THE GLOBAL STANDARD
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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

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1. Introduction

Dear Participant,

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

In this report you will find sections 2 and 3 which are dedicated to "your" quality assurance management and section 4 dedicated to the "general" 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-Score_{PT} (standard deviation calculated on this proficiency test) and the Z-Score_{FIX} (standard deviation of the reference method) are summarized. If you have obtained all the -2<Z-Score results<+2 the cell will be filled in green. If you have obtained one or more results in the moderate or poor performance range the cells will be filled in yellow or red respectively.

G) In table 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.

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.

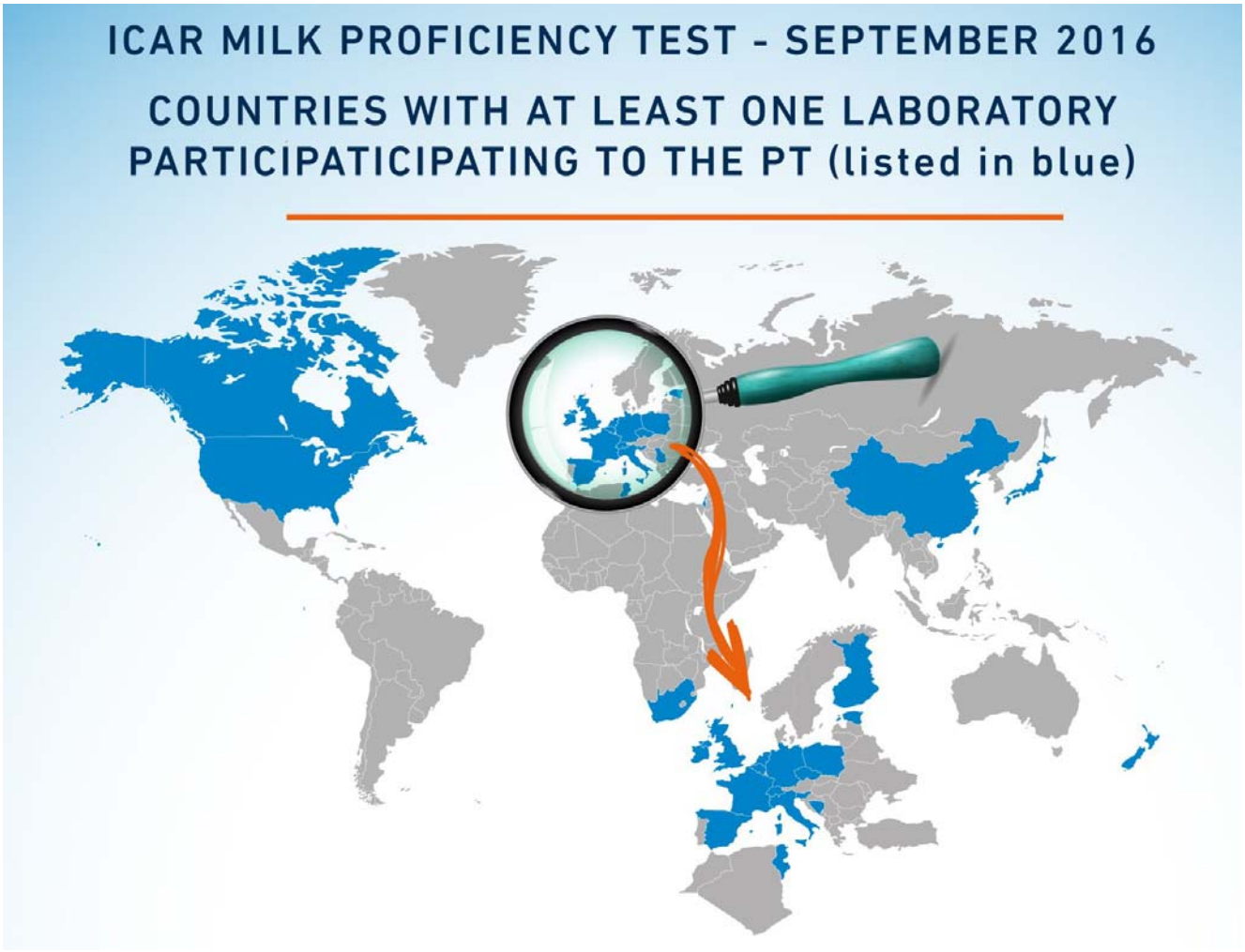
For each parameter the SR=standard deviation of reproducibility has been calculated after the outlier elimination. If you have participated, and your results are in the repeatability limits, you can use this value for the calculation of your uncertainty of measurement.

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/icar-certifications/milk-laboratories-proficiency-test-2016/


ICAR MILK PROFICIENCY TEST - SEPTEMBER 2016
COUNTRIES WITH AT LEAST ONE LABORATORY
PARTICIPATICIPATING TO THE PT (listed in blue)

ICAR Member having a milk laboratory participating in at least one ICAR Proficiency Round Test in 2016	
Comité du Lait ASBL	Belgium
Département Valorisation des Productions agricoles (DVP), Centre Wallon de Recherche Agronomique – CRA -W	Belgium
T&V – ILVO Eenheid Technologie en Voeding Productkwaliteit en Voedselveiligheid	Belgium
Valacta – Centre d'Expertise en Production Laitière du Québec	Canada
Dairy Cattle Research Center of Shandong Academy of Agricultural Science	China
University of Zagreb, Faculty of Agriculture, Department of Dairy Science, Reference Laboratory for Milk and Dairy Products	Croatia
Laborator pro rozbor mléka Bustehrad, Ceskomoravská spolecnost chovatelú a.s.	Czech Republic
Milkom a.s Dairy research institute	Czech Republic
Eurofins Steins Laboratorium A/S	Denmark
Eesti Pollumajandusloomade Joudluskontrolli AS, Milk Analysing Laboratory	Estonia
VALIO R & D Chemistry	Finland
ACTALIA / ACTILAIT / CECALAIT	France
Milchprüfring Baden-Württemberg e.V., Zentrallabor Kirchheim	Germany
ÁT. KFT. Tejvizsgáló Laboratórium (Livestock Performance Testing Ltd)	Hungary
Teagasc, Technical Services Laboratory	Ireland
Central Milk Laboratory – ICBA	Israel
Associazione Italiana Allevatori, Laboratorio Standard Latte (LSL-AIA)	Italy
Federazione Latterie Alto Adige Soc. Agr. Coop.	Italy
Japan Dairy Technical Association	Japan
State Laboratory for Milk Control Pieno Tyrimai	Lithuania
MilTestNZ	New Zealand
Laboratorium Oceny Mleka, Krajowego Centrum Hodowli Zwierzat (KCHZ), Laboratorium Referencyjne z siedziba w Parzniewie	Poland
PFHBiPM Laboratorium w Bialymstoku zs.w jezewie Starym	Poland
PFHBiPM Laboratorium w Kobiernie	Poland
PFHBiPM Laboratorium w Parzniewie	Poland
PFHBiPM Region Oceny Bydgoszcz z/s w Minikowie	Poland
Laboratorija za ispitivanje kvaliteta mleka, Poljoprivredni fakultet Novi Sad,	Serbia
Plemenárske sluzby SR s.p.	Slovak Republic
University of Ljubljana, Biotechnical Faculty, Zootech. Dept., Laboratory for Dairying	Slovenia
Deltamune	South Africa
Lactolab	South Africa
Merieux Nutriscience South Africa	South Africa

