +0,33 | +0,60 | +0,41 | +0,83 | +0,59 | +0,21 | +0,50 | +0,78 |
| 17 | -0,12 | +0,27 | +0,70 | -0,15 | -0,59 | +0,21 | +0,22 | -0,29 | -0,50 | -1,72 |
| 18 | +10,13 | +0,02 | +1,20 | +0,48 | +0,53 | +0,71 | -0,03 | +0,59 | +0,50 | +0,90 |
| 19 | -1,24 | +0,90 | +0,70 | -1,27 | +0,41 | -2,92 | -1,16 | +0,34 | -8,13 | -8,35 |
| 20 | -1,74 | -1,73 | -1,30 | -1,02 | -1,09 | -1,54 | -1,28 | -1,41 | -1,00 | -0,72 |
| 21 | +1,51 | +1,02 | +0,95 | +0,85 | +0,66 | +1,46 | +0,84 | +0,84 | +0,75 | +0,78 |

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=0,040

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



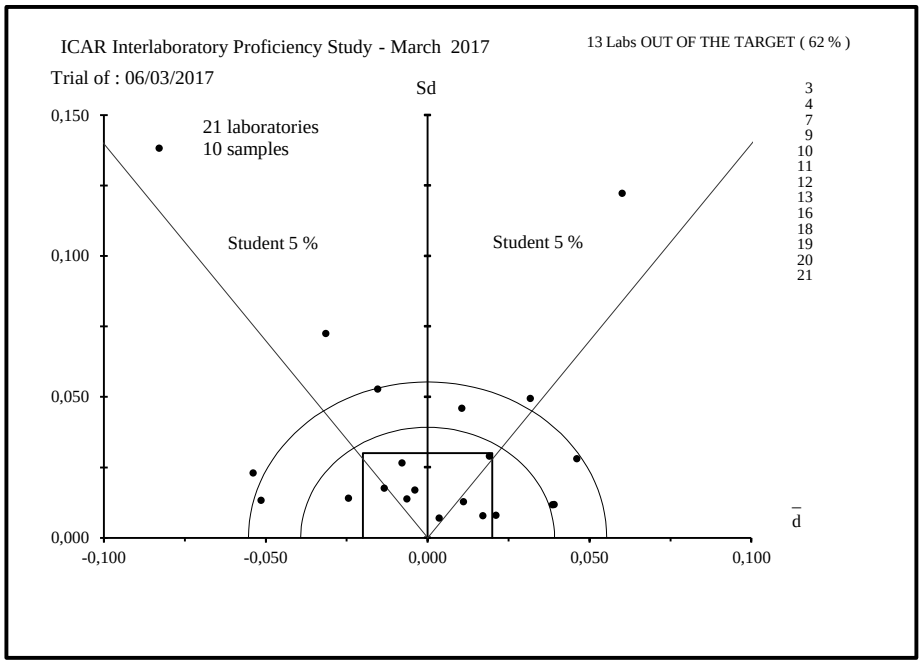



**Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).**

LIST OF ICAR PARTICIPANTS
RAW MILK

FAT ROUTINE METHOD
MARCH 2017

| | | |
|---|---|---|
| AIA-LAB. STANDARD LATTE | MACCARESE (ROMA) | ITALY |
| CATTLE INFORMATION SERVICE (CIS) | TELFORD | UNITED KINGDOM |
| COOPRINSEM | OSORNO | CHILE |
| CROATIAN AGRICULTURAL AGENCY MILK QUALITY CONTROL | KRIZEVCI | CROATIA |
| EASTERN LAB SERVICES | MEDINA | USA |
| EUROFINS STEINS LABORATORY A/B | VEJEN | DENMARK |
| EUROFINS STEINS LABORATORY A/B | JÖNGÖPING | SWEDEN |
| FED.LATTERIE SOCIALI DI BOLZANO | BOLZANO | ITALY |
| LAB. POLJOPRIVREDNI | NOVI SAD | SERBIA |
| LOM KCHZ LABORATORIUM REFERENCYJNE z/s w | PARZNIEWIE | POLAND |
| MERIEUX NUTRISCIENCE | CAPE TOWN | SOUTH AFRICA |
| MERIEUX NUTRISCIENCE | MIDRAND | SOUTH AFRICA |
| MERIEUX NUTRISCIENCE J BAY | CAPE TOWN | SOUTH AFRICA |
| MILCHPRUFRING BADEN-WURTTEMBERG E.V. | KIRCHEIM / TECK | GERMANY |
| SHANGAI DAIRY BREEDING CENTER Co.Ltd | SHANGHAI | CHINA |
| TAIWAN LIVESTOCK ESEARCH INT. | TAIWAN | TAIWAN |
| VETLAB AGRICULTURAL SHOWGROUNDS | LUSAKA | ZAMBIA |

# ICAR
# PROFICIENCY TESTING SCHEME

---

## March 2017

## Raw Milk

## Determination of CRUDE PROTEIN CONTENT
### Routine method

Sending date of statistical treatment : 21th April 2017


| | | |
|---|---|---|
| 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 |

ACTALIA
CECALAIT

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

| Nb | % | N° | d | Sd | D | Method |
|---|---|---|---|---|---|---|
| 1 | 5 | 20 | + 0,004 | 0,008 | 0,009 | IR |
| 2 | 10 | 3 | - 0,006 | 0,009 | 0,011 | IR |
| 3 | 14 | 9 | - 0,010 | 0,005 | 0,012 | IR |
| 4 | 19 | 13 | + 0,014 | 0,010 | 0,017 | IR |
| 5 | 24 | 21 | - 0,007 | 0,019 | 0,020 | IR |
| 6 | 29 | 19 | - 0,021 | 0,014 | 0,025 | IR |
| 7 | 33 | 16 | + 0,026 | 0,009 | 0,028 | IR |
| 8 | 38 | 15 | - 0,023 | 0,022 | 0,032 | IR |
| 9 | 43 | 5 | + 0,032 | 0,008 | 0,032 | IR |
| 10 | 48 | 2 | - 0,010 | 0,037 | 0,038 | Other* |
| 11 | 52 | 8 | + 0,038 | 0,008 | 0,038 | IR |
| 12 | 57 | 14 | + 0,041 | 0,010 | 0,042 | IR |
| 13 | 62 | 17 | - 0,043 | 0,017 | 0,047 | IR |
| 14 | 67 | 10 | - 0,046 | 0,012 | 0,048 | IR |
| 15 | 71 | 4 | - 0,037 | 0,033 | 0,050 | IR |
| 16 | 76 | 6 | + 0,055 | 0,009 | 0,055 | IR |
| 17 | 81 | 7 | + 0,056 | 0,010 | 0,057 | IR |
| 18 | 86 | 18 | + 0,056 | 0,011 | 0,057 | IR |
| 19 | 90 | 11 | + 0,076 | 0,016 | 0,078 | IR |
| 20 | 95 | 12 | - 0,080 | 0,011 | 0,081 | IR |
| 21 | 100 | 1 | - 0,136 | 0,017 | 0,137 | IR |

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 $\bar{d}$ 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 20 sets of results send by 20 laboratories using routine method ISO 9622|IDF 141, after outlier discarging using Grubbs test at 5 % risk level

Other* : ASU L 01.00-78 IR-mesurement

(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.($d^2$ + $Sd^2$))

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 %) | $Sr_{PT}$ | 0,007 |
| Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) | $SR_{PT}$ | 0,052 |

**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 | 0,020 | 0,010 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,010 | 0,030 * | 0,010 | 0,010 | 20 |
| 2 | 0,010 | 0,010 | 0,010 | 0,020 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,020 | 0,009 | 20 |
| 3 | 0,010 | 0,000 | 0,000 | 0,010 | 0,010 | 0,010 | 0,010 | 0,000 | 0,000 | 0,010 | 0,005 | 20 |
| 4 | 0,020 | 0,020 | 0,010 | 0,000 | 0,010 | 0,010 | 0,000 | 0,010 | 0,000 | 0,000 | 0,008 | 20 |
| 5 | 0,000 | 0,010 | 0,000 | 0,010 | 0,010 | 0,010 | 0,010 | 0,000 | 0,000 | 0,020 | 0,007 | 20 |
| 6 | 0,010 | 0,010 | 0,010 | 0,010 | 0,000 | 0,020 | 0,010 | 0,000 | 0,010 | 0,010 | 0,007 | 20 |
| 7 | 0,000 | 0,010 | 0,005 | 0,015 | 0,020 | 0,020 | 0,020 | 0,020 | 0,010 | 0,020 | 0,011 | 20 |
| 8 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,002 | 20 |
| 9 | 0,010 | 0,000 | 0,010 | 0,010 | 0,010 | 0,000 | 0,000 | 0,000 | 0,010 | 0,020 | 0,007 | 20 |
| 10 | 0,010 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,003 | 20 |
| 11 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,000 | 0,003 | 20 |
| 12 | 0,000 | 0,000 | 0,010 | 0,010 | 0,000 | 0,000 | 0,020 | 0,000 | 0,000 | 0,010 | 0,006 | 20 |
| 13 | 0,000 | 0,000 | 0,000 | 0,010 | 0,010 | 0,000 | 0,010 | 0,000 | 0,010 | 0,000 | 0,004 | 20 |
| 14 | 0,000 | 0,005 | 0,005 | 0,005 | 0,010 | 0,005 | 0,005 | 0,005 | 0,005 | 0,005 | 0,004 | 20 |
| 15 | 0,010 | 0,010 | 0,010 | 0,000 | 0,020 | 0,010 | 0,010 | 0,010 | 0,020 | 0,010 | 0,009 | 20 |
| 16 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,010 | 0,010 | 0,000 | 0,006 | 20 |
| 17 | 0,010 | 0,000 | 0,000 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,000 | 0,000 | 0,005 | 20 |
| 18 | 0,000 | 0,000 | 0,010 | 0,020 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,010 | 0,005 | 20 |
| 19 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,010 | 0,000 | 0,000 | 0,000 | 0,010 | 0,005 | 20 |
| 20 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,005 | 20 |
| 21 | 0,010 | 0,000 | 0,010 | 0,000 | 0,020 | 0,000 | 0,000 | 0,020 | 0,020 | 0,020 | 0,009 | 20 |
| Sr | 0,007 | 0,006 | 0,006 | 0,007 | 0,008 | 0,006 | 0,006 | 0,006 | 0,007 | 0,008 | | 420 |
| NE | 42 | 42 | 42 | 42 | 42 | 42 | 42 | 42 | 42 | 42 | | |
| L | 0,027 | 0,023 | 0,023 | 0,028 | 0,030 | 0,025 | 0,026 | 0,023 | 0,024 | 0,032 | | |

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 betwen two replicates=0,040 according ISO 9622|IDF 141

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

| Sample/Lab Code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 3,600 | 3,425 | 3,135 | 2,775 | 2,585 | 3,500 | 3,325 * | 2,955 | 2,675 | 3,115 |
| 2 | 3,695 | 3,535 | 3,245 | 2,900 | 2,725 | 3,605 | 3,445 | 3,075 | 2,825 | 3,300 |
| 3 | 3,765 | 3,560 | 3,260 | 2,885 | 2,675 | 3,665 | 3,475 | 3,070 | 2,780 | 3,255 |
| 4 | 3,760 | 3,570 | 3,235 | 2,830 | 2,605 | 3,655 | 3,460 | 3,025 | 2,720 | 3,220 |
| 5 | 3,780 | 3,605 | 3,300 | 2,935 | 2,735 | 3,685 | 3,495 | 3,110 | 2,830 | 3,290 |
| 6 | 3,805 | 3,615 | 3,325 | 2,955 | 2,760 | 3,700 | 3,525 | 3,140 | 2,865 | 3,305 |
| 7 | 3,810 | 3,615 | 3,328 | 2,963 | 2,760 | 3,700 | 3,520 | 3,140 | 2,865 | 3,310 |
| 8 | 3,810 | 3,615 | 3,310 | 2,930 | 2,720 | 3,700 | 3,510 | 3,110 | 2,830 | 3,290 |
| 9 | 3,745 | 3,560 | 3,255 | 2,885 | 2,685 | 3,640 | 3,460 | 3,070 | 2,795 | 3,250 |
| 10 | 3,705 | 3,520 | 3,220 | 2,855 | 2,660 | 3,580 | 3,430 | 3,040 | 2,760 | 3,220 |
| 11 | 3,860 | 3,660 | 3,345 | 2,960 | 2,750 | 3,745 | 3,560 | 3,150 | 2,850 | 3,330 |
| 12 | 3,660 | 3,480 | 3,195 | 2,825 | 2,620 | 3,570 | 3,390 | 3,010 | 2,730 | 3,165 |
| 13 | 3,780 | 3,590 | 3,290 | 2,895 | 2,695 | 3,680 | 3,495 | 3,090 | 2,795 | 3,280 |
| 14 | 3,795 | 3,613 | 3,308 | 2,938 | 2,745 | 3,693 | 3,498 | 3,118 | 2,848 | 3,303 |
| 15 | 3,705 | 3,525 | 3,245 | 2,880 | 2,690 | 3,615 | 3,435 | 3,055 | 2,810 | 3,255 |
| 16 | 3,795 | 3,605 | 3,295 | 2,915 | 2,705 | 3,690 | 3,495 | 3,105 | 2,815 | 3,290 |
| 17 | 3,735 | 3,540 | 3,230 | 2,845 | 2,635 | 3,635 | 3,440 | 3,035 | 2,740 | 3,180 |
| 18 | 3,810 | 3,640 | 3,335 | 2,940 | 2,730 | 3,730 | 3,540 | 3,130 | 2,850 | 3,305 |
| 19 | 3,715 | 3,535 | 3,245 | 2,890 | 2,675 | 3,625 | 3,450 | 3,070 | 2,800 | 3,235 |
| 20 | 3,765 | 3,575 | 3,275 | 2,890 | 2,685 | 3,670 | 3,485 | 3,080 | 2,790 | 3,270 |
| 21 | 3,735 | 3,550 | 3,265 | 2,910 | 2,710 | 3,620 | 3,450 | 3,080 | 2,810 | 3,250 |
| M | 3,754 | 3,568 | 3,269 | 2,895 | 2,693 | 3,653 | 3,478 | 3,079 | 2,799 | 3,258 |
| REF. | 3,757 | 3,570 | 3,270 | 2,895 | 2,691 | 3,655 | 3,480 | 3,079 | 2,798 | 3,256 |
| SD | 0,060 | 0,055 | 0,051 | 0,049 | 0,050 | 0,058 | 0,042 | 0,048 | 0,051 | 0,054 |

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 20 laboratories using the Routine method ISO 9622|IDF 141, after outliers discargingd using Grubbs test at 5 % risk level.

**Table IV :** Outlier identification

| **Sample** | **1** | **2** | **3** | **4** | **5** | **6** | **7** | **8** | **9** | **10** |
|---|---|---|---|---|---|---|---|---|---|---|
| **Outliers Cochran** | | | | | | | | | 1 | |
| **Outlier Grubbs** | | | | | | | 1 | | | |
| **sr** | 0,007 | 0,006 | 0,006 | 0,007 | 0,008 | 0,006 | 0,006 | 0,006 | 0,006 | 0,008 |
| **SR** | 0,060 | 0,056 | 0,051 | 0,049 | 0,050 | 0,058 | 0,042 | 0,048 | 0,043 | 0,054 |

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

| Sample / Lab Code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | d | $Sd_{lab}$ | t |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | - 0,157 | - 0,145 | - 0,135 | - 0,120 | - 0,106 | - 0,155 | - 0,155 | - 0,124 | - 0,123 | - 0,141 | - 0,136 | 0,017 | 24,87 |
| 2 | - 0,062 | - 0,035 | - 0,025 | + 0,005 | + 0,034 | - 0,050 | - 0,035 | - 0,004 | + 0,027 | + 0,044 | - 0,010 | 0,037 | 0,86 |
| 3 | + 0,008 | - 0,010 | - 0,010 | - 0,010 | - 0,016 | + 0,010 | - 0,005 | - 0,009 | - 0,018 | - 0,001 | - 0,006 | 0,009 | 2,02 |
| 4 | + 0,003 | + 0,000 | - 0,035 | - 0,065 | - 0,086 | + 0,000 | - 0,020 | - 0,054 | - 0,078 | - 0,036 | - 0,037 | 0,033 | 3,53 |
| 5 | + 0,023 | + 0,035 | + 0,030 | + 0,040 | + 0,044 | + 0,030 | + 0,015 | + 0,031 | + 0,032 | + 0,034 | + 0,032 | 0,008 | 12,47 |
| 6 | + 0,048 | + 0,045 | + 0,055 | + 0,060 | + 0,069 | + 0,045 | + 0,045 | + 0,061 | + 0,067 | + 0,049 | + 0,055 | 0,009 | 18,75 |
| 7 | + 0,053 | + 0,045 | + 0,058 | + 0,067 | + 0,069 | + 0,045 | + 0,040 | + 0,061 | + 0,067 | + 0,054 | + 0,056 | 0,010 | 17,36 |
| 8 | + 0,053 | + 0,045 | + 0,040 | + 0,035 | + 0,029 | + 0,045 | + 0,030 | + 0,031 | + 0,032 | + 0,034 | + 0,038 | 0,008 | 14,65 |
| 9 | - 0,012 | - 0,010 | - 0,015 | - 0,010 | - 0,006 | - 0,015 | - 0,020 | - 0,009 | - 0,003 | - 0,006 | - 0,010 | 0,005 | 6,69 |
| 10 | - 0,052 | - 0,050 | - 0,050 | - 0,040 | - 0,031 | - 0,075 | - 0,050 | - 0,039 | - 0,038 | - 0,036 | - 0,046 | 0,012 | 11,75 |
| 11 | + 0,103 | + 0,090 | + 0,075 | + 0,065 | + 0,059 | + 0,090 | + 0,080 | + 0,071 | + 0,052 | + 0,074 | + 0,076 | 0,016 | 15,44 |
| 12 | - 0,097 | - 0,090 | - 0,075 | - 0,070 | - 0,071 | - 0,085 | - 0,090 | - 0,069 | - 0,068 | - 0,091 | - 0,080 | 0,011 | 23,25 |
| 13 | + 0,023 | + 0,020 | + 0,020 | - 0,000 | + 0,004 | + 0,025 | + 0,015 | + 0,011 | - 0,003 | + 0,024 | + 0,014 | 0,010 | 4,23 |
| 14 | + 0,038 | + 0,043 | + 0,038 | + 0,042 | + 0,054 | + 0,038 | + 0,018 | + 0,038 | + 0,050 | + 0,047 | + 0,041 | 0,010 | 13,25 |
| 15 | - 0,052 | - 0,045 | - 0,025 | - 0,015 | - 0,001 | - 0,040 | - 0,045 | - 0,024 | + 0,012 | - 0,001 | - 0,023 | 0,022 | 3,39 |
| 16 | + 0,038 | + 0,035 | + 0,025 | + 0,020 | + 0,014 | + 0,035 | + 0,015 | + 0,026 | + 0,017 | + 0,034 | + 0,026 | 0,009 | 8,95 |
| 17 | - 0,022 | - 0,030 | - 0,040 | - 0,050 | - 0,056 | - 0,020 | - 0,040 | - 0,044 | - 0,058 | - 0,076 | - 0,043 | 0,017 | 7,93 |
| 18 | + 0,053 | + 0,070 | + 0,065 | + 0,045 | + 0,039 | + 0,075 | + 0,060 | + 0,051 | + 0,052 | + 0,049 | + 0,056 | 0,011 | 15,43 |
| 19 | - 0,042 | - 0,035 | - 0,025 | - 0,005 | - 0,016 | - 0,030 | - 0,030 | - 0,009 | + 0,002 | - 0,021 | - 0,021 | 0,014 | 4,77 |
| 20 | + 0,008 | + 0,005 | + 0,005 | - 0,005 | - 0,006 | + 0,015 | + 0,005 | + 0,001 | - 0,008 | + 0,014 | + 0,004 | 0,008 | 1,38 |
| 21 | - 0,022 | - 0,020 | - 0,005 | + 0,015 | + 0,019 | - 0,035 | - 0,030 | + 0,001 | + 0,012 | - 0,006 | - 0,007 | 0,019 | 1,16 |
| d | - 0,003 | - 0,002 | - 0,001 | + 0,000 | + 0,002 | - 0,002 | - 0,002 | - 0,000 | + 0,001 | + 0,002 | - 0,001 | 0,052 | |
| Sd | 0,060 | 0,055 | 0,051 | 0,049 | 0,050 | 0,058 | 0,042 | 0,048 | 0,051 | 0,054 | 0,052 | | |

d = mean of differences  Sd = standard deviation of differences  t = Student test - comparison to 0

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

**ISO 9622|IDF141 : Precision of the method :** Sr = 0.014 g / 100 g
SR =0,04 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 | -2,62 | -2,62 | -2,63 | -2,45 | -2,12 | -2,66 | -3,68 | -2,57 | -2,43 | -2,60 |
| 2 | -1,03 | -0,63 | -0,48 | +0,10 | +0,67 | -0,86 | -0,82 | -0,09 | +0,54 | +0,82 |
| 3 | +0,14 | -0,18 | -0,19 | -0,20 | -0,32 | +0,17 | -0,11 | -0,19 | -0,35 | -0,02 |
| 4 | +0,05 | +0,00 | -0,68 | -1,33 | -1,72 | +0,00 | -0,47 | -1,12 | -1,54 | -0,66 |
| 5 | +0,39 | +0,63 | +0,59 | +0,82 | +0,87 | +0,52 | +0,37 | +0,64 | +0,63 | +0,63 |
| 6 | +0,81 | +0,82 | +1,08 | +1,23 | +1,37 | +0,77 | +1,08 | +1,26 | +1,33 | +0,91 |
| 7 | +0,89 | +0,82 | +1,13 | +1,38 | +1,37 | +0,77 | +0,96 | +1,26 | +1,33 | +1,00 |
| 8 | +0,89 | +0,82 | +0,79 | +0,72 | +0,57 | +0,77 | +0,72 | +0,64 | +0,63 | +0,63 |
| 9 | -0,20 | -0,18 | -0,29 | -0,20 | -0,12 | -0,26 | -0,47 | -0,19 | -0,06 | -0,11 |
| 10 | -0,87 | -0,90 | -0,97 | -0,82 | -0,62 | -1,29 | -1,18 | -0,81 | -0,75 | -0,66 |
| 11 | +1,73 | +1,63 | +1,47 | +1,33 | +1,17 | +1,55 | +1,91 | +1,47 | +1,03 | +1,37 |
| 12 | -1,62 | -1,62 | -1,46 | -1,43 | -1,42 | -1,46 | -2,13 | -1,43 | -1,34 | -1,68 |
| 13 | +0,39 | +0,36 | +0,40 | -0,00 | +0,07 | +0,43 | +0,37 | +0,23 | -0,06 | +0,45 |
| 14 | +0,64 | +0,77 | +0,74 | +0,87 | +1,07 | +0,65 | +0,43 | +0,79 | +0,98 | +0,86 |
| 15 | -0,87 | -0,81 | -0,48 | -0,31 | -0,02 | -0,68 | -1,06 | -0,50 | +0,24 | -0,02 |
| 16 | +0,64 | +0,63 | +0,49 | +0,41 | +0,27 | +0,60 | +0,37 | +0,54 | +0,34 | +0,63 |
| 17 | -0,36 | -0,54 | -0,78 | -1,02 | -1,12 | -0,34 | -0,94 | -0,91 | -1,14 | -1,40 |
| 18 | +0,89 | +1,27 | +1,27 | +0,92 | +0,77 | +1,29 | +1,44 | +1,05 | +1,03 | +0,91 |
| 19 | -0,70 | -0,63 | -0,48 | -0,10 | -0,32 | -0,51 | -0,70 | -0,19 | +0,04 | -0,39 |
| 20 | +0,14 | +0,09 | +0,10 | -0,10 | -0,12 | +0,26 | +0,13 | +0,02 | -0,16 | +0,26 |
| 21 | -0,36 | -0,36 | -0,09 | +0,31 | +0,37 | -0,60 | -0,70 | +0,02 | +0,24 | -0,11 |

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

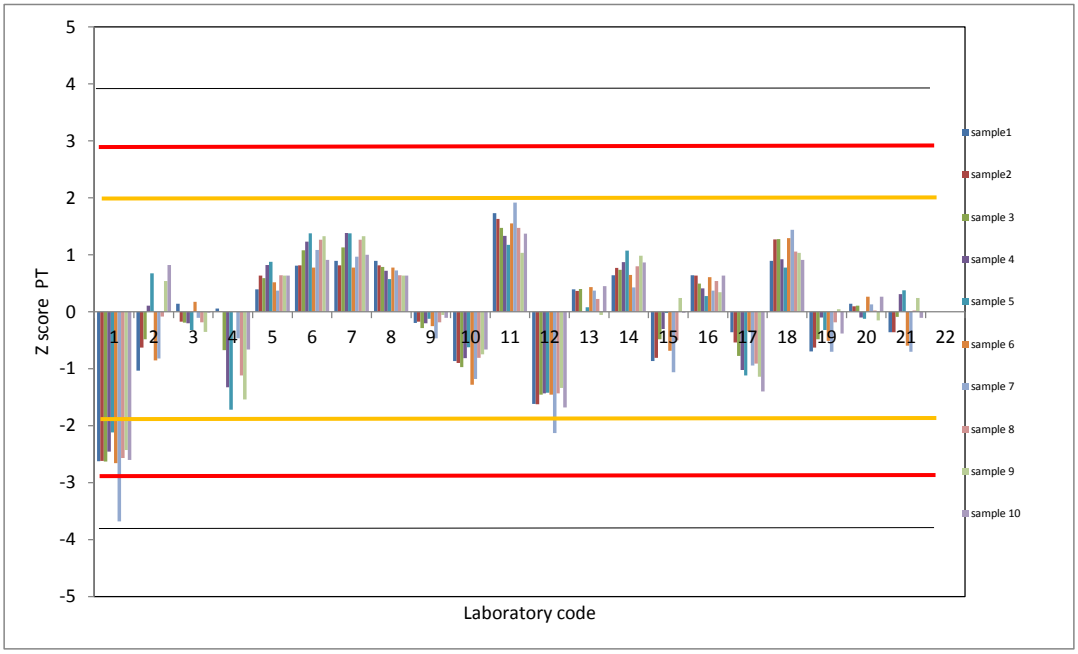


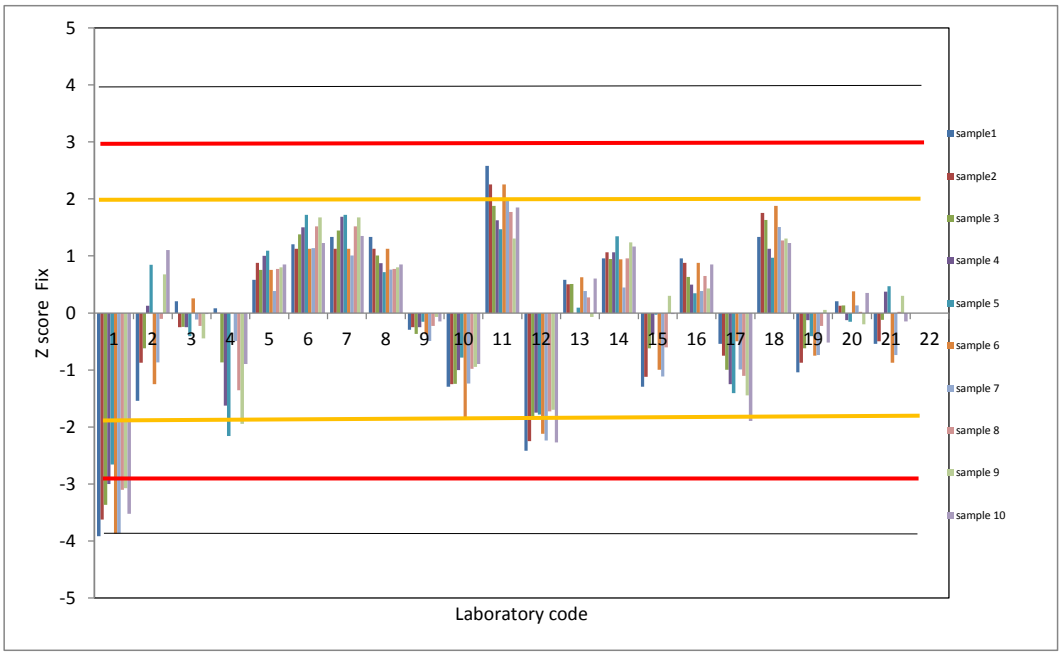
**Table VII :** **Zscore of the different laboratories for each sample.**
**ZS calculated on the standard deviation of reproducibility of the method**