ICAR Member having a milk laboratory participating in at least one ICAR Proficiency Round Test in 2016	
Merieux Nutriscience South Africa (Midrand)	South Africa
Laboratorio Agroalimentario de Santander	Spain
Agroscope, Institute for Food Sciences IFS	Switzerland
Suisselab AG	Switzerland
Qlip N.V.	The Netherlands
Direction de l' Amelioration Genetique	Tunisia
Cattle Information Service (CIS)	United Kingdom
Eastern Laboratory Services	USA
Vetlab Agricultural Showgrouds	Zambia

ICAR Member with no milk laboratory participating in any ICAR Proficiency Round Test in 2016	
INTI - Lacteos	Argentina
Milchprüfring Süd	Austria
Shanghai Dairy Cattle Breeding Center Co., Ltd	China
Institute of Food Safety, Animal Health and Environment BIOR, Laboratory of Food and Environmental Investigations	Latvia
TINE, Centre for R&D	Norway
Milk Laboratory, Slovak Agricultural Research Centre	Slovak Republic
Veterinárny a potravinový ústav Bratislava	Slovak Republic
ANPI Dairy Laboratory	South Africa
Eurofins Steins Laboratorium AB	Sweden
Milk Analysis Laboratory, Hsin-chu Branch, Livestock Research Institute, Council of Agriculture	Taiwan
National Milk Records plc / Direct laboratories	UK

Attached to this report you find the certificate of your participation in the ICAR PT.-

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

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

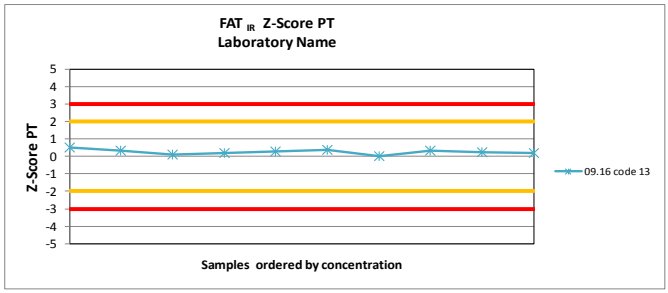
Legenda:

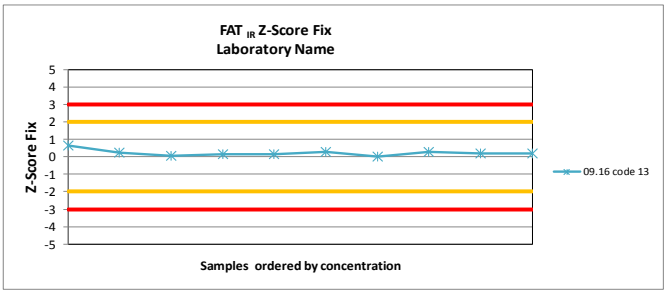
- positive performance for all the sample
- same sample showed a performance out of range or there are miss data
- The parameter was not analyzed

H

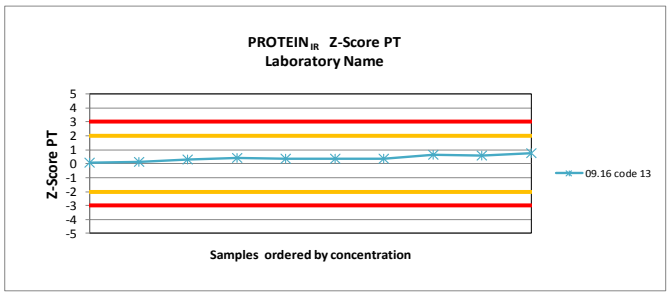
Control Charts

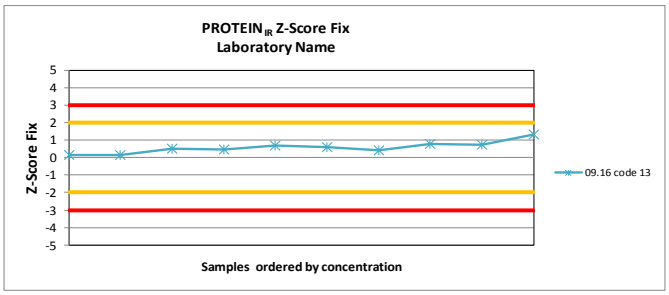
Control Charts Fat





Control Charts Protein





ICAR
PROFICIENCY TESTING SCHEME

September 2016

Raw Milk

Determination of FAT CONTENT
Routine method

Sending date of statistical treatment : 29th september 2016

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

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

Nb	%	N°	d	Sd	D	Method
1	5	13	+ 0,009	0,007	0,011	IR
2	11	19	+ 0,008	0,010	0,013	IR
3	16	11	+ 0,005	0,012	0,013	IR
4	21	10	- 0,012	0,006	0,014	IR
5	26	7	- 0,002	0,019	0,019	IR
6	32	16	- 0,015	0,013	0,021	IR
7	37	4	+ 0,018	0,012	0,021	IR
8	42	14	- 0,017	0,024	0,029	IR
9	47	1	+ 0,031	0,008	0,032	IR
10	53	6	+ 0,030	0,015	0,033	IR
11	58	2	- 0,029	0,016	0,034	IR
12	63	3	+ 0,041	0,008	0,041	IR
13	68	18	+ 0,030	0,029	0,042	IR
14	74	15	- 0,034	0,027	0,044	IR
15	79	5	- 0,040	0,026	0,048	IR
16	84	9	- 0,049	0,018	0,053	IR
17	89	17	+ 0,103	0,056	0,117	IR
18	95	8	+ 0,040	0,142	0,147	IR
19	100	12	+ 0,183	0,046	0,189	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 19 sets of results send by 19 laboratories using routine method ISO 9622 Idf 141, after outlier discarging using Grubbs test at 5 % risk level

(NC : OUT of RANKING because of insufficient data number)
(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)
(d et Sd : mean and standard deviation of the differences (laboratory -reference))
(D : Euclidian distance to YX-axis origin = SQUARE ROOT.(d^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}	0,005
Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)	SR_{PT}	0,034