| Sample / Lab code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | -3,92 | -3,62 | -3,37 | -3,00 | -2,66 | -3,87 | -3,87 | -3,10 | -3,07 | -3,52 |
| 2 | -1,54 | -0,87 | -0,62 | +0,12 | +0,84 | -1,25 | -0,87 | -0,10 | +0,68 | +1,10 |
| 3 | +0,21 | -0,25 | -0,24 | -0,25 | -0,41 | +0,25 | -0,12 | -0,23 | -0,45 | -0,02 |
| 4 | +0,08 | +0,00 | -0,87 | -1,63 | -2,16 | +0,00 | -0,49 | -1,35 | -1,95 | -0,90 |
| 5 | +0,58 | +0,88 | +0,76 | +1,00 | +1,09 | +0,75 | +0,38 | +0,77 | +0,80 | +0,85 |
| 6 | +1,21 | +1,13 | +1,38 | +1,50 | +1,72 | +1,13 | +1,13 | +1,52 | +1,68 | +1,23 |
| 7 | +1,33 | +1,13 | +1,44 | +1,69 | +1,72 | +1,13 | +1,01 | +1,52 | +1,68 | +1,35 |
| 8 | +1,33 | +1,13 | +1,01 | +0,87 | +0,72 | +1,13 | +0,76 | +0,77 | +0,80 | +0,85 |
| 9 | -0,29 | -0,25 | -0,37 | -0,25 | -0,16 | -0,37 | -0,49 | -0,23 | -0,07 | -0,15 |
| 10 | -1,29 | -1,25 | -1,24 | -1,00 | -0,78 | -1,87 | -1,24 | -0,98 | -0,95 | -0,90 |
| 11 | +2,58 | +2,25 | +1,88 | +1,62 | +1,47 | +2,25 | +2,01 | +1,77 | +1,30 | +1,85 |
| 12 | -2,42 | -2,25 | -1,87 | -1,75 | -1,78 | -2,12 | -2,24 | -1,73 | -1,70 | -2,27 |
| 13 | +0,58 | +0,50 | +0,51 | -0,00 | +0,09 | +0,63 | +0,38 | +0,27 | -0,07 | +0,60 |
| 14 | +0,96 | +1,07 | +0,94 | +1,06 | +1,34 | +0,94 | +0,45 | +0,96 | +1,24 | +1,17 |
| 15 | -1,29 | -1,12 | -0,62 | -0,38 | -0,03 | -1,00 | -1,12 | -0,60 | +0,30 | -0,02 |
| 16 | +0,96 | +0,88 | +0,63 | +0,50 | +0,34 | +0,88 | +0,38 | +0,65 | +0,43 | +0,85 |
| 17 | -0,54 | -0,75 | -0,99 | -1,25 | -1,41 | -0,50 | -0,99 | -1,10 | -1,45 | -1,90 |
| 18 | +1,33 | +1,75 | +1,63 | +1,13 | +0,97 | +1,88 | +1,51 | +1,27 | +1,30 | +1,23 |
| 19 | -1,04 | -0,87 | -0,62 | -0,13 | -0,41 | -0,75 | -0,74 | -0,23 | +0,05 | -0,52 |
| 20 | +0,21 | +0,13 | +0,13 | -0,13 | -0,16 | +0,38 | +0,13 | +0,02 | -0,20 | +0,35 |
| 21 | -0,54 | -0,50 | -0,12 | +0,37 | +0,47 | -0,87 | -0,74 | +0,02 | +0,30 | -0,15 |

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=0,040

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



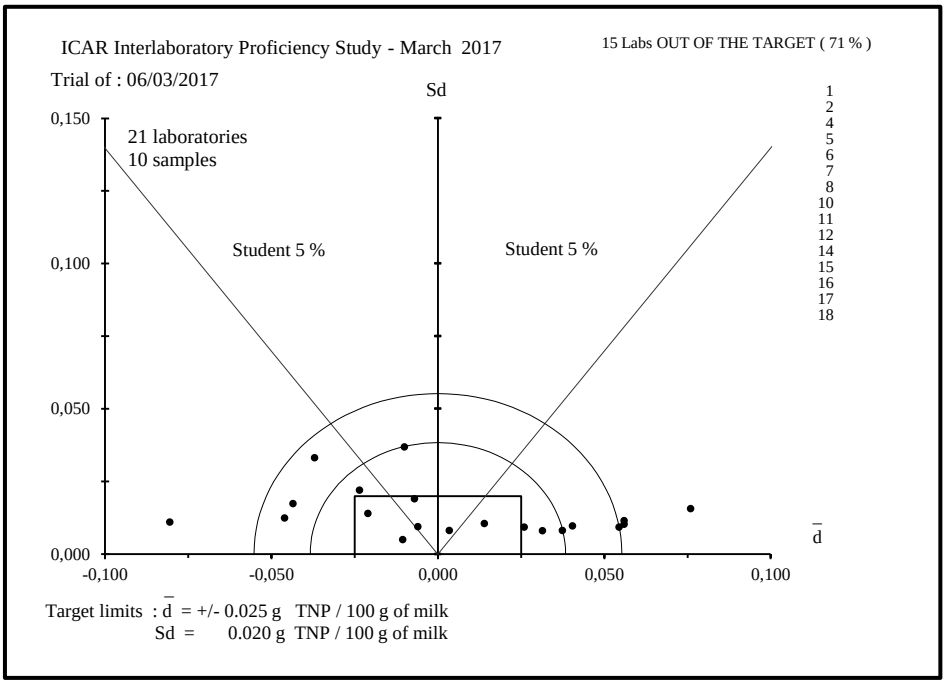



**Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).**

LIST OF ICAR PARTICIPANTS
RAW MILK
PROTEIN ROUTINE METHOD
MARCH 2017

| | | |
|---|---|---|
| AIA-LAB. STANDARD LATTE | MACCARESE (ROMA) | ITALY |
| CATTLE INFORMATION SERVICE (CIS) | TELFORD | UNITED KINGDOM |
| COOPRINSEM | OSORNO | CHILE |
| CROATIAN AGRICULTURAL AGENCY MILK QUALITY CONTROL | KRIZEVCI | CROATIA |
| EASTERN LAB SERVICES | MEDINA | USA |
| EUROFINS STEINS LABORATORY A/B | VEJEN | DENMARK |
| EUROFINS STEINS LABORATORY A/B | JÖNGÖPING | SWEDEN |
| FED.LATTERIE SOCIALI DI BOLZANO | BOLZANO | ITALY |
| LAB. POLJOPRIVREDNI | NOVI SAD | SERBIA |
| LOM KCHZ LABORATORIUM REFERENCYJNE z/s w | PARZNIEWIE | POLAND |
| MERIEUX NUTRISCIENCE | CAPE TOWN | SOUTH AFRICA |
| MERIEUX NUTRISCIENCE | MIDRAND | SOUTH AFRICA |
| MERIEUX NUTRISCIENCE J BAY | CAPE TOWN | SOUTH AFRICA |
| MILCHPRUFRING BADEN-WURTTEMBERG E.V. | KIRCHEIM / TECK | GERMANY |
| SHANGAI DAIRY BREEDING CENTER Co.Ltd | SHANGHAI | CHINA |
| TAIWAN LIVESTOCK ESEARCH INT. | TAIWAN | TAIWAN |
| VETLAB AGRICULTURAL SHOWGROUNDS | LUSAKA | ZAMBIA |

# ICAR
# PROFICIENCY TESTING SCHEME

---

## March 2017

## Raw Milk

## Determination of LACTOSE CONTENT
### Routine method

Sending date of statistical treatment : 21th April 2017


| | | |
|---|---|---|
| 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 |

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

| Nb | % | N° | d | Sd | D | Method |
|---|---|---|---|---|---|---|
| 1 | 6 | 14 | - 0,019 | 0,010 | 0,021 | IR |
| 2 | 11 | 4 | - 0,007 | 0,021 | 0,022 | IR |
| 3 | 17 | 18 | - 0,029 | 0,010 | 0,030 | IR |
| 4 | 22 | 10 | - 0,007 | 0,030 | 0,031 | IR |
| 5 | 28 | 13 | - 0,034 | 0,026 | 0,043 | IR |
| 6 | 33 | 17 | + 0,038 | 0,029 | 0,048 | IR |
| 7 | 39 | 3 | + 0,008 | 0,055 | 0,056 | IR |
| 8 | 44 | 11 | - 0,066 | 0,009 | 0,067 | IR |
| 9 | 50 | 5 | - 0,068 | 0,010 | 0,068 | IR |
| 10 | 56 | 15 | + 0,080 | 0,006 | 0,080 | IR |
| 11 | 61 | 9 | - 0,081 | 0,009 | 0,082 | IR |
| 12 | 67 | 8 | + 0,094 | 0,021 | 0,096 | IR |
| 13 | 72 | 2 | - 0,098 | 0,011 | 0,099 | Other* |
| 14 | 78 | 1 | - 0,104 | 0,023 | 0,107 | IR |
| 15 | 83 | 7 | + 0,108 | 0,007 | 0,108 | IR |
| 16 | 89 | 16 | + 0,115 | 0,008 | 0,115 | IR |
| 17 | 94 | 12 | + 0,120 | 0,010 | 0,120 | IR |
| 18 | 100 | 6 | - 0,149 | 0,006 | 0,149 | IR |

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 :

$$+/- 0.100 \text{ g / 100 g for } \bar{d} \text{ and } 0.100 \text{ g / 100g for Sd}$$

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 17 sets of results send by 17 laboratories using routine method ISO 9622|IDF 141, after outlier discarging using Grubbs test at 5 % risk level

Other* : ASU L 01.00-78 IR-mesurement

(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.($d^2$ + $Sd^2$))

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 %) | $Sr_{PT}$ | 0,007 |
| Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) | $SR_{PT}$ | 0,085 |

**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 | 0,010 | 0,020 | 0,010 | 0,010 | 0,010 | 0,020 | 0,000 | 0,010 | 0,010 | 0,010 | 0,009 | 20 |
| 2 | 0,020 | 0,010 | 0,010 | 0,010 | 0,020 | 0,020 | 0,010 | 0,010 | 0,020 | 0,020 | 0,011 | 20 |
| 3 | 0,010 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,006 | 20 |
| 4 | 0,010 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,020 | 0,000 | 0,005 | 20 |
| 5 | 0,001 | 0,000 | 0,010 | 0,010 | 0,000 | 0,011 | 0,000 | 0,011 | 0,011 | 0,011 | 0,006 | 20 |
| 6 | 0,010 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,000 | 0,010 | 0,010 | 0,000 | 0,006 | 20 |
| 7 | 0,010 | 0,010 | 0,010 | 0,020 | 0,020 | 0,020 | 0,000 | 0,010 | 0,000 | 0,010 | 0,009 | 20 |
| 8 | 0,020 | 0,030 | 0,010 | 0,020 | 0,000 | 0,000 | 0,005 | 0,000 | 0,005 | 0,003 | 0,010 | 20 |
| 9 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,002 | 20 |
| 10 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,010 | 0,010 | 0,000 | 0,010 | 0,010 | 0,004 | 20 |
| 11 | 0,010 | 0,010 | 0,000 | 0,000 | 0,000 | 0,010 | 0,020 | 0,010 | 0,000 | 0,010 | 0,007 | 20 |
| 12 | 0,010 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,010 | 0,010 | 0,000 | 0,010 | 0,005 | 20 |
| 13 | 0,010 | 0,020 | 0,010 | 0,010 | 0,000 | 0,000 | 0,010 | 0,010 | 0,010 | 0,010 | 0,007 | 20 |
| 14 | 0,010 | 0,010 | 0,010 | 0,000 | 0,000 | 0,000 | 0,010 | 0,010 | 0,000 | 0,020 | 0,007 | 20 |
| 15 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,007 | 20 |
| 16 | 0,010 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,010 | 0,000 | 0,010 | 0,000 | 0,006 | 20 |
| 17 | 0,000 | 0,000 | 0,010 | 0,010 | 0,010 | 0,010 | 0,020 | 0,010 | 0,020 | 0,010 | 0,008 | 20 |
| 18 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 20 |
| Sr | 0,007 | 0,008 | 0,006 | 0,007 | 0,006 | 0,007 | 0,007 | 0,006 | 0,007 | 0,007 | | 360 |
| NE | 36 | 36 | 36 | 36 | 36 | 36 | 36 | 36 | 36 | 36 | | |
| L | 0,028 | 0,032 | 0,024 | 0,027 | 0,021 | 0,029 | 0,026 | 0,023 | 0,028 | 0,028 | | |

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 betwen two replicates=0,040 according ISO 9622|IDF 141

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

| Sample / Lab code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 4,965 | 5,010 | 4,865 | 4,895 | 4,785 | 4,810 | 4,740 | 4,755 | 4,625 | 4,715 |
| 2 | 5,000 | 5,055 | 4,865 | 4,935 | 4,780 | 4,800 | 4,725 | 4,745 | 4,610 | 4,710 |
| 3 | 5,035 | 5,085 | 4,935 | 4,995 | 4,890 | 4,895 | 4,865 | 4,875 | 4,840 | 4,875 |
| 4 | 5,105 | 5,170 | 4,965 | 5,030 | 4,860 | 4,890 | 4,810 | 4,830 | 4,680 | 4,800 |
| 5 | 5,029 | 5,092 | 4,897 | 4,960 | 4,810 | 4,824 | 4,734 | 4,771 | 4,687 | 4,729 |
| 6 | 4,945 | 5,015 | 4,815 | 4,875 | 4,720 | 4,745 | 4,670 | 4,685 | 4,595 | 4,650 |
| 7 | 5,205 | 5,275 | 5,065 | 5,140 | 4,980 | 5,000 | 4,920 | 4,945 | 4,850 | 4,905 |
| 8 | 5,210 | 5,275 | 5,055 | 5,130 | 4,960 | 4,990 | 4,908 | 4,940 | 4,783 | 4,892 |
| 9 | 5,020 | 5,080 | 4,880 | 4,950 | 4,790 | 4,820 | 4,730 | 4,755 | 4,660 | 4,710 |
| 10 | 5,120 | 5,210 | 4,960 | 5,030 | 4,860 | 4,885 | 4,785 | 4,810 | 4,705 | 4,775 |
| 11 | 5,035 | 5,105 | 4,890 | 4,960 | 4,810 | 4,835 | 4,740 | 4,775 | 4,660 | 4,735 |
| 12 | 5,215 | 5,290 | 5,075 | 5,150 | 5,000 | 5,010 | 4,925 | 4,955 | 4,870 | 4,915 |
| 13 | 5,035 | 5,080 | 4,905 | 4,975 | 4,850 | 4,860 | 4,815 | 4,835 | 4,695 | 4,815 |
| 14 | 5,065 | 5,155 | 4,945 | 5,010 | 4,860 | 4,880 | 4,785 | 4,815 | 4,720 | 4,780 |
| 15 | 5,165 | 5,245 | 5,035 | 5,105 | 4,945 | 4,975 | 4,895 | 4,925 | 4,830 | 4,885 |
| 16 | 5,215 | 5,285 | 5,075 | 5,145 | 4,980 | 5,005 | 4,925 | 4,950 | 4,855 | 4,920 |
| 17 | 5,100 | 5,160 | 4,985 | 5,035 | 4,915 | 4,925 | 4,900 | 4,915 | 4,770 | 4,885 |
| 18 | 5,070 | 5,140 | 4,940 | 5,000 | 4,840 | 4,870 | 4,780 | 4,810 | 4,710 | 4,760 |
| M | 5,085 | 5,152 | 4,953 | 5,018 | 4,869 | 4,890 | 4,814 | 4,838 | 4,730 | 4,803 |
| REF. | 5,090 | 5,157 | 4,958 | 5,023 | 4,874 | 4,895 | 4,819 | 4,844 | 4,737 | 4,809 |
| SD | 0,087 | 0,093 | 0,081 | 0,086 | 0,081 | 0,080 | 0,083 | 0,084 | 0,090 | 0,086 |

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 17 laboratories using the Routine method ISO 9622|IDF 141 , after outliers discarging using Grubbs test at 5 % risk level.

**Table IV :** Outlier identification

| **Sample** | **1** | **2** | **3** | **4** | **5** | **6** | **7** | **8** | **9** | **10** |
|---|---|---|---|---|---|---|---|---|---|---|
| **Outliers Cochran** | | | | | | | | | | |
| **Outlier Grubbs** | | | | | | | | | | |
| **sr** | 0,007 | 0,008 | 0,006 | 0,007 | 0,006 | 0,007 | 0,007 | 0,006 | 0,007 | 0,007 |
| **SR** | 0,087 | 0,094 | 0,081 | 0,086 | 0,081 | 0,080 | 0,083 | 0,084 | 0,090 | 0,086 |

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

| Sample / Lab Code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | d | $Sd_{lab}$ | t |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | - 0,125 | - 0,147 | - 0,093 | - 0,128 | - 0,089 | - 0,085 | - 0,079 | - 0,089 | - 0,112 | - 0,094 | - 0,104 | 0,023 | 14,56 |
| 2 | - 0,090 | - 0,102 | - 0,093 | - 0,088 | - 0,094 | - 0,095 | - 0,094 | - 0,099 | - 0,127 | - 0,099 | - 0,098 | 0,011 | 27,95 |
| 3 | - 0,055 | - 0,072 | - 0,023 | - 0,028 | + 0,016 | - 0,000 | + 0,046 | + 0,031 | + 0,103 | + 0,066 | + 0,008 | 0,055 | 0,48 |
| 4 | + 0,015 | + 0,013 | + 0,007 | + 0,007 | - 0,014 | - 0,005 | - 0,009 | - 0,014 | - 0,057 | - 0,009 | - 0,007 | 0,021 | 1,00 |
| 5 | - 0,062 | - 0,065 | - 0,061 | - 0,063 | - 0,064 | - 0,072 | - 0,085 | - 0,073 | - 0,051 | - 0,080 | - 0,068 | 0,010 | 21,14 |
| 6 | - 0,145 | - 0,142 | - 0,143 | - 0,148 | - 0,154 | - 0,150 | - 0,149 | - 0,159 | - 0,142 | - 0,159 | - 0,149 | 0,006 | 75,01 |
| 7 | + 0,115 | + 0,118 | + 0,107 | + 0,117 | + 0,106 | + 0,105 | + 0,101 | + 0,101 | + 0,113 | + 0,096 | + 0,108 | 0,007 | 45,84 |
| 8 | + 0,120 | + 0,118 | + 0,097 | + 0,107 | + 0,086 | + 0,095 | + 0,088 | + 0,096 | + 0,045 | + 0,083 | + 0,094 | 0,021 | 14,00 |
| 9 | - 0,070 | - 0,077 | - 0,078 | - 0,073 | - 0,084 | - 0,075 | - 0,089 | - 0,089 | - 0,077 | - 0,099 | - 0,081 | 0,009 | 29,06 |
| 10 | + 0,030 | + 0,053 | + 0,002 | + 0,007 | - 0,014 | - 0,010 | - 0,034 | - 0,034 | - 0,032 | - 0,034 | - 0,007 | 0,030 | 0,70 |
| 11 | - 0,055 | - 0,052 | - 0,068 | - 0,063 | - 0,064 | - 0,060 | - 0,079 | - 0,069 | - 0,077 | - 0,074 | - 0,066 | 0,009 | 23,24 |
| 12 | + 0,125 | + 0,133 | + 0,117 | + 0,127 | + 0,126 | + 0,115 | + 0,106 | + 0,111 | + 0,133 | + 0,106 | + 0,120 | 0,010 | 37,09 |
| 13 | - 0,055 | - 0,077 | - 0,053 | - 0,048 | - 0,024 | - 0,035 | - 0,004 | - 0,009 | - 0,042 | + 0,006 | - 0,034 | 0,026 | 4,11 |
| 14 | - 0,025 | - 0,002 | - 0,013 | - 0,013 | - 0,014 | - 0,015 | - 0,034 | - 0,029 | - 0,017 | - 0,029 | - 0,019 | 0,010 | 6,18 |
| 15 | + 0,075 | + 0,088 | + 0,077 | + 0,082 | + 0,071 | + 0,080 | + 0,076 | + 0,081 | + 0,093 | + 0,076 | + 0,080 | 0,006 | 39,20 |
| 16 | + 0,125 | + 0,128 | + 0,117 | + 0,122 | + 0,106 | + 0,110 | + 0,106 | + 0,106 | + 0,118 | + 0,111 | + 0,115 | 0,008 | 44,28 |
| 17 | + 0,010 | + 0,003 | + 0,027 | + 0,012 | + 0,041 | + 0,030 | + 0,081 | + 0,071 | + 0,033 | + 0,076 | + 0,038 | 0,029 | 4,26 |
| 18 | - 0,020 | - 0,017 | - 0,018 | - 0,023 | - 0,034 | - 0,025 | - 0,039 | - 0,034 | - 0,027 | - 0,049 | - 0,029 | 0,010 | 8,92 |
| d | - 0,005 | - 0,006 | - 0,005 | - 0,005 | - 0,005 | - 0,005 | - 0,005 | - 0,005 | - 0,007 | - 0,005 | - 0,005 | 0,083 | |
| Sd | 0,087 | 0,093 | 0,081 | 0,086 | 0,081 | 0,080 | 0,083 | 0,084 | 0,090 | 0,086 | 0,085 | | |

d = mean of differences  Sd = standard deviation of differences  t = Student test - comparison to 0

Upper limits : $\bar{d}$ = +/- 0.100 g / 100g  Sd = 0.100 g / 100g

**ISO 9622|IDF141 : Precision of the method :** Sr = 0.014 g / 100 g
SR =0,04 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 | -1,44 | -1,57 | -1,15 | -1,48 | -1,10 | -1,07 | -0,95 | -1,06 | -1,25 | -1,09 |
| 2 | -1,04 | -1,09 | -1,15 | -1,02 | -1,17 | -1,20 | -1,14 | -1,18 | -1,42 | -1,15 |
| 3 | -0,63 | -0,77 | -0,29 | -0,32 | +0,20 | -0,00 | +0,55 | +0,37 | +1,15 | +0,78 |
| 4 | +0,17 | +0,14 | +0,09 | +0,09 | -0,17 | -0,07 | -0,11 | -0,17 | -0,64 | -0,10 |
| 5 | -0,71 | -0,70 | -0,76 | -0,73 | -0,79 | -0,90 | -1,03 | -0,87 | -0,57 | -0,93 |
| 6 | -1,67 | -1,52 | -1,77 | -1,72 | -1,91 | -1,89 | -1,80 | -1,89 | -1,59 | -1,85 |
| 7 | +1,32 | +1,26 | +1,33 | +1,36 | +1,32 | +1,32 | +1,21 | +1,21 | +1,26 | +1,12 |
| 8 | +1,38 | +1,26 | +1,20 | +1,25 | +1,07 | +1,19 | +1,06 | +1,15 | +0,50 | +0,97 |
| 9 | -0,81 | -0,83 | -0,97 | -0,84 | -1,04 | -0,95 | -1,07 | -1,06 | -0,86 | -1,15 |
| 10 | +0,34 | +0,57 | +0,02 | +0,09 | -0,17 | -0,13 | -0,41 | -0,40 | -0,36 | -0,39 |
| 11 | -0,63 | -0,56 | -0,84 | -0,73 | -0,79 | -0,76 | -0,95 | -0,82 | -0,86 | -0,86 |
| 12 | +1,43 | +1,42 | +1,45 | +1,48 | +1,57 | +1,44 | +1,27 | +1,33 | +1,48 | +1,24 |
| 13 | -0,63 | -0,83 | -0,66 | -0,55 | -0,30 | -0,44 | -0,05 | -0,11 | -0,47 | +0,08 |
| 14 | -0,29 | -0,02 | -0,16 | -0,15 | -0,17 | -0,19 | -0,41 | -0,34 | -0,19 | -0,33 |
| 15 | +0,86 | +0,94 | +0,95 | +0,96 | +0,88 | +1,00 | +0,91 | +0,97 | +1,04 | +0,89 |
| 16 | +1,43 | +1,37 | +1,45 | +1,42 | +1,32 | +1,38 | +1,27 | +1,27 | +1,31 | +1,30 |
| 17 | +0,11 | +0,03 | +0,33 | +0,14 | +0,51 | +0,37 | +0,97 | +0,85 | +0,37 | +0,89 |
| 18 | -0,23 | -0,18 | -0,22 | -0,26 | -0,42 | -0,32 | -0,47 | -0,40 | -0,30 | -0,57 |

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

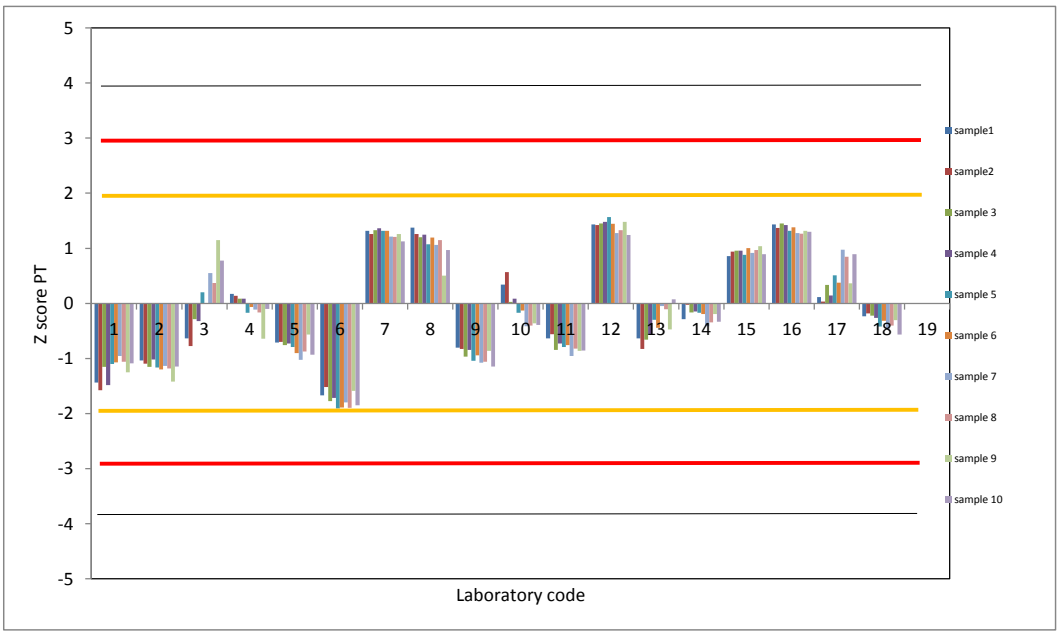


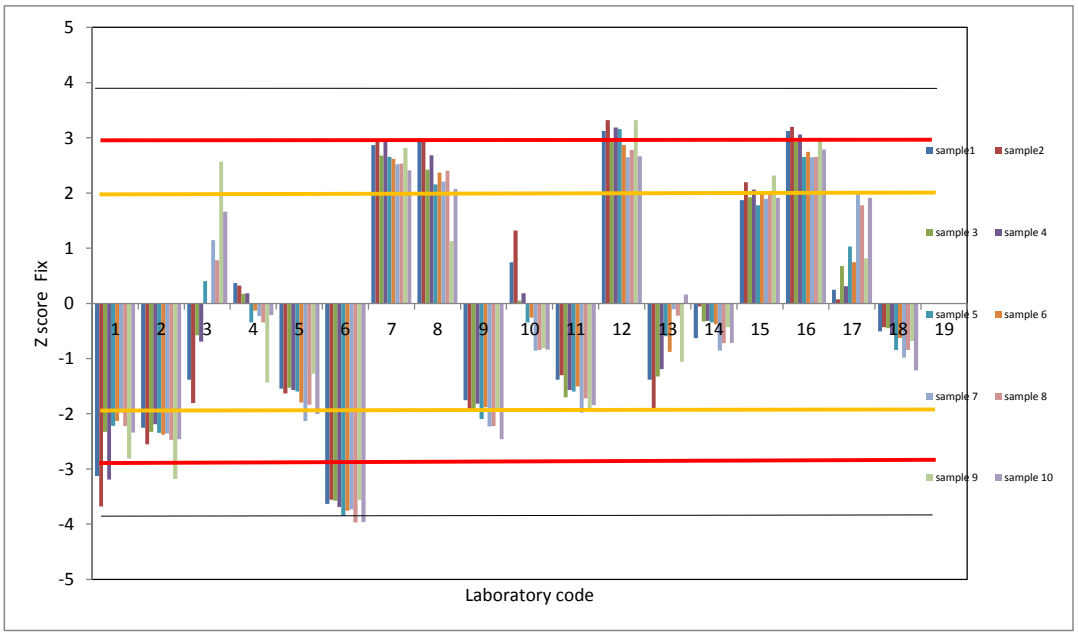
**Table VII :** **Zscore of the different laboratories for each sample.**
**ZS calculated on the standard deviation of reproducibility of the method**