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	0,010	0,010	0,010	0,010	0,000	0,000	0,000	0,010	0,006	20
2	0,010	0,010	0,010	0,000	0,000	0,010	0,000	0,000	0,020	0,020	0,008	20
3	0,010	0,010	0,000	0,000	0,020	0,000	0,010	0,000	0,000	0,010	0,006	20
4	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	20
5	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,010	0,010	0,003	20
6	0,010	0,000	0,000	0,000	0,010	0,010	0,000	0,010	0,000	0,000	0,004	20
7	0,000	0,000	0,000	0,000	0,010	0,000	0,010	0,010	0,020	0,010	0,006	20
8	0,010	0,000	0,010	0,000	0,000	0,010	0,250 *	0,000	0,000	0,000	0,056	20
9	0,020	0,010	0,000	0,000	0,000	0,000	0,010	0,010	0,000	0,010	0,006	20
10	0,000	0,000	0,000	0,010	0,000	0,000	0,010	0,000	0,000	0,000	0,003	20
11	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,000	0,010	0,003	20
12	0,010	0,010	0,010	0,010	0,010	0,010	0,000	0,010	0,010	0,010	0,007	20
13	0,009	0,010	0,009	0,000	0,009	0,000	0,010	0,000	0,000	0,010	0,005	20
14	0,000	0,000	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,002	20
15	0,010	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,000	0,003	20
16	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	20
17	0,000	0,020	0,040 *	0,030 *	0,030 *	0,050 *	0,060 *	0,030 *	0,030 *	0,070 *	0,029	20
18	0,135 *	0,000	0,000	0,000	0,000	0,020	0,000	0,000	0,010	0,000	0,031	20
19	0,000	0,000	0,010	0,000	0,000	0,000	0,000	0,010	0,000	0,010	0,004	20
Sr	0,023	0,005	0,008	0,006	0,007	0,010	0,042	0,006	0,007	0,013		380
NE	38	38	38	38	38	38	38	38	38	38		
L	0,022	0,020	0,016	0,011	0,020	0,020	0,016	0,014	0,021	0,023		

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	2,135	2,805	3,455	4,145	4,695	1,495	2,420	3,090	3,820	4,475
2	2,065	2,745	3,395	4,070	4,650	1,405	2,360	3,040	3,760	4,440
3	2,165	2,815	3,460	4,150	4,700	1,500	2,435	3,110	3,830	4,465
4	2,120	2,790	3,440	4,150	4,680	1,470	2,400	3,090	3,790	4,470
5	2,110	2,740	3,410	4,080	4,580	1,410	2,360	3,040	3,695	4,395
6	2,155	2,820	3,460	4,140	4,675	1,445	2,430	3,095	3,820	4,480
7	2,130	2,770	3,410	4,090	4,685	1,460	2,395	3,055	3,800	4,405
8	2,085	2,750	3,855 *	4,150	4,650	1,415	2,475	3,040	3,770	4,430
9	2,090	2,725	3,380	4,050	4,580	1,410	2,375	3,025	3,720	4,375
10	2,110	2,760	3,410	4,095	4,650	1,450	2,385	3,050	3,760	4,430
11	2,090	2,780	3,430	4,120	4,675	1,450	2,400	3,070	3,800	4,455
12	2,235 *	2,915 *	3,635 *	4,325 *	4,905 *	1,575	2,560 *	3,245 *	3,985 *	4,675 *
13	2,124	2,784	3,436	4,121	4,670	1,477	2,405	3,071	3,780	4,446
14	2,130	2,780	3,410	4,070	4,610	1,450	2,405	3,060	3,750	4,390
15	2,075	2,740	3,370	4,090	4,660	1,355	2,360	3,040	3,760	4,430
16	2,100	2,770	3,410	4,100	4,650	1,400	2,380	3,060	3,770	4,430
17	2,170	2,860	3,540 *	4,245 *	4,825 *	1,455	2,450	3,165 *	3,915 *	4,625 *
18	2,083	2,820	3,470	4,140	4,680	1,540	2,430	3,090	3,805	4,470
19	2,120	2,770	3,435	4,110	4,680	1,460	2,400	3,075	3,790	4,465
M	2,114	2,779	3,424	4,110	4,657	1,454	2,404	3,065	3,778	4,438
REF.	2,114	2,777	3,424	4,110	4,662	1,451	2,402	3,064	3,780	4,439
SD	0,030	0,035	0,029	0,032	0,036	0,051	0,032	0,024	0,036	0,032

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 19 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	18		17	17	17	17	8;17	17	17	17
Outlier Grubbs	12	12	8;12 17	12;17	12;17			12;17	12;17	12;17
sr	0,006	0,005	0,003	0,002	0,005	0,005	0,004	0,003	0,005	0,006
SR	0,030	0,035	0,029	0,033	0,036	0,053	0,025	0,024	0,036	0,032

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	+ 0,021	+ 0,028	+ 0,031	+ 0,035	+ 0,033	+ 0,044	+ 0,018	+ 0,026	+ 0,040	+ 0,036	+ 0,031	0,008	11,98
2	- 0,049	- 0,032	- 0,029	- 0,040	- 0,012	- 0,046	- 0,042	- 0,024	- 0,020	+ 0,001	- 0,029	0,016	5,82
3	+ 0,051	+ 0,038	+ 0,036	+ 0,040	+ 0,038	+ 0,049	+ 0,033	+ 0,046	+ 0,050	+ 0,026	+ 0,041	0,008	15,64
4	+ 0,006	+ 0,013	+ 0,016	+ 0,040	+ 0,018	+ 0,019	- 0,002	+ 0,026	+ 0,010	+ 0,031	+ 0,018	0,012	4,53
5	- 0,004	- 0,037	- 0,014	- 0,030	- 0,082	- 0,041	- 0,042	- 0,024	- 0,085	- 0,044	- 0,040	0,026	4,91
6	+ 0,041	+ 0,043	+ 0,036	+ 0,030	+ 0,013	- 0,006	+ 0,028	+ 0,031	+ 0,040	+ 0,041	+ 0,030	0,015	6,05
7	+ 0,016	- 0,007	- 0,014	- 0,020	+ 0,023	+ 0,009	- 0,007	- 0,009	+ 0,020	- 0,034	- 0,002	0,019	0,42
8	- 0,029	- 0,027	+ 0,431	+ 0,040	- 0,012	- 0,036	+ 0,073	- 0,024	- 0,010	- 0,009	+ 0,040	0,142	0,88
9	- 0,024	- 0,052	- 0,044	- 0,060	- 0,082	- 0,041	- 0,027	- 0,039	- 0,060	- 0,064	- 0,049	0,018	8,81
10	- 0,004	- 0,017	- 0,014	- 0,015	- 0,012	- 0,001	- 0,017	- 0,014	- 0,020	- 0,009	- 0,012	0,006	6,49
11	- 0,024	+ 0,003	+ 0,006	+ 0,010	+ 0,013	- 0,001	- 0,002	+ 0,006	+ 0,020	+ 0,016	+ 0,005	0,012	1,16
12	+ 0,121	+ 0,138	+ 0,211	+ 0,215	+ 0,243	+ 0,124	+ 0,158	+ 0,181	+ 0,205	+ 0,236	+ 0,183	0,046	12,68
13	+ 0,010	+ 0,007	+ 0,011	+ 0,011	+ 0,008	+ 0,026	+ 0,003	+ 0,007	- 0,000	+ 0,007	+ 0,009	0,007	4,02
14	+ 0,016	+ 0,003	- 0,014	- 0,040	- 0,052	- 0,001	+ 0,003	- 0,004	- 0,030	- 0,049	- 0,017	0,024	2,23
15	- 0,039	- 0,037	- 0,054	- 0,020	- 0,002	- 0,096	- 0,042	- 0,024	- 0,020	- 0,009	- 0,034	0,027	4,05
16	- 0,014	- 0,007	- 0,014	- 0,010	- 0,012	- 0,051	- 0,022	- 0,004	- 0,010	- 0,009	- 0,015	0,013	3,64
17	+ 0,056	+ 0,083	+ 0,116	+ 0,135	+ 0,163	+ 0,004	+ 0,048	+ 0,101	+ 0,135	+ 0,186	+ 0,103	0,056	5,79
18	- 0,031	+ 0,043	+ 0,046	+ 0,030	+ 0,018	+ 0,089	+ 0,028	+ 0,026	+ 0,025	+ 0,031	+ 0,030	0,029	3,25
19	+ 0,006	- 0,007	+ 0,011	- 0,000	+ 0,018	+ 0,009	- 0,002	+ 0,011	+ 0,010	+ 0,026	+ 0,008	0,010	2,58
d	+ 0,000	+ 0,002	- 0,000	- 0,000	- 0,005	+ 0,003	+ 0,001	+ 0,000	- 0,002	- 0,001	+ 0,015	0,065	
Sd	0,030	0,035	0,029	0,032	0,036	0,051	0,032	0,024	0,036	0,032	0,034		