| Sample / Lab code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | -3,13 | -3,68 | -2,33 | -3,19 | -2,22 | -2,13 | -1,98 | -2,22 | -2,81 | -2,34 |
| 2 | -2,26 | -2,55 | -2,33 | -2,19 | -2,35 | -2,38 | -2,36 | -2,47 | -3,18 | -2,46 |
| 3 | -1,38 | -1,80 | -0,58 | -0,69 | +0,40 | -0,01 | +1,14 | +0,78 | +2,57 | +1,66 |
| 4 | +0,37 | +0,32 | +0,17 | +0,18 | -0,35 | -0,13 | -0,23 | -0,35 | -1,43 | -0,21 |
| 5 | -1,54 | -1,63 | -1,53 | -1,57 | -1,60 | -1,79 | -2,13 | -1,83 | -1,27 | -2,00 |
| 6 | -3,63 | -3,55 | -3,58 | -3,69 | -3,85 | -3,76 | -3,73 | -3,97 | -3,56 | -3,96 |
| 7 | +2,87 | +2,95 | +2,67 | +2,93 | +2,65 | +2,62 | +2,52 | +2,53 | +2,82 | +2,41 |
| 8 | +2,99 | +2,95 | +2,42 | +2,68 | +2,15 | +2,37 | +2,21 | +2,40 | +1,13 | +2,07 |
| 9 | -1,76 | -1,93 | -1,95 | -1,82 | -2,10 | -1,88 | -2,23 | -2,22 | -1,93 | -2,46 |
| 10 | +0,74 | +1,32 | +0,05 | +0,18 | -0,35 | -0,26 | -0,86 | -0,85 | -0,81 | -0,84 |
| 11 | -1,38 | -1,30 | -1,70 | -1,57 | -1,60 | -1,51 | -1,98 | -1,72 | -1,93 | -1,84 |
| 12 | +3,12 | +3,32 | +2,92 | +3,18 | +3,15 | +2,87 | +2,64 | +2,78 | +3,32 | +2,66 |
| 13 | -1,38 | -1,93 | -1,33 | -1,19 | -0,60 | -0,88 | -0,11 | -0,22 | -1,06 | +0,16 |
| 14 | -0,63 | -0,05 | -0,33 | -0,32 | -0,35 | -0,38 | -0,86 | -0,72 | -0,43 | -0,71 |
| 15 | +1,87 | +2,20 | +1,92 | +2,06 | +1,78 | +1,99 | +1,89 | +2,03 | +2,32 | +1,91 |
| 16 | +3,12 | +3,20 | +2,92 | +3,06 | +2,65 | +2,74 | +2,64 | +2,65 | +2,94 | +2,79 |
| 17 | +0,24 | +0,07 | +0,67 | +0,31 | +1,03 | +0,74 | +2,02 | +1,78 | +0,82 | +1,91 |
| 18 | -0,51 | -0,43 | -0,45 | -0,57 | -0,85 | -0,63 | -0,98 | -0,85 | -0,68 | -1,21 |

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=0,040

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



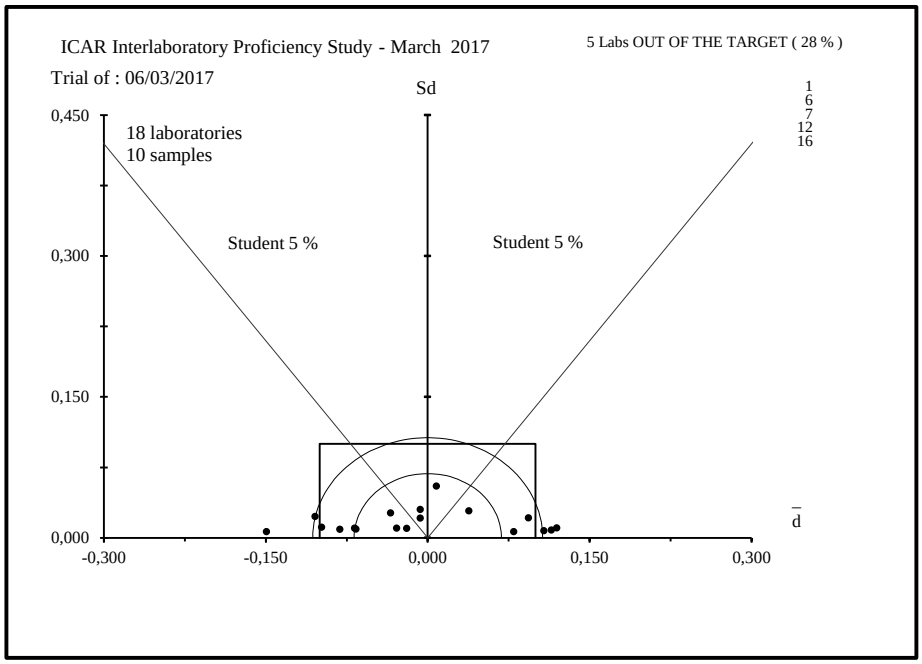



**Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).**

LIST OF PARTICIPANT LABORATORIES
RAW MILK
LACTOSE ROUTINE METHOD
MARCH 2017

| | | |
|---|---|---|
| AIA-LAB. STANDARD LATTE | MACCARESE (ROMA) | ITALY |
| CATTLE INFORMATION SERVICE (CIS) | TELFORD | UNITED KINGDOM |
| CENTRAL MILK LABORATORY ICBA | CAESAREA | ISRAEL |
| EASTERN LAB SERVICES | MEDINA | USA |
| EUROFINS STEINS LABORATORY A/B | VEJEN | DENMARK |
| FED.LATTERIE SOCIALI DI BOLZANO | BOLZANO | ITALY |
| LAB. POLJOPRIVREDNI | NOVI SAD | SERBIA |
| LOM KCHZ LABORATORIUM REFERENCYJNE z/s w | PARZNIEWIE | POLAND |
| MERIEUX NUTRISCIENCE | CAPE TOWN | SOUTH AFRICA |
| MERIEUX NUTRISCIENCE | MIDRAND | SOUTH AFRICA |
| MERIEUX NUTRISCIENCE J BAY | CAPE TOWN | SOUTH AFRICA |
| MILCHPRUFRING BADEN-WURTTEMBERG E.V. | KIRCHEIM / TECK | GERMANY |
| SHANGAI DAIRY BREEDING CENTER Co.Ltd | SHANGHAI | CHINA |
| TAIWAN LIVESTOCK RESEARCH INT. | TAIWAN | TAIWAN |
| VETLAB AGRICULTURAL SHOWGROUNDS | LUSAKA | ZAMBIA |

# ICAR
# PROFICIENCY TESTING SCHEME

---

## March 2017

## Raw Milk

## Determination of UREA CONTENT
### Routine method

Sending date of statistical treatment : 21th April 2017

| | | |
|---|---|---|
| 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 |

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

| Nb | % | N° | d | Sd | D | Method |
|---|---|---|---|---|---|---|
| 1 | 7 | 11 | + 0,94 | 0,96 | 1,35 | Other* |
| 2 | 13 | 1 | + 0,24 | 1,40 | 1,43 | IR |
| 3 | 20 | 14 | + 1,15 | 0,93 | 1,48 | IR |
| 4 | 27 | 6 | + 1,60 | 0,68 | 1,74 | IR |
| 5 | 33 | 2 | - 2,19 | 0,40 | 2,23 | IR |
| 6 | 40 | 12 | + 2,16 | 0,73 | 2,28 | IR |
| 7 | 47 | 10 | + 1,58 | 1,65 | 2,28 | IR |
| 8 | 53 | 15 | + 2,17 | 1,00 | 2,39 | IR |
| 9 | 60 | 13 | - 1,14 | 2,95 | 3,17 | IR |
| 10 | 67 | 9 | - 3,57 | 1,18 | 3,75 | IR |
| 11 | 73 | 3 | + 3,76 | 2,40 | 4,46 | IR |
| 12 | 80 | 5 | - 4,47 | 0,93 | 4,57 | IR |
| 13 | 87 | 7 | + 4,60 | 1,28 | 4,77 | IR |
| 14 | 93 | 8 | - 5,45 | 10,32 | 11,67 | IR |
| 15 | 100 | 4 | - 1,28 | 12,89 | 12,95 | IR |

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 14 sets of results send by 14 laboratories using routine method ISO 9622|IDF 141, after outlier discarging using Grubbs test at 5 % risk level

Other* : home method based on ASU L 01.00.78 (ISO 9622)

(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.($d^2$ + $Sd^2$))

$Sr_{PT}$ 0,99
$SR_{PT}$ 3,34

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 %)

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

| Sample/Lab Code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | Sr | NL |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0,86 | 0,43 | 0,43 | 0,86 | 0,21 | 0,64 | 0,86 | 0,86 | 0,43 | 0,64 | 0,47 | 20 |
| 2 | 1,00 | 2,00 | 1,00 | 1,00 | 1,00 | 1,00 | 1,00 | 0,00 | 0,00 | 1,00 | 0,74 | 20 |
| 3 | 7,00 * | 16,00 * | 2,00 | 2,00 | 1,00 | 1,00 | 4,00 | 3,00 | 3,00 | 1,00 | 4,18 | 20 |
| 4 | 1,50 | 0,40 | 2,60 | 0,50 | 0,80 | 1,10 | 2,00 | 0,20 | 0,10 | 0,40 | 0,88 | 20 |
| 5 | 1,10 | 0,70 | 0,90 | 1,30 | 1,00 | 1,30 | 0,90 | 1,00 | 3,00 | 2,20 | 1,06 | 20 |
| 6 | 0,60 | 0,20 | 0,00 | 0,90 | 0,10 | 0,90 | 1,70 | 1,20 | 0,40 | 0,00 | 0,57 | 20 |
| 7 | 0,00 | 0,43 | 0,64 | 2,14 | 0,64 | 0,43 | 0,64 | 2,14 | 0,64 | 0,86 | 0,77 | 20 |
| 8 | 1,00 | 1,10 | 0,40 | 1,60 | 0,50 | 1,20 | 0,20 | 1,10 | 0,20 | 2,90 | 0,90 | 20 |
| 9 | 3,06 | 2,02 | 8,82 * | 2,67 | 1,65 | 1,81 | 1,87 | 4,14 | 4,00 | 1,48 | 2,68 | 20 |
| 10 | 1,34 | 1,34 | 1,65 | 2,06 | 1,44 | 0,21 | 1,03 | 2,27 | 0,21 | 0,21 | 0,97 | 20 |
| 11 | 0,58 | 0,58 | 0,39 | 2,13 | 0,78 | 0,10 | 1,36 | 0,19 | 0,48 | 1,74 | 0,75 | 20 |
| 12 | 0,00 | 0,80 | 1,40 | 0,60 | 1,20 | 2,50 | 0,20 | 1,50 | 0,40 | 0,40 | 0,81 | 20 |
| 13 | 0,60 | 0,60 | 1,20 | 0,60 | 0,60 | 0,60 | 1,20 | 0,00 | 0,00 | 0,60 | 0,50 | 20 |
| 14 | 2,30 | 0,00 | 2,00 | 0,40 | 0,40 | 0,00 | 1,10 | 0,80 | 1,30 | 1,90 | 0,92 | 20 |
| 15 | 0,31 | 0,72 | 1,75 | 0,21 | 1,96 | 1,24 | 0,52 | 1,34 | 0,72 | 0,21 | 0,77 | 20 |
| Sr | 1,56 | 3,00 | 1,87 | 1,04 | 0,72 | 0,80 | 1,08 | 1,22 | 1,12 | 0,93 | | 300 |
| NE | 30 | 30 | 30 | 30 | 30 | 30 | 30 | 30 | 30 | 30 | | |
| L | 3,42 | 2,62 | 3,62 | 3,91 | 2,71 | 3,01 | 4,07 | 4,60 | 4,20 | 3,50 | | |

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 betwen two replicates=1,50 according ISO 14637|IDF 195

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

**(*) : Data modified in right units.**

| | Sample / Lab code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| (*) | 1 | 34,24 | 46,22 | 32,10 | 22,47 | 63,02 | 48,47 | 29,53 | 55,00 | 60,13 | 42,69 |
| | 2 | 33,50 | 43,00 | 29,50 | 20,50 | 61,50 | 48,50 | 25,50 | 52,00 | 57,00 | 38,50 |
| | 3 | 37,50 | 43,00 | 37,00 | 28,00 | 69,50 | 55,50 | 32,00 | 58,50 | 63,50 | 44,50 |
| | 4 | 30,45 | 39,70 | 26,60 | 58,05 * | 58,10 | 44,35 | 21,90 | 49,20 | 53,95 | 36,30 |
| | 5 | 33,25 | 41,75 | 26,25 | 17,65 | 59,60 | 45,35 | 21,85 | 50,80 | 55,20 | 35,00 |
| | 6 | 37,90 | 47,50 | 32,60 | 23,05 | 65,75 | 51,75 | 28,25 | 56,20 | 61,20 | 43,20 |
| (*) | 7 | 40,87 | 49,65 | 35,42 | 25,25 | 68,80 | 54,14 | 30,28 | 61,20 | 65,16 | 46,65 |
| | 8 | 32,60 | 41,75 | 29,60 | 20,00 | 29,25 * | 48,30 | 25,50 | 52,55 | 58,00 | 39,35 |
| | 9 | 34,21 | 41,16 | 29,31 | 17,82 | 60,28 | 47,00 | 22,88 | 51,51 | 56,70 | 34,94 |
| | 10 | 38,99 | 47,53 | 31,72 | 22,87 | 67,57 | 51,91 | 25,75 | 57,37 | 62,21 | 41,30 |
| | 11 | 37,69 | 47,19 | 33,72 | 24,32 | 63,76 | 50,05 | 29,07 | 54,55 | 59,35 | 41,18 |
| | 12 | 37,90 | 47,90 | 33,40 | 23,40 | 66,00 | 51,45 | 30,30 | 57,15 | 62,00 | 43,50 |
| | 13 | 36,90 | 44,10 | 33,00 | 25,50 | 58,50 | 47,70 | 29,40 | 50,40 | 54,60 | 39,90 |
| | 14 | 38,65 | 46,90 | 31,80 | 22,30 | 66,20 | 50,30 | 29,05 | 55,90 | 59,95 | 41,85 |
| | 15 | 38,88 | 47,74 | 33,11 | 24,21 | 67,31 | 53,46 | 28,48 | 57,11 | 61,75 | 41,10 |
| | M | 36,24 | 45,01 | 31,68 | 22,67 | 63,99 | 49,88 | 27,32 | 54,63 | 59,38 | 40,66 |
| | REF. | 36,24 | 45,01 | 31,68 | 22,67 | 63,99 | 49,88 | 27,32 | 54,63 | 59,38 | 40,66 |
| | SD | 2,96 | 3,08 | 2,98 | 2,90 | 3,87 | 3,20 | 3,22 | 3,44 | 3,38 | 3,40 |

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 14 laboratories using the Routine method , after discard outliers with Grubbs test at 5 %.

**Table IV :** Outlier identification

| **Sample** | **1** | **2** | **3** | **4** | **5** | **6** | **7** | **8** | **9** | **10** |
|---|---|---|---|---|---|---|---|---|---|---|
| **Outliers Cochran** | 3 | 3 | 9 | | | | | | | |
| **Outlier Grubbs** | | | | 4 | 8 | | | | | |
| **sr** | 0,92 | 0,71 | 0,98 | 1,07 | 0,74 | 0,80 | 1,08 | 1,22 | 1,12 | 0,93 |
| **SR** | 3,12 | 3,18 | 3,09 | 3,00 | 3,90 | 3,25 | 3,31 | 3,55 | 3,47 | 3,47 |

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

| Sample \ Lab Code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | d | $Sd_{lab}$ | t |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | - 2,00 | + 1,22 | + 0,42 | - 0,20 | - 0,97 | - 1,41 | + 2,22 | + 0,37 | + 0,75 | + 2,03 | + 0,24 | 1,40 | 0,55 |
| 2 | - 2,74 | - 2,01 | - 2,18 | - 2,17 | - 2,49 | - 1,38 | - 1,82 | - 2,63 | - 2,38 | - 2,16 | - 2,19 | 0,40 | 17,31 |
| 3 | + 1,26 | - 2,01 | + 5,32 | + 5,33 | + 5,51 | + 5,62 | + 4,68 | + 3,87 | + 4,12 | + 3,84 | + 3,76 | 2,40 | 4,94 |
| 4 | - 5,79 | - 5,31 | - 5,08 | + 35,38 | - 5,89 | - 5,53 | - 5,42 | - 5,43 | - 5,43 | - 4,36 | - 1,28 | 12,89 | 0,32 |
| 5 | - 2,99 | - 3,26 | - 5,43 | - 5,02 | - 4,39 | - 4,53 | - 5,47 | - 3,83 | - 4,18 | - 5,66 | - 4,47 | 0,93 | 15,19 |
| 6 | + 1,66 | + 2,49 | + 0,92 | + 0,38 | + 1,76 | + 1,87 | + 0,93 | + 1,57 | + 1,82 | + 2,54 | + 1,60 | 0,68 | 7,38 |
| 7 | + 4,64 | + 4,64 | + 3,74 | + 2,59 | + 4,81 | + 4,26 | + 2,97 | + 6,57 | + 5,78 | + 5,99 | + 4,60 | 1,28 | 11,32 |
| 8 | - 3,64 | - 3,26 | - 2,08 | - 2,67 | - 34,74 | - 1,58 | - 1,82 | - 2,08 | - 1,38 | - 1,31 | - 5,45 | 10,32 | 1,67 |
| 9 | - 2,03 | - 3,85 | - 2,37 | - 4,85 | - 3,72 | - 2,89 | - 4,44 | - 3,12 | - 2,68 | - 5,72 | - 3,57 | 1,18 | 9,59 |
| 10 | + 2,75 | + 2,53 | + 0,05 | + 0,20 | + 3,58 | + 2,03 | - 1,57 | + 2,74 | + 2,83 | + 0,64 | + 1,58 | 1,65 | 3,03 |
| 11 | + 1,46 | + 2,18 | + 2,05 | + 1,66 | - 0,23 | + 0,17 | + 1,75 | - 0,08 | - 0,03 | + 0,52 | + 0,94 | 0,96 | 3,10 |
| 12 | + 1,66 | + 2,89 | + 1,72 | + 0,73 | + 2,01 | + 1,57 | + 2,98 | + 2,52 | + 2,62 | + 2,84 | + 2,16 | 0,73 | 9,28 |
| 13 | + 0,66 | - 0,91 | + 1,32 | + 2,83 | - 5,49 | - 2,18 | + 2,08 | - 4,23 | - 4,78 | - 0,76 | - 1,14 | 2,95 | 1,23 |
| 14 | + 2,41 | + 1,89 | + 0,12 | - 0,37 | + 2,21 | + 0,42 | + 1,73 | + 1,27 | + 0,57 | + 1,19 | + 1,15 | 0,93 | 3,88 |
| 15 | + 2,65 | + 2,73 | + 1,44 | + 1,54 | + 3,32 | + 3,58 | + 1,16 | + 2,48 | + 2,37 | + 0,43 | + 2,17 | 1,00 | 6,88 |
| d | - 0,00 | - 0,00 | + 0,00 | + 0,00 | - 0,00 | - 0,00 | + 0,00 | + 0,00 | - 0,00 | + 0,00 | + 0,00 | 5,13 | |
| Sd | 2,96 | 3,08 | 2,98 | 2,90 | 3,87 | 3,20 | 3,22 | 3,44 | 3,38 | 3,40 | 3,26 | | |

d = mean of differences  Sd = standard deviation of differences  t = Student test - comparison to 0

Upper limits : $\bar{d}$ = +/- 2,50 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 code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | -0,67 | +0,40 | +0,14 | -0,07 | -0,25 | -0,44 | +0,69 | +0,11 | +0,22 | +0,60 |
| 2 | -0,92 | -0,65 | -0,73 | -0,75 | -0,64 | -0,43 | -0,56 | -0,77 | -0,70 | -0,64 |
| 3 | +0,43 | -0,65 | +1,79 | +1,84 | +1,42 | +1,76 | +1,45 | +1,13 | +1,22 | +1,13 |
| 4 | -1,95 | -1,72 | -1,70 | +12,18 | -1,52 | -1,73 | -1,68 | -1,58 | -1,61 | -1,28 |
| 5 | -1,01 | -1,06 | -1,82 | -1,73 | -1,14 | -1,42 | -1,70 | -1,11 | -1,24 | -1,66 |
| 6 | +0,56 | +0,81 | +0,31 | +0,13 | +0,45 | +0,58 | +0,29 | +0,46 | +0,54 | +0,74 |
| 7 | +1,57 | +1,51 | +1,26 | +0,89 | +1,24 | +1,33 | +0,92 | +1,91 | +1,71 | +1,76 |
| 8 | -1,23 | -1,06 | -0,70 | -0,92 | -8,98 | -0,49 | -0,56 | -0,61 | -0,41 | -0,39 |
| 9 | -0,68 | -1,25 | -0,79 | -1,67 | -0,96 | -0,90 | -1,38 | -0,91 | -0,79 | -1,68 |
| 10 | +0,93 | +0,82 | +0,02 | +0,07 | +0,92 | +0,63 | -0,49 | +0,80 | +0,84 | +0,19 |
| 11 | +0,49 | +0,71 | +0,69 | +0,57 | -0,06 | +0,05 | +0,54 | -0,02 | -0,01 | +0,15 |
| 12 | +0,56 | +0,94 | +0,58 | +0,25 | +0,52 | +0,49 | +0,93 | +0,73 | +0,78 | +0,83 |
| 13 | +0,22 | -0,29 | +0,44 | +0,98 | -1,42 | -0,68 | +0,65 | -1,23 | -1,42 | -0,22 |
| 14 | +0,82 | +0,61 | +0,04 | -0,13 | +0,57 | +0,13 | +0,54 | +0,37 | +0,17 | +0,35 |
| 15 | +0,89 | +0,89 | +0,48 | +0,53 | +0,86 | +1,12 | +0,36 | +0,72 | +0,70 | +0,13 |

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

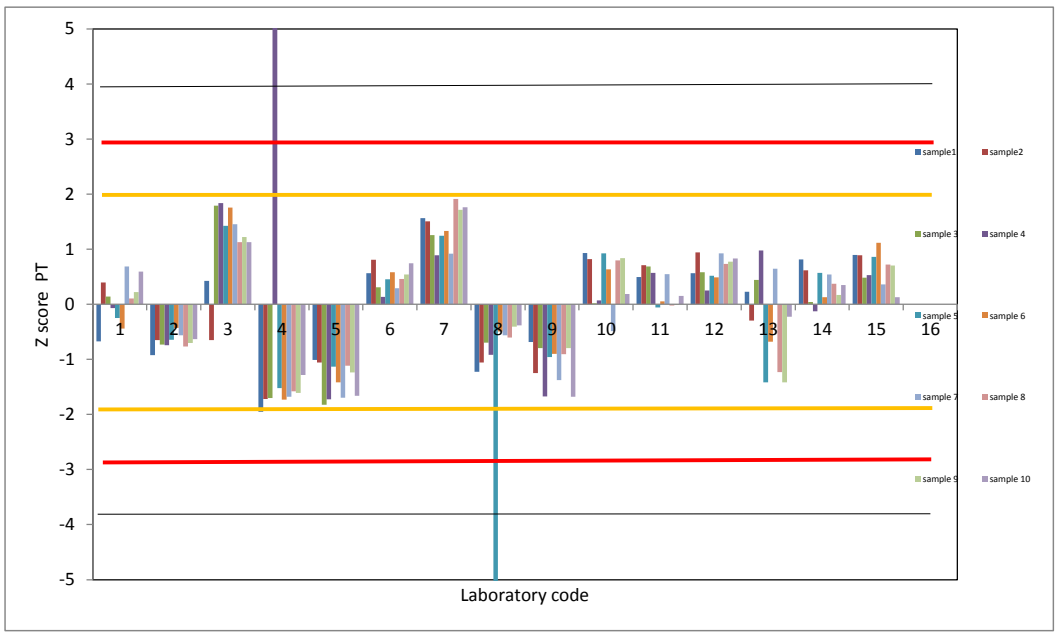


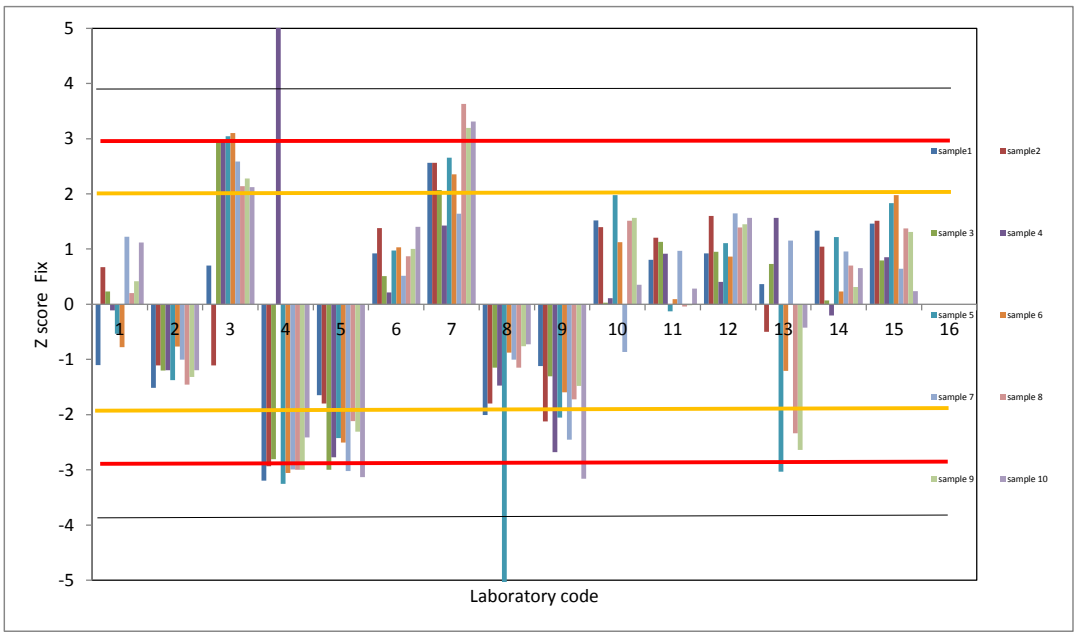
**Table VII :** **Zscore of the different laboratories for each sample.**
**ZS calculated on the standard deviation of reproducibility of the method**

| Sample / Lab code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | -1,10 | +0,67 | +0,23 | -0,11 | -0,54 | -0,78 | +1,22 | +0,20 | +0,42 | +1,12 |
| 2 | -1,51 | -1,11 | -1,20 | -1,20 | -1,38 | -0,76 | -1,00 | -1,45 | -1,32 | -1,20 |
| 3 | +0,70 | -1,11 | +2,94 | +2,95 | +3,04 | +3,10 | +2,59 | +2,14 | +2,28 | +2,12 |
| 4 | -3,20 | -2,93 | -2,80 | +19,55 | -3,26 | -3,06 | -2,99 | -3,00 | -3,00 | -2,41 |
| 5 | -1,65 | -1,80 | -3,00 | -2,77 | -2,43 | -2,50 | -3,02 | -2,12 | -2,31 | -3,13 |
| 6 | +0,92 | +1,38 | +0,51 | +0,21 | +0,97 | +1,03 | +0,52 | +0,87 | +1,01 | +1,40 |
| 7 | +2,56 | +2,56 | +2,07 | +1,43 | +2,66 | +2,35 | +1,64 | +3,63 | +3,19 | +3,31 |
| 8 | -2,01 | -1,80 | -1,15 | -1,47 | -19,19 | -0,87 | -1,00 | -1,15 | -0,76 | -0,73 |
| 9 | -1,12 | -2,13 | -1,31 | -2,68 | -2,05 | -1,59 | -2,45 | -1,72 | -1,48 | -3,16 |
| 10 | +1,52 | +1,40 | +0,03 | +0,11 | +1,98 | +1,12 | -0,87 | +1,51 | +1,56 | +0,35 |
| 11 | +0,81 | +1,21 | +1,13 | +0,91 | -0,13 | +0,09 | +0,97 | -0,04 | -0,02 | +0,29 |
| 12 | +0,92 | +1,60 | +0,95 | +0,41 | +1,11 | +0,87 | +1,65 | +1,39 | +1,45 | +1,57 |
| 13 | +0,37 | -0,50 | +0,73 | +1,57 | -3,03 | -1,21 | +1,15 | -2,34 | -2,64 | -0,42 |
| 14 | +1,33 | +1,05 | +0,07 | -0,20 | +1,22 | +0,23 | +0,96 | +0,70 | +0,31 | +0,65 |
| 15 | +1,46 | +1,51 | +0,79 | +0,85 | +1,83 | +1,98 | +0,64 | +1,37 | +1,31 | +0,24 |