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	+0,70	+0,79	+1,05	+1,06	+0,92	+0,85	+0,55	+1,05	+1,12	+1,12
2	-1,61	-0,93	-0,99	-1,25	-0,33	-0,90	-1,32	-1,01	-0,56	+0,03
3	+1,68	+1,08	+1,22	+1,22	+1,06	+0,95	+1,02	+1,88	+1,40	+0,81
4	+0,20	+0,36	+0,54	+1,22	+0,50	+0,37	-0,07	+1,05	+0,28	+0,97
5	-0,13	-1,08	-0,48	-0,94	-2,27	-0,80	-1,32	-1,01	-2,38	-1,37
6	+1,35	+1,22	+1,22	+0,91	+0,36	-0,12	+0,86	+1,26	+1,12	+1,28
7	+0,53	-0,22	-0,48	-0,63	+0,64	+0,17	-0,23	-0,39	+0,56	-1,06
8	-0,95	-0,79	+14,70	+1,22	-0,33	-0,71	+2,27	-1,01	-0,28	-0,28
9	-0,79	-1,51	-1,51	-1,86	-2,27	-0,80	-0,85	-1,62	-1,68	-1,99
10	-0,13	-0,50	-0,48	-0,48	-0,33	-0,02	-0,54	-0,60	-0,56	-0,28
11	-0,79	+0,07	+0,20	+0,29	+0,36	-0,02	-0,07	+0,23	+0,56	+0,50
12	+3,99	+3,95	+7,19	+6,61	+6,75	+2,41	+4,92	+7,43	+5,75	+7,36
13	+0,32	+0,19	+0,39	+0,33	+0,21	+0,50	+0,08	+0,27	-0,00	+0,22
14	+0,53	+0,07	-0,48	-1,25	-1,44	-0,02	+0,08	-0,18	-0,84	-1,53
15	-1,28	-1,08	-1,85	-0,63	-0,05	-1,88	-1,32	-1,01	-0,56	-0,28
16	-0,46	-0,22	-0,48	-0,32	-0,33	-1,00	-0,70	-0,18	-0,28	-0,28
17	+1,85	+2,37	+3,95	+4,14	+4,53	+0,07	+1,49	+4,14	+3,78	+5,80
18	-1,03	+1,22	+1,57	+0,91	+0,50	+1,73	+0,86	+1,05	+0,70	+0,97
19	+0,20	-0,22	+0,37	-0,01	+0,50	+0,17	-0,07	+0,43	+0,28	+0,81