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



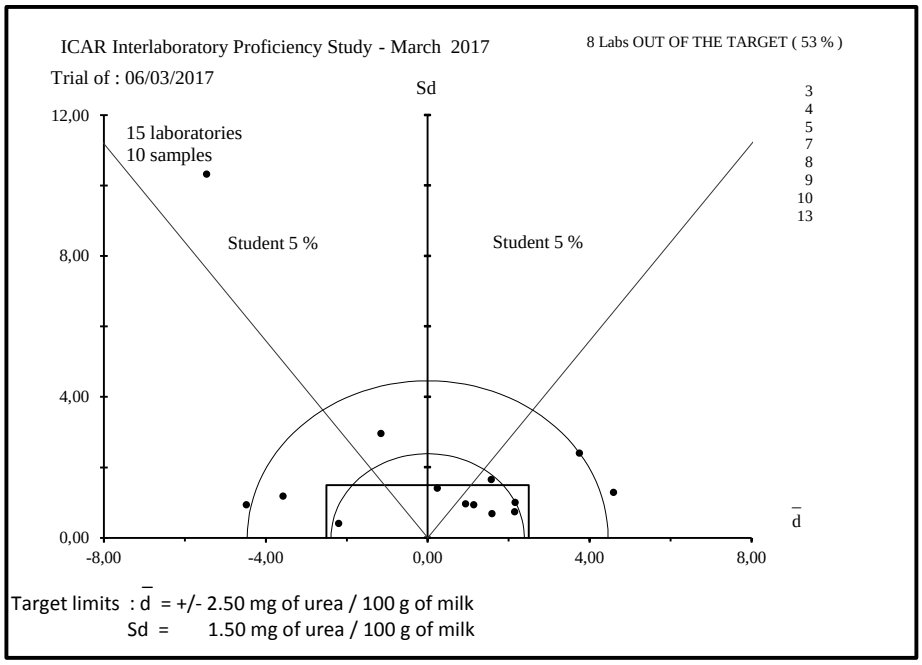



**Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).**

LIST OF ICAR PARTICIPANTS

RAW MILK
UREA ROUTINE METHOD
MARCH 2017

| | | |
|---|---|---|
| CATTLE INFORMATION SERVICE (CIS) | TELFORD | UNITED KINGDOM |
| COOPRINSEM | OSORNO | CHILE |
| CROATIAN AGRICULTURAL AG., CENTRAL LAB. for MILK QUALITY CONTROL | KRIZEVCI | CROATIA |
| EASTERN LAB SERVICES | MEDINA | USA |
| EUROFINS STEINS LABORATORY A/B | JÖNGÖPING | SWEDEN |
| FED.LATTERIE SOCIALI DI BOLZANO | BOLZANO | ITALY |
| LAB. POLJOPRIVREDNI | NOVI SAD | SERBIA |
| LOM KCHZ LABORATORIUM REFERENCYJNE z/s w | PARZNIEWIE | POLAND |
| MERIEUX NUTRISCIENCE | CAPE TOWN | SOUTH AFRICA |
| MERIEUX NUTRISCIENCE | MIDRAND | SOUTH AFRICA |
| MERIEUX NUTRISCIENCE J BAY | CAPE TOWN | SOUTH AFRICA |
| MILCHPRUFRING BADEN-WURTTEMBERG E.V. | KIRCHEIM / TECK | GERMANY |
| SHANGAI DAIRY BREEDING CENTER Co.Ltd | SHANGHAI | CHINA |
| TAIWAN LIVESTOCK RESEARCH INT. | TAIWAN | TAIWAN |

# ICAR
# PROFICIENCY TESTING SCHEME

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## March 2017

## Raw Milk

## BHB Beta-HydroxyButyrate

Sending date of statistical treatment : 21th April 2017


| | | |
|---|---|---|
| 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 |


ACTALIA CECALAIT

**Table I :** **Ranking of the laboratories** Units : milimole of BHB / liter of milk

| Nb | % | N° | d | Sd | D | Method |
|---|---|---|---|---|---|---|
| 1 | 6 | 8 | - 0,012 | 0,020 | 0,023 | A |
| 2 | 13 | 15 | + 0,013 | 0,020 | 0,024 | A |
| 3 | 19 | 13 | - 0,021 | 0,015 | 0,026 | A |
| 4 | 25 | 7 | - 0,022 | 0,018 | 0,029 | A |
| 5 | 31 | 5 | + 0,033 | 0,020 | 0,038 | A |
| 6 | 38 | 3 | - 0,036 | 0,013 | 0,038 | A |
| 7 | 44 | 1 | + 0,028 | 0,026 | 0,039 | B |
| 8 | 50 | 14 | - 0,045 | 0,013 | 0,047 | A |
| 9 | 56 | 12 | - 0,010 | 0,047 | 0,048 | A |
| 10 | 63 | 9 | + 0,056 | 0,032 | 0,065 | A |
| 11 | 69 | 10 | + 0,065 | 0,042 | 0,077 | A |
| 12 | 75 | 6 | - 0,081 | 0,015 | 0,082 | A |
| 13 | 81 | 2 | - 0,046 | 0,097 | 0,107 | A |
| 14 | 88 | 11 | + 0,006 | 0,114 | 0,115 | A |
| 15 | 94 | 16 | + 0,111 | 0,062 | 0,127 | A |
| 16 | 100 | 4 | + 0,074 | 0,134 | 0,153 | B |

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 :

$\bar{d}$ = +/- 0,045 milimole of BHB / liter of milk
Sd = 0,045 milimole of BHB / liter of milk

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 16 set of results send by 16 laboratories discarging using Grubbs test at 5 % risk level

A I.R.
B Continuous flow analyzer

(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.($d^2$ + $Sd^2$))

Repeatability standard deviation of this ICAR proficiency test (after Cochran elimination at 5 %) $Sr_{PT}$ 0,011
Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) $SR_{PT}$ 0,058

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.

**Table II :** **REPEATABILITY - Absolute difference between replicates in milimole of BHB / liter of milk**

| Sample / Lab code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | Sr | NL |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0,000 | 0,000 | 0,000 | 0,010 | 0,000 | 0,000 | 0,010 | 0,010 | 0,000 | 0,000 | 0,004 | 20 |
| 2 | 0,010 | 0,010 | 0,020 | 0,010 | 0,010 | 0,010 | 0,030 | 0,010 | 0,010 | 0,000 | 0,010 | 20 |
| 3 | 0,009 | 0,008 | 0,013 | 0,012 | 0,007 | 0,009 | 0,044 | 0,011 | 0,014 | 0,001 | 0,012 | 20 |
| 4 | 0,006 | 0,000 | 0,002 | 0,001 | 0,000 | 0,002 | 0,001 | 0,002 | 0,001 | 0,002 | 0,002 | 20 |
| 5 | 0,000 | 0,000 | 0,010 | 0,000 | 0,010 | 0,000 | 0,000 | 0,000 | 0,000 | 0,000 | 0,003 | 20 |
| 6 | ** | 0,010 | 0,010 | 0,020 | 0,000 | 0,010 | 0,010 | ** | 0,000 | 0,010 | 0,008 | 16 |
| 7 | 0,000 | 0,000 | 0,000 | 0,010 | 0,000 | 0,010 | 0,050 | 0,010 | 0,010 | 0,010 | 0,012 | 20 |
| 8 | 0,000 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,010 | 0,000 | 0,010 | 0,010 | 0,006 | 20 |
| 9 | 0,010 | 0,020 | 0,030 | 0,010 | 0,050 * | 0,020 | 0,060 | 0,010 | 0,010 | 0,010 | 0,020 | 20 |
| 10 | 0,010 | 0,070 * | 0,050 | 0,020 | 0,080 * | 0,000 | 0,040 | 0,030 | 0,000 | 0,020 | 0,029 | 20 |
| 11 | 0,010 | 0,000 | 0,020 | 0,030 | 0,000 | 0,000 | 0,010 | 0,020 | 0,020 | 0,000 | 0,011 | 20 |
| 12 | ** | ** | ** | ** | ** | ** | ** | ** | ** | ** | | |
| 13 | 0,010 | 0,030 | 0,030 | 0,020 | 0,020 | 0,010 | 0,030 | 0,030 | 0,010 | 0,010 | 0,015 | 20 |
| 14 | 0,000 | 0,020 | 0,010 | 0,010 | 0,000 | 0,010 | 0,020 | 0,010 | 0,010 | 0,020 | 0,009 | 20 |
| 15 | 0,000 | 0,010 | 0,000 | 0,010 | 0,010 | 0,010 | 0,020 | 0,000 | 0,010 | 0,010 | 0,007 | 20 |
| 16 | ** | ** | ** | ** | ** | ** | ** | ** | ** | ** | | |
| Sr | 0,005 | 0,016 | 0,014 | 0,010 | 0,019 | 0,007 | 0,021 | 0,010 | 0,007 | 0,007 | | 276 |
| NE | 26 | 28 | 28 | 28 | 28 | 28 | 28 | 26 | 28 | 28 | | |
| L | 0,018 | 0,033 | 0,053 | 0,038 | 0,021 | 0,024 | 0,079 | 0,038 | 0,025 | 0,026 | | |

Sr : repeatability standard deviation of each laboratory limit 0,011 milimole of BHB / liter of milk
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 betwen two replicates=0,030 milimole of BHB / liter of milk

**Table III :** **Means of the replicates in milimole of BHB / liter of milk**

| Sample / Lab code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0,050 | 0,230 | 0,100 | 0,125 | 0,280 | 0,180 | 0,205 | 0,075 | 0,250 | 0,150 |
| 2 | 0,145 | 0,045 | 0,050 | 0,185 | 0,085 | 0,115 | 0,025 | 0,175 | 0,065 | 0,010 |
| 3 | 0,026 | 0,150 | 0,040 | 0,075 | 0,175 | 0,111 | 0,137 | 0,031 | 0,184 | 0,075 |
| 4 | 0,305 * | 0,124 | 0,163 | 0,375 * | 0,224 | 0,260 | 0,083 | 0,330 * | 0,184 | 0,055 |
| 5 | 0,080 | 0,230 | 0,115 | 0,130 | 0,285 | 0,160 | 0,200 | 0,090 | 0,250 | 0,150 |
| 6 | | 0,085 | 0,015 | 0,020 | 0,140 | 0,055 | 0,075 | 0,010 | 0,120 | 0,045 |
| 7 | 0,020 | 0,160 | 0,070 | 0,065 | 0,220 | 0,125 | 0,145 | 0,035 | 0,205 | 0,095 |
| 8 | 0,030 | 0,195 | 0,095 | 0,085 | 0,215 | 0,115 | 0,145 | 0,050 | 0,205 | 0,105 |
| 9 | 0,085 | 0,250 | 0,135 | 0,135 | 0,335 | 0,180 | 0,230 | 0,105 | 0,295 | 0,175 |
| 10 | 0,065 | 0,305 | 0,135 | 0,160 | 0,360 | 0,210 | 0,230 | 0,115 | 0,280 | 0,150 |
| 11 | 0,215 | 0,060 | 0,090 | 0,265 | 0,140 | 0,190 | 0,055 | 0,250 * | 0,130 | 0,030 |
| 12 | 0,140 | 0,150 | 0,070 | 0,100 | 0,170 | | | | | |
| 13 | 0,025 | 0,175 | 0,065 | 0,090 | 0,190 | 0,125 | 0,145 | 0,045 | 0,205 | 0,085 |
| 14 | 0,020 | 0,140 | 0,035 | 0,065 | 0,190 | 0,085 | 0,120 | 0,025 | 0,155 | 0,080 |
| 15 | 0,050 | 0,205 | 0,100 | 0,105 | 0,255 | 0,145 | 0,180 | 0,080 | 0,225 | 0,145 |
| 16 | 0,100 | 0,340 | 0,160 | 0,190 | 0,450 | 0,240 | 0,310 | 0,110 | 0,360 | 0,210 |
| M | 0,075 | 0,178 | 0,090 | 0,120 | 0,232 | 0,153 | 0,152 | 0,073 | 0,208 | 0,104 |
| REF. | 0,071 | 0,176 | 0,090 | 0,116 | 0,227 | 0,152 | 0,150 | 0,070 | 0,207 | 0,103 |
| SD | 0,058 | 0,082 | 0,044 | 0,061 | 0,093 | 0,057 | 0,076 | 0,046 | 0,075 | 0,058 |

M = mean per sample
SD = standard deviation per sample
REF. = reference values
*: 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 16 laboratories , after outliers discarging using Grubbs test at 5 % risk level.

**Table IV :** Outlier identification

| **Sample** | **1** | **2** | **3** | **4** | **5** | **6** | **7** | **8** | **9** | **10** |
|---|---|---|---|---|---|---|---|---|---|---|
| **Outliers Cochran** | | 10 | | | 9; 10 | | | | | |
| **Outlier Grubbs** | 4 | | | 4 | | | | 4; 11 | | |
| **sr** | 0,005 | 0,009 | 0,014 | 0,011 | 0,006 | 0,007 | 0,021 | 0,010 | 0,007 | 0,007 |
| **SR** | 0,059 | 0,066 | 0,044 | 0,063 | 0,060 | 0,054 | 0,066 | 0,046 | 0,064 | 0,052 |

**Table V :** **ACCURACY - differences (laboratory - reference) in milimole of BHB / liter of milk**

| Sample / Lab code | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | d | $Sd_{lab}$ | t |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | - 0,021 | + 0,054 | + 0,010 | + 0,009 | + 0,053 | + 0,028 | + 0,055 | + 0,005 | + 0,043 | + 0,047 | + 0,028 | 0,026 | 3,42 |
| 2 | + 0,074 | - 0,131 | - 0,040 | + 0,069 | - 0,142 | - 0,037 | - 0,125 | + 0,105 | - 0,142 | - 0,093 | - 0,046 | 0,097 | 1,50 |
| 3 | - 0,045 | - 0,026 | - 0,050 | - 0,041 | - 0,053 | - 0,042 | - 0,013 | - 0,039 | - 0,023 | - 0,029 | - 0,036 | 0,013 | 8,84 |
| 4 | + 0,234 | - 0,052 | + 0,073 | + 0,258 | - 0,003 | + 0,108 | - 0,067 | + 0,260 | - 0,023 | - 0,048 | + 0,074 | 0,134 | 1,75 |
| 5 | + 0,009 | + 0,054 | + 0,025 | + 0,014 | + 0,058 | + 0,008 | + 0,050 | + 0,020 | + 0,043 | + 0,047 | + 0,033 | 0,020 | 5,30 |
| 6 | | - 0,091 | - 0,075 | - 0,096 | - 0,087 | - 0,097 | - 0,075 | - 0,060 | - 0,087 | - 0,058 | - 0,081 | 0,015 | 16,61 |
| 7 | - 0,051 | - 0,016 | - 0,020 | - 0,051 | - 0,007 | - 0,027 | - 0,005 | - 0,035 | - 0,002 | - 0,008 | - 0,022 | 0,018 | 3,83 |
| 8 | - 0,041 | + 0,019 | + 0,005 | - 0,031 | - 0,012 | - 0,037 | - 0,005 | - 0,020 | - 0,002 | + 0,002 | - 0,012 | 0,020 | 1,95 |
| 9 | + 0,014 | + 0,074 | + 0,045 | + 0,019 | + 0,108 | + 0,028 | + 0,080 | + 0,035 | + 0,088 | + 0,072 | + 0,056 | 0,032 | 5,52 |
| 10 | - 0,006 | + 0,129 | + 0,045 | + 0,044 | + 0,133 | + 0,058 | + 0,080 | + 0,045 | + 0,073 | + 0,047 | + 0,065 | 0,042 | 4,92 |
| 11 | + 0,144 | - 0,116 | + 0,000 | + 0,149 | - 0,087 | + 0,038 | - 0,095 | + 0,180 | - 0,077 | - 0,073 | + 0,006 | 0,114 | 0,18 |
| 12 | + 0,069 | - 0,026 | - 0,020 | - 0,016 | - 0,057 | | | | | | - 0,010 | 0,047 | 0,47 |
| 13 | - 0,046 | - 0,001 | - 0,025 | - 0,026 | - 0,037 | - 0,027 | - 0,005 | - 0,025 | - 0,002 | - 0,018 | - 0,021 | 0,015 | 4,49 |
| 14 | - 0,051 | - 0,036 | - 0,055 | - 0,051 | - 0,037 | - 0,067 | - 0,030 | - 0,045 | - 0,052 | - 0,023 | - 0,045 | 0,013 | 10,73 |
| 15 | - 0,021 | + 0,029 | + 0,010 | - 0,011 | + 0,028 | - 0,007 | + 0,030 | + 0,010 | + 0,018 | + 0,042 | + 0,013 | 0,020 | 1,98 |
| 16 | + 0,029 | + 0,164 | + 0,070 | + 0,074 | + 0,223 | + 0,088 | + 0,160 | + 0,040 | + 0,153 | + 0,107 | + 0,111 | 0,062 | 5,63 |
| d | + 0,004 | + 0,002 | - 0,000 | + 0,004 | + 0,005 | + 0,001 | + 0,002 | + 0,003 | + 0,001 | + 0,001 | + 0,008 | 0,074 | |
| Sd | 0,058 | 0,082 | 0,044 | 0,061 | 0,093 | 0,057 | 0,076 | 0,046 | 0,075 | 0,058 | 0,067 | | |

d = mean of differences  Sd = standard deviation of differences  t = Student test - comparison to 0