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

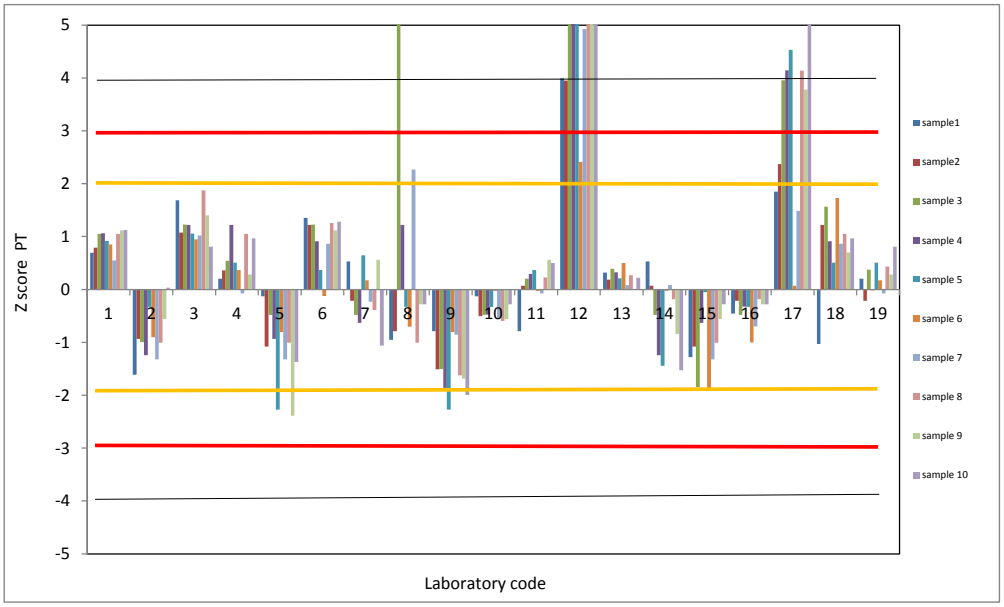


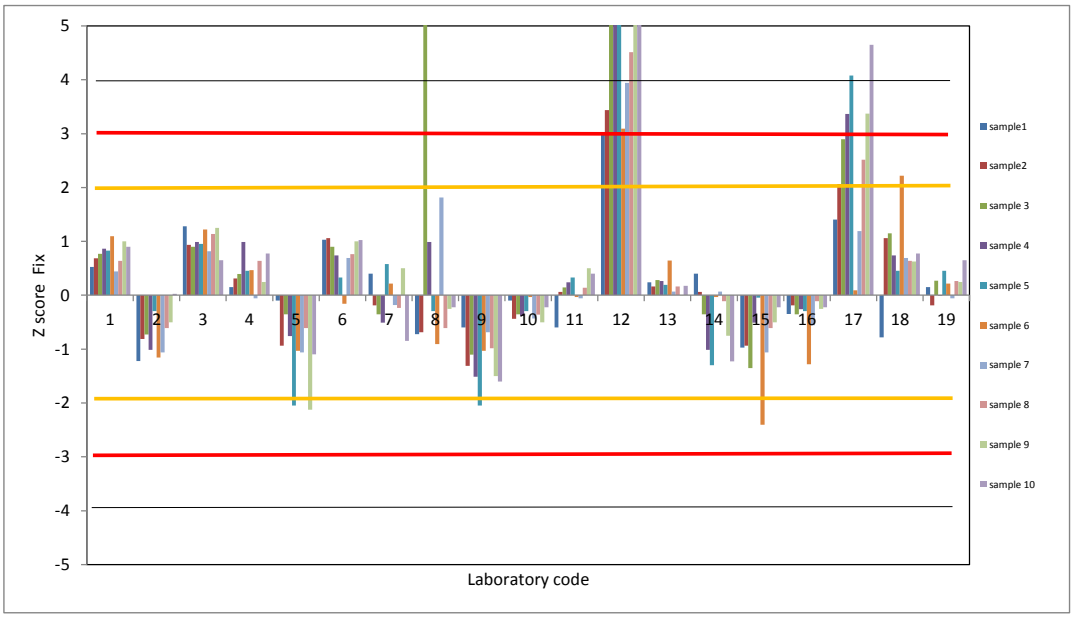
Table VII : **Zscore of the different laboratories for each sample.**
ZS calculated on the standard deviation of reproducibility of the method

Sample / Lab Code	1	2	3	4	5	6	7	8	9	10
1	+0,53	+0,69	+0,77	+0,86	+0,83	+1,09	+0,44	+0,64	+1,00	+0,90
2	-1,22	-0,81	-0,73	-1,01	-0,30	-1,16	-1,06	-0,61	-0,50	+0,03
3	+1,28	+0,94	+0,90	+0,99	+0,95	+1,22	+0,82	+1,14	+1,25	+0,65
4	+0,15	+0,31	+0,40	+0,99	+0,45	+0,47	-0,06	+0,64	+0,25	+0,78
5	-0,10	-0,94	-0,35	-0,76	-2,05	-1,03	-1,06	-0,61	-2,13	-1,10
6	+1,03	+1,06	+0,90	+0,74	+0,33	-0,16	+0,69	+0,76	+1,00	+1,03
7	+0,40	-0,19	-0,35	-0,51	+0,58	+0,22	-0,18	-0,24	+0,50	-0,85
8	-0,72	-0,69	+10,77	+0,99	-0,30	-0,91	+1,82	-0,61	-0,25	-0,22
9	-0,60	-1,31	-1,10	-1,51	-2,05	-1,03	-0,68	-0,99	-1,50	-1,60
10	-0,10	-0,44	-0,35	-0,39	-0,30	-0,03	-0,43	-0,36	-0,50	-0,22
11	-0,60	+0,06	+0,15	+0,24	+0,33	-0,03	-0,06	+0,14	+0,50	+0,40
12	+3,03	+3,44	+5,27	+5,36	+6,08	+3,09	+3,94	+4,51	+5,12	+5,90
13	+0,24	+0,16	+0,28	+0,26	+0,19	+0,64	+0,07	+0,16	-0,00	+0,18
14	+0,40	+0,06	-0,35	-1,01	-1,30	-0,03	+0,07	-0,11	-0,75	-1,22
15	-0,97	-0,94	-1,35	-0,51	-0,05	-2,41	-1,06	-0,61	-0,50	-0,22
16	-0,35	-0,19	-0,35	-0,26	-0,30	-1,28	-0,56	-0,11	-0,25	-0,22
17	+1,40	+2,06	+2,90	+3,36	+4,08	+0,09	+1,19	+2,51	+3,37	+4,65
18	-0,78	+1,06	+1,15	+0,74	+0,45	+2,22	+0,69	+0,64	+0,62	+0,78
19	+0,15	-0,19	+0,27	-0,01	+0,45	+0,22	-0,06	+0,26	+0,25	+0,65

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



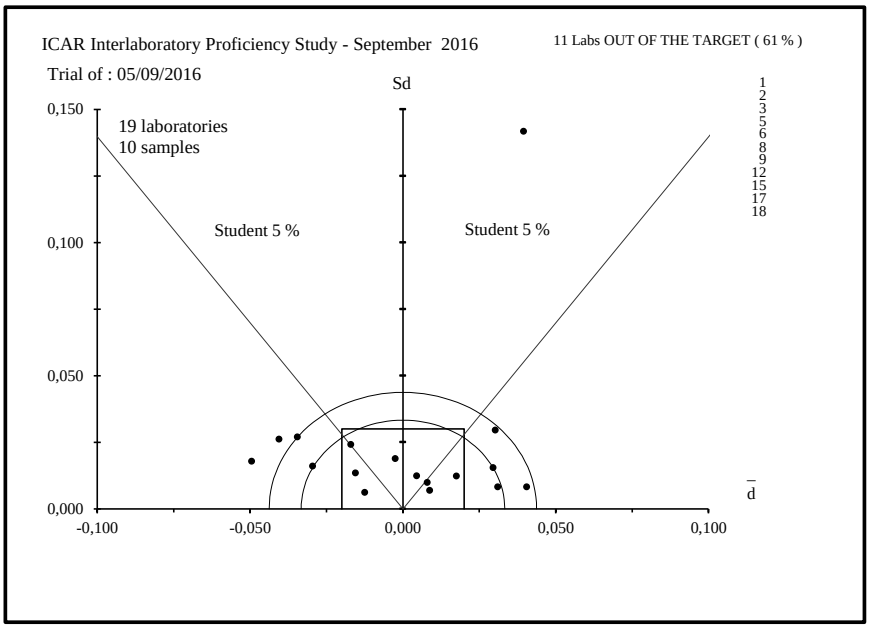


Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).

LIST OF THE PARTICIPANTS ICAR
ICAR PROFICIENCY TEST

RAW MILK
FAT CONTENT Infrared method
Sep 16

AIA-LAB. STANDARD LATTE	MACCARESE (ROMA)	ITALY
CATTLE INFORMATION SERVICE (CIS)	TELFORD	UNITED KINGDOM
CENTRAL MILK LABORATORY - ICBA	CAESAREA	ISRAEL
COMITÉ DU LAIT ASBL	BATTICE	BELGIUM
DAIRY CATTLE RESEARCH	SHANDONG	CHINA
DELTAMUNE	PRETORIA	SOUTH AFRICA
DIR. DE L'AMELIORATION GENETIQUE	SIDI THABET	TUNISIA
EASTERN LAB SERVICES	MEDINA	USA
FED.LATTERIE SOCIALI DI BOLZANO	BOLZANO	ITALY
LAB PRO ROZBOR MLÉKA	BUSTEHRAD	CHECH REPUBLIC
LAB. POLJOPRIVREDNI	NOVI SAD	SERBIA
LACTOLAB	IRENE	SOUTH AFRICA
LOM KCHZ LABORATORIUM REFERENCYJNE z/s w	PARZNIEWIE	POLAND
MERIEUX NUTRISCIENCE	MIDRAND	SOUTH AFRICA
MILCHPRUFRING BADEN-WURTTEMBERG E.V.	KIRCHEIM / TECK	GERMANY
OSUUSKUNTA SATAMAITO	PORI	FINLAND
PLEMENÁRSKE SLUZBY SR S.P.	ZILINA	SLOVAKIA
SUISSE LAB AG	ZOLLIKOFEN	SWITZERLAND
VETLAB AGRICULTURAL SHOWGROUNDS	LUSAKA	ZAMBIA