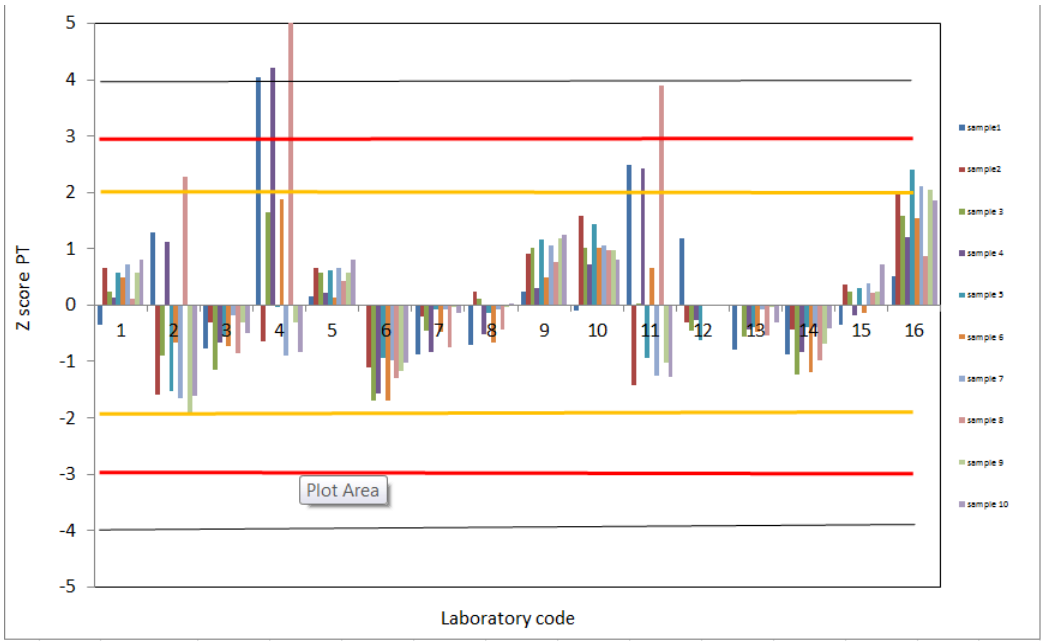
Upper limits : $\bar{d}$ = +/- 0,045 milimole of BHB / liter of milk
Sd = 0,045 milimole of BHB / liter of milk

**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 | -0,35 | +0,66 | +0,23 | +0,14 | +0,57 | +0,48 | +0,72 | +0,11 | +0,58 | +0,81 |
| 2 | +1,28 | -1,60 | -0,90 | +1,12 | -1,53 | -0,65 | -1,65 | +2,27 | -1,90 | -1,62 |
| 3 | -0,78 | -0,31 | -1,14 | -0,67 | -0,56 | -0,73 | -0,17 | -0,85 | -0,30 | -0,50 |
| 4 | +4,04 | -0,63 | +1,65 | +4,20 | -0,03 | +1,88 | -0,89 | +5,62 | -0,31 | -0,84 |
| 5 | +0,16 | +0,66 | +0,57 | +0,23 | +0,62 | +0,13 | +0,66 | +0,43 | +0,58 | +0,81 |
| 6 | | -1,11 | -1,69 | -1,56 | -0,94 | -1,70 | -0,99 | -1,29 | -1,16 | -1,01 |
| 7 | -0,87 | -0,19 | -0,45 | -0,83 | -0,08 | -0,48 | -0,07 | -0,75 | -0,02 | -0,14 |
| 8 | -0,70 | +0,23 | +0,12 | -0,51 | -0,13 | -0,65 | -0,07 | -0,43 | -0,02 | +0,03 |
| 9 | +0,25 | +0,91 | +1,02 | +0,31 | +1,16 | +0,48 | +1,05 | +0,76 | +1,18 | +1,25 |
| 10 | -0,10 | +1,58 | +1,02 | +0,71 | +1,43 | +1,01 | +1,05 | +0,97 | +0,98 | +0,81 |
| 11 | +2,49 | -1,41 | +0,00 | +2,42 | -0,94 | +0,66 | -1,25 | +3,89 | -1,03 | -1,27 |
| 12 | +1,20 | -0,31 | -0,45 | -0,26 | -0,61 | | | | | |
| 13 | -0,78 | -0,01 | -0,56 | -0,43 | -0,40 | -0,48 | -0,07 | -0,54 | -0,02 | -0,32 |
| 14 | -0,87 | -0,44 | -1,24 | -0,83 | -0,40 | -1,18 | -0,39 | -0,97 | -0,69 | -0,40 |
| 15 | -0,35 | +0,36 | +0,23 | -0,18 | +0,30 | -0,13 | +0,40 | +0,22 | +0,24 | +0,73 |
| 16 | +0,51 | +2,01 | +1,59 | +1,20 | +2,40 | +1,53 | +2,11 | +0,87 | +2,05 | +1,86 |

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



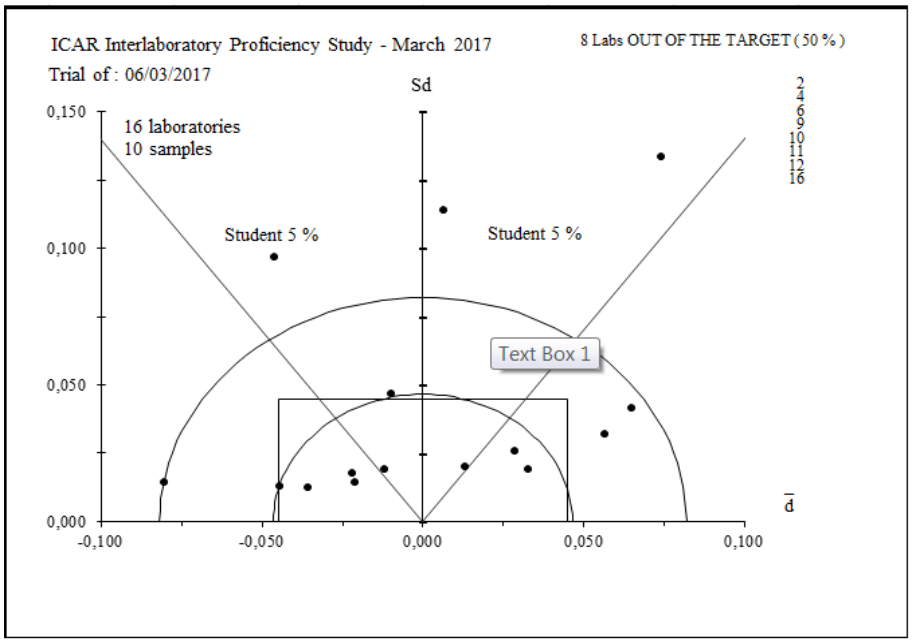



**Figure 1 :** **ACCURACY - Evaluation of the individual performances (to see table I).**

LIST OF ICAR PARTICIPANTS

RAW MILK
BHB
MARCH 2017

| Name | City | Country |
|---|---|---|
| ACTALIA | POLIGNY | FRANCE |
| CENTRAL MILK TESTING LAB | ALBERTA | CANADA |
| ESTONIAN LIVESTOCK PERFORMANCE RECORDING Ltd | TARTU | ESTONIA |
| EUROFINS STEINS LABORATORY A/S | VEJEN | DENMARK |
| EUROFINS STEINS LABORATORY A/S | JÖNKÖPING | SWEDEN |
| HORIZON LAB | WINNIPEG | CANADA |
| LAB. POLJOPRIVREDNI | NOVI SAD | SERBIA |
| LABORATORIO REGIONAL DE VETERINARIA | AZORES | PORTUGAL |
| LIGAL | LA CORUNA | SPAIN |
| PACIFIC MILK ANALYSIS | CHILLIWACK | CANADA |
| QLIP N.V. | CM ZUTPHEN | NETHERLAND |
| TAIWAN LIVESTOCK RESEARCH INST. | HSINHUA | TAIWAN |
| THE CATTLE INFORMATION SERVICE | TELFORD | UNITED KINGDOM |
| VALACTA | STE ANNE DE BELLEVUE | CANADA |

# ICAR

# PROFICIENCY TESTING SCHEME

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## MARCH 2017

## Cow Raw Milk

## DETECTION of PAG (Pregnancy Associated Glycoproteins)

Sending date of statistical treatment : 21th April 2017



| | | |
|---|---|---|
| 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 |



ACTALIA CECALAIT ®

**Table I:** **Methods**

| N° | METHOD USED |
|---|---|
| **1** | IDEXX |
| **2** | IDEXX |
| **3** | IDEXX |
| **4** | IDEXX |
| **5** | IDEXX |
| **6** | IDEXX |
| **7** | IDEXX |

**Table II :** **Laboratory results**

| N° | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| **1** | Y | Y | N | Y | N |
| **2** | Y | Y | N | Y | N |
| **3** | Y | Y | N | Y | N |
| **4** | Y | Y | N | Y | N |
| **5** | Y | Y | N | Y | N |
| **6** | Y | Y | N | Y | N |
| **7** | Y | Y | N | Y | N |
| **REF** | **Y** | **Y** | **N** | **Y** | **N** |

Answers : Y = YES; N = NO; to the questions: Presence of PAG (Pregnancy Associated Glycoproteins)

**Table III :**

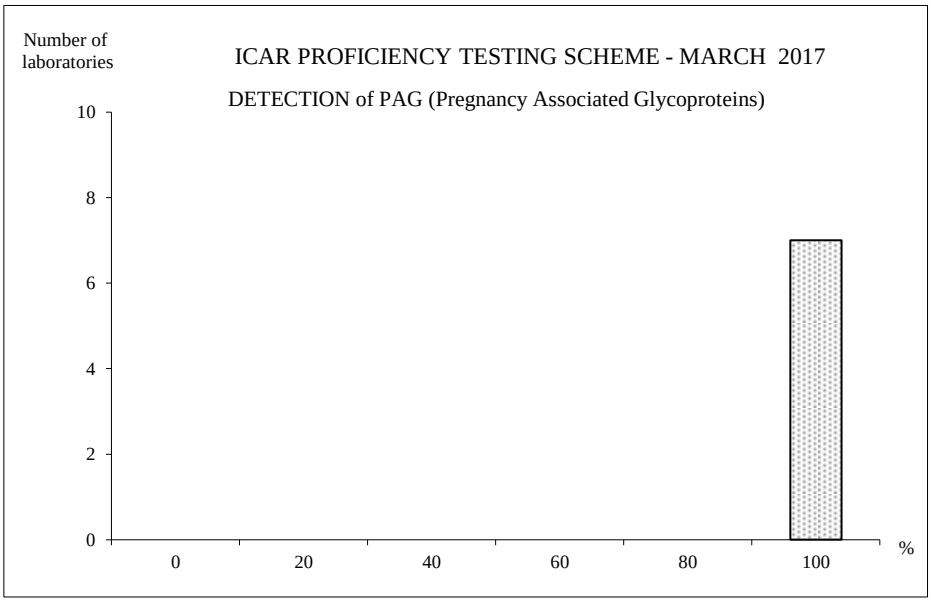
| SAMPLES | LACTATION PERIOD | Date |
|---|---|---|
| **1** | Pregnant - Artificial insemination | 19.12.2016 |
| **2** | Pregnant - Artificial insemination | 11.12.2016 |
| **3** | Non pregnant | — |
| **4** | Pregnant - Artificial insemination | 09.10.2016 |
| **5** | Non pregnant | — |

**Table IV :** **Laboratory accuracy with respect to correct results**

| N° | 1 | 2 | 3 | 4 | 5 | FLR% |
|---|---|---|---|---|---|---|
| **1** | T | T | T | T | T | 100 |
| **2** | T | T | T | T | T | 100 |
| **3** | T | T | T | T | T | 100 |
| **4** | T | T | T | T | T | 100 |
| **5** | T | T | T | T | T | 100 |
| **6** | T | T | T | T | T | 100 |
| **7** | T | T | T | T | T | 100 |
| NSR | 7 | 7 | 7 | 7 | 7 | |
| NS | 7 | 7 | 7 | 7 | 7 | |
| FSR% | 100 | 100 | 100 | 100 | 100 | |

T : True F : False

NSR : number of right answers per sample and criterion
NS : total number of answers per sample and criterion
FSR% : frequency in right answers per sample and criterion
FLR% : relative frequency in right answers per laboratory




List of participant
PAG
March 2017

BIOR INST.of FOOD SAFETY, ANIMAL HEALTH and ENVIROMENT
CROATIAN AGRICULTURAL AG. CENTRAL LAB.for Milk Quality Control
ESTONIAN LIVESTOCK PERFORMANCE RECORDING Ltd
EUROFINS STEINS LABORATORY A/S
LRV-LABORATORIO REGIONAL DE VETERINARIA
QLIP N.V.
THE CATTLE INFORMATION SERVICE