ICAR
PROFICIENCY TESTING SCHEME

September 2016

Raw Milk

Determination of CRUDE PROTEIN CONTENT

Routine method

Sending date of statistical treatment : 29th september 2016

Frame of activity :	ICAR Milk Analyses Sub Committee (MA SC)	
Contact :	Gavin Scott	gavin@milktest.co.nz
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Table I : **Ranking of the laboratories** Units : g / 100 g

Nb	%	N°	d	Sd	D	Method
1	5	15	- 0,001	0,009	0,009	IR
2	11	3	- 0,008	0,007	0,011	IR
3	16	19	+ 0,021	0,007	0,022	IR
4	21	16	- 0,021	0,015	0,026	IR
5	26	13	+ 0,023	0,013	0,027	IR
6	32	11	- 0,028	0,010	0,030	IR
7	37	10	+ 0,037	0,009	0,038	IR
8	42	5	- 0,023	0,035	0,042	IR
9	47	7	+ 0,041	0,008	0,042	IR
10	53	4	+ 0,045	0,008	0,046	IR
11	58	14	- 0,052	0,012	0,053	IR
12	63	8	- 0,026	0,048	0,055	IR
13	68	12	- 0,058	0,030	0,066	IR
14	74	2	+ 0,048	0,049	0,068	IR
15	79	6	+ 0,100	0,013	0,101	IR
16	84	1	- 0,165	0,016	0,166	IR
17	89	18	+ 0,117	0,296	0,318	IR
18	95	9	+ 0,079	0,389	0,397	IR
19	100	17	+ 0,778	0,179	0,798	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 18 sets of results send by 19 laboratories using routine method ISO 9622 Idgf 141, after outlier discarging using Grubbs test at 5 % risk level

(NC : OUT of RANKING because of insufficient data number)
(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)
(d et Sd : mean and standard deviation of the differences (laboratory -reference))
(D : Euclidian distance to YX-axis origin = SQUARE ROOT.(d^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}	0,006
Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)	SR_{PT}	0,057

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	0,000	0,010	0,020	0,040 *	0,010	0,010	0,010	0,030	0,020	0,014	20
2	0,000	0,010	0,010	0,010	0,010	0,000	0,020	0,000	0,010	0,000	0,007	20
3	0,000	0,000	0,000	0,000	0,010	0,000	0,010	0,010	0,000	0,010	0,004	20
4	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	20
5	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,010	0,010	0,010	0,004	20
6	0,000	0,000	0,010	0,000	0,010	0,000	0,000	0,010	0,010	0,000	0,004	20
7	0,010	0,020	0,020	0,010	0,000	0,020	0,010	0,010	0,020	0,000	0,010	20
8	0,020	0,000	0,000	0,000	0,024	0,010	0,000	0,000	0,010	0,010	0,008	20
9	0,020	0,010	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,005	20
10	0,000	0,000	0,010	0,010	0,010	0,000	0,000	0,000	0,000	0,000	0,004	20
11	0,000	0,000	0,010	0,000	0,010	0,010	0,000	0,010	0,010	0,000	0,005	20
12	0,010	0,020	0,010	0,020	0,020	0,010	0,020	0,020	0,020	0,020	0,012	20
13	0,010	0,000	0,000	0,000	0,000	0,000	0,010	0,019	0,000	0,010	0,006	20
14	0,010	0,010	0,010	0,000	0,010	0,010	0,010	0,000	0,010	0,010	0,006	20
15	0,010	0,010	0,000	0,000	0,000	0,010	0,010	0,010	0,000	0,010	0,005	20
16	0,000	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,000	0,002	20
17	0,010	0,000	0,000	0,000	0,020	0,000	0,010	0,000	0,000	0,010	0,006	20
18	0,507 *	0,000	0,010	0,010	0,010	0,000	0,000	0,000	0,000	0,000	0,113	20
19	0,000	0,000	0,000	0,010	0,000	0,010	0,010	0,010	0,010	0,000	0,005	20
Sr	0,082	0,006	0,006	0,006	0,010	0,006	0,007	0,006	0,008	0,006		380
NE	38	38	38	38	38	38	38	38	38	38		
L	0,025	0,022	0,022	0,023	0,029	0,022	0,026	0,025	0,031	0,025		

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	2,515	2,710	3,065	3,340 *	3,540	3,075	2,615	2,875 *	3,245 *	3,430 *
2	2,760	2,915	3,275	3,535	3,695	3,370	2,850	3,100	3,435	3,600
3	2,650	2,840	3,220	3,510	3,715	3,240	2,745	3,035	3,410	3,615
4	2,710	2,880	3,270	3,560	3,770	3,280	2,810	3,090	3,470	3,670
5	2,590	2,800	3,210	3,520	3,740	3,210	2,700	3,005	3,415	3,635
6	2,750	2,960	3,335	3,620	3,825	3,320	2,850	3,145	3,525	3,730
7	2,695	2,900	3,270	3,555	3,760	3,280	2,805	3,085	3,460	3,660
8	2,680	2,860	3,160	3,450	3,628	3,295	2,780	3,030	3,375	3,545
9	2,090 *	2,725	3,380	4,050 *	4,580 *	3,210	2,705	3,020	3,440	3,650
10	2,690	2,880	3,265	3,555	3,765	3,280	2,790	3,080	3,460	3,660
11	2,630	2,830	3,205	3,490	3,695	3,195	2,730	3,015	3,395	3,590
12	2,645	2,830	3,155	3,430	3,620	3,215	2,730	2,980	3,340	3,530
13	2,677	2,867	3,256	3,547	3,761	3,265	2,765	3,062	3,440	3,649
14	2,595	2,795	3,185	3,470	3,675	3,175	2,705	2,990	3,375	3,575
15	2,655	2,855	3,230	3,520	3,720	3,225	2,755	3,045	3,420	3,625
16	2,650	2,850	3,210	3,490	3,680	3,205	2,760	3,030	3,390	3,580
17	3,175 *	3,470 *	4,020 *	4,450 *	4,740 *	4,020 *	3,325 *	3,740 *	4,300 *	4,595 *
18	2,873	2,810	3,185	3,475	3,675	3,200	2,729	3,000	3,810 *	4,470 *
19	2,690	2,880	3,250	3,545	3,730	3,255	2,785	3,065	3,435	3,630
M	2,674	2,844	3,229	3,517	3,706	3,239	2,756	3,046	3,424	3,622
REF.	2,671	2,847	3,229	3,516	3,709	3,241	2,758	3,044	3,423	3,620
SD	0,078	0,062	0,070	0,048	0,068	0,065	0,057	0,044	0,045	0,051

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 18 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	18				1					
Outlier Grubbs	9;17	17	17	1;9 17	9;17	17	17	1;17	1;17 18	1;17 18
sr	0,006	0,006	0,006	0,005	0,007	0,006	0,007	0,007	0,007	0,006
SR	0,061	0,062	0,071	0,048	0,055	0,065	0,058	0,045	0,045	0,051

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	- 0,156	- 0,137	- 0,164	- 0,176	- 0,169	- 0,166	- 0,143	- 0,169	- 0,178	- 0,190	- 0,165	0,016	32,91
2	+ 0,089	+ 0,068	+ 0,046	+ 0,019	- 0,014	+ 0,129	+ 0,092	+ 0,056	+ 0,012	- 0,020	+ 0,048	0,049	3,11
3	- 0,021	- 0,007	- 0,009	- 0,006	+ 0,006	- 0,001	- 0,013	- 0,009	- 0,013	- 0,005	- 0,008	0,007	3,31
4	+ 0,039	+ 0,033	+ 0,041	+ 0,044	+ 0,061	+ 0,039	+ 0,052	+ 0,046	+ 0,047	+ 0,050	+ 0,045	0,008	17,76
5	- 0,081	- 0,047	- 0,019	+ 0,004	+ 0,031	- 0,031	- 0,058	- 0,039	- 0,008	+ 0,015	- 0,023	0,035	2,12
6	+ 0,079	+ 0,113	+ 0,106	+ 0,104	+ 0,116	+ 0,079	+ 0,092	+ 0,101	+ 0,102	+ 0,110	+ 0,100	0,013	24,39
7	+ 0,024	+ 0,053	+ 0,041	+ 0,039	+ 0,051	+ 0,039	+ 0,047	+ 0,041	+ 0,037	+ 0,040	+ 0,041	0,008	16,12
8	+ 0,008	+ 0,012	- 0,069	- 0,066	- 0,081	+ 0,054	+ 0,021	- 0,014	- 0,048	- 0,075	- 0,026	0,048	1,69
9	- 0,581	- 0,122	+ 0,151	+ 0,534	+ 0,871	- 0,031	- 0,053	- 0,024	+ 0,017	+ 0,030	+ 0,079	0,389	0,64
10	+ 0,019	+ 0,033	+ 0,036	+ 0,039	+ 0,056	+ 0,039	+ 0,032	+ 0,036	+ 0,037	+ 0,040	+ 0,037	0,009	12,54
11	- 0,041	- 0,017	- 0,024	- 0,026	- 0,014	- 0,046	- 0,028	- 0,029	- 0,028	- 0,030	- 0,028	0,010	9,34
12	- 0,026	- 0,017	- 0,074	- 0,086	- 0,089	- 0,026	- 0,028	- 0,064	- 0,083	- 0,090	- 0,058	0,030	6,09
13	+ 0,006	+ 0,020	+ 0,027	+ 0,031	+ 0,052	+ 0,024	+ 0,007	+ 0,018	+ 0,017	+ 0,029	+ 0,023	0,013	5,45
14	- 0,076	- 0,052	- 0,044	- 0,046	- 0,034	- 0,066	- 0,053	- 0,054	- 0,048	- 0,045	- 0,052	0,012	13,77
15	- 0,016	+ 0,008	+ 0,001	+ 0,004	+ 0,011	- 0,016	- 0,003	+ 0,001	- 0,003	+ 0,005	- 0,001	0,009	0,27
16	- 0,021	+ 0,003	- 0,019	- 0,026	- 0,029	- 0,036	+ 0,002	- 0,014	- 0,033	- 0,040	- 0,021	0,015	4,60
17	+ 0,504	+ 0,623	+ 0,791	+ 0,934	+ 1,031	+ 0,779	+ 0,567	+ 0,696	+ 0,877	+ 0,975	+ 0,778	0,179	13,75
18	+ 0,202	- 0,038	- 0,044	- 0,041	- 0,034	- 0,040	- 0,029	- 0,044	+ 0,387	+ 0,850	+ 0,117	0,296	1,25
19	+ 0,019	+ 0,033	+ 0,021	+ 0,029	+ 0,021	+ 0,014	+ 0,027	+ 0,021	+ 0,012	+ 0,010	+ 0,021	0,007	8,92
d	+ 0,003	- 0,004	+ 0,000	+ 0,001	- 0,003	- 0,002	- 0,002	+ 0,002	+ 0,001	+ 0,001	+ 0,048	0,217	
Sd	0,078	0,062	0,070	0,048	0,068	0,065	0,057	0,044	0,045	0,051	0,060		

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	-2,00	-2,23	-2,33	-3,64	-2,49	-2,57	-2,50	-3,80	-3,99	-3,75
2	+1,14	+1,10	+0,65	+0,40	-0,20	+2,00	+1,60	+1,27	+0,27	-0,40
3	-0,27	-0,12	-0,13	-0,12	+0,09	-0,01	-0,23	-0,20	-0,29	-0,10
4	+0,50	+0,53	+0,58	+0,91	+0,91	+0,61	+0,90	+1,04	+1,06	+0,98
5	-1,04	-0,77	-0,27	+0,09	+0,46	-0,47	-1,02	-0,87	-0,18	+0,29
6	+1,01	+1,83	+1,50	+2,16	+1,72	+1,23	+1,60	+2,28	+2,29	+2,16
7	+0,31	+0,85	+0,58	+0,81	+0,76	+0,61	+0,81	+0,93	+0,83	+0,78
8	+0,11	+0,20	-0,98	-1,37	-1,19	+0,84	+0,37	-0,31	-1,08	-1,48
9	-7,43	-1,99	+2,14	+11,06	+12,85	-0,47	-0,93	-0,53	+0,38	+0,59
10	+0,24	+0,53	+0,51	+0,81	+0,83	+0,61	+0,55	+0,82	+0,83	+0,78
11	-0,53	-0,28	-0,34	-0,53	-0,20	-0,71	-0,49	-0,65	-0,62	-0,60
12	-0,33	-0,28	-1,05	-1,78	-1,31	-0,40	-0,49	-1,44	-1,86	-1,78
13	+0,08	+0,32	+0,38	+0,65	+0,77	+0,38	+0,12	+0,40	+0,38	+0,57
14	-0,97	-0,85	-0,63	-0,95	-0,50	-1,02	-0,93	-1,21	-1,07	-0,89
15	-0,21	+0,12	+0,01	+0,09	+0,17	-0,24	-0,06	+0,03	-0,06	+0,09
16	-0,27	+0,04	-0,27	-0,53	-0,42	-0,55	+0,03	-0,31	-0,74	-0,80
17	+6,44	+10,11	+11,23	+19,35	+15,21	+12,07	+9,87	+15,69	+19,67	+19,23
18	+2,58	-0,61	-0,63	-0,85	-0,50	-0,63	-0,50	-0,99	+8,68	+16,76
19	+0,24	+0,53	+0,30	+0,60	+0,32	+0,22	+0,46	+0,48	+0,27	+0,19

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

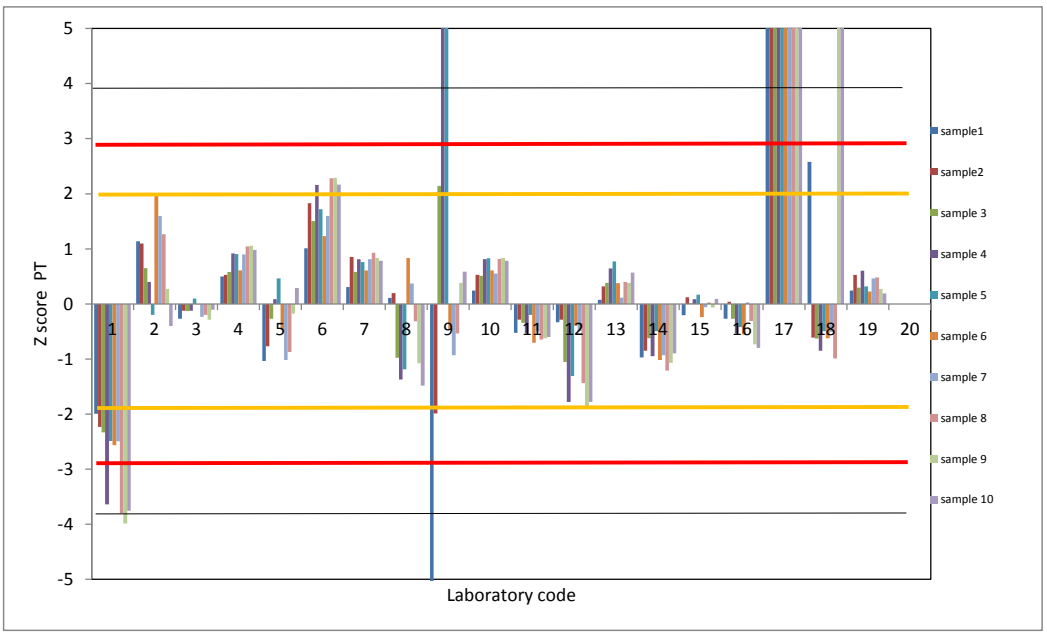


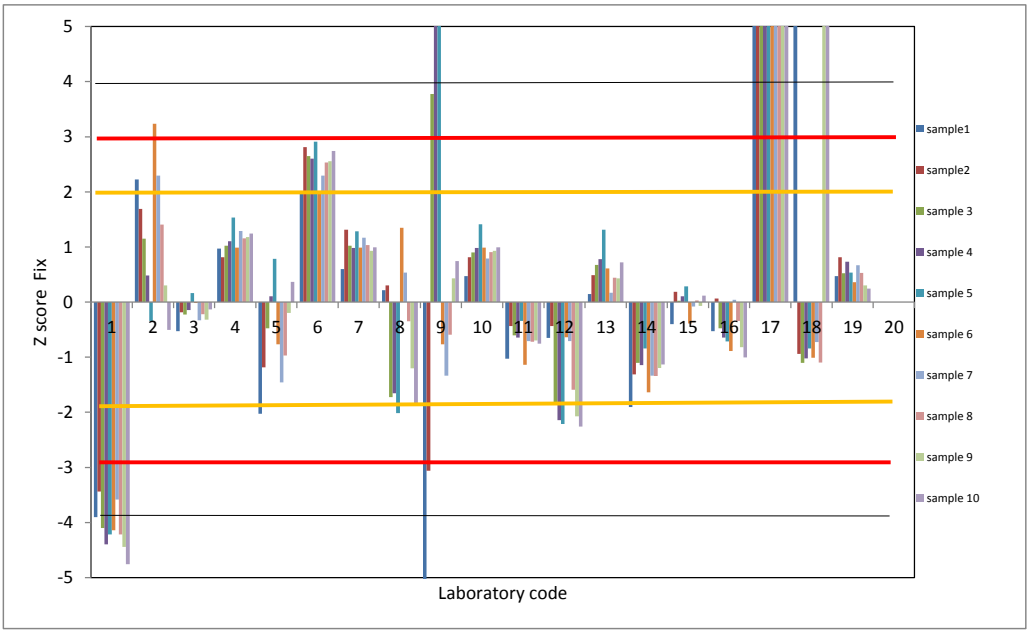
Table VII : **Zscore of the different laboratories for each sample.**
ZS calculated on the standard deviation of reproducibility of the method

Sample / Lab code	1	2	3	4	5	6	7	8	9	10
1	-3,90	-3,44	-4,10	-4,40	-4,21	-4,14	-3,58	-4,22	-4,45	-4,76
2	+2,22	+1,69	+1,15	+0,48	-0,34	+3,23	+2,29	+1,41	+0,30	-0,51
3	-0,53	-0,19	-0,23	-0,15	+0,16	-0,02	-0,33	-0,22	-0,32	-0,13
4	+0,97	+0,81	+1,02	+1,10	+1,54	+0,98	+1,29	+1,16	+1,18	+1,24
5	-2,03	-1,19	-0,48	+0,10	+0,79	-0,77	-1,46	-0,97	-0,20	+0,37
6	+1,97	+2,81	+2,65	+2,60	+2,91	+1,98	+2,29	+2,53	+2,55	+2,74
7	+0,60	+1,31	+1,02	+0,98	+1,29	+0,98	+1,17	+1,03	+0,93	+0,99
8	+0,21	+0,30	-1,73	-1,65	-2,02	+1,35	+0,54	-0,35	-1,20	-1,88
9	-14,53	-3,06	+3,77	+13,35	+21,79	-0,77	-1,33	-0,59	+0,43	+0,74
10	+0,47	+0,81	+0,90	+0,98	+1,41	+0,98	+0,79	+0,91	+0,93	+0,99
11	-1,03	-0,44	-0,60	-0,65	-0,34	-1,14	-0,71	-0,72	-0,70	-0,76
12	-0,65	-0,44	-1,85	-2,15	-2,21	-0,64	-0,71	-1,59	-2,07	-2,26
13	+0,15	+0,49	+0,67	+0,78	+1,31	+0,61	+0,17	+0,44	+0,43	+0,72
14	-1,90	-1,31	-1,10	-1,15	-0,84	-1,64	-1,33	-1,34	-1,20	-1,13
15	-0,40	+0,19	+0,02	+0,10	+0,29	-0,39	-0,08	+0,03	-0,07	+0,12
16	-0,53	+0,06	-0,48	-0,65	-0,71	-0,89	+0,04	-0,34	-0,82	-1,01
17	+12,60	+15,56	+19,77	+23,35	+25,79	+19,48	+14,17	+17,41	+21,93	+24,37
18	+5,05	-0,94	-1,10	-1,02	-0,84	-1,01	-0,72	-1,10	+9,68	+21,24
19	+0,47	+0,81	+0,52	+0,73	+0,54	+0,36	+0,67	+0,53	+0,30	+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=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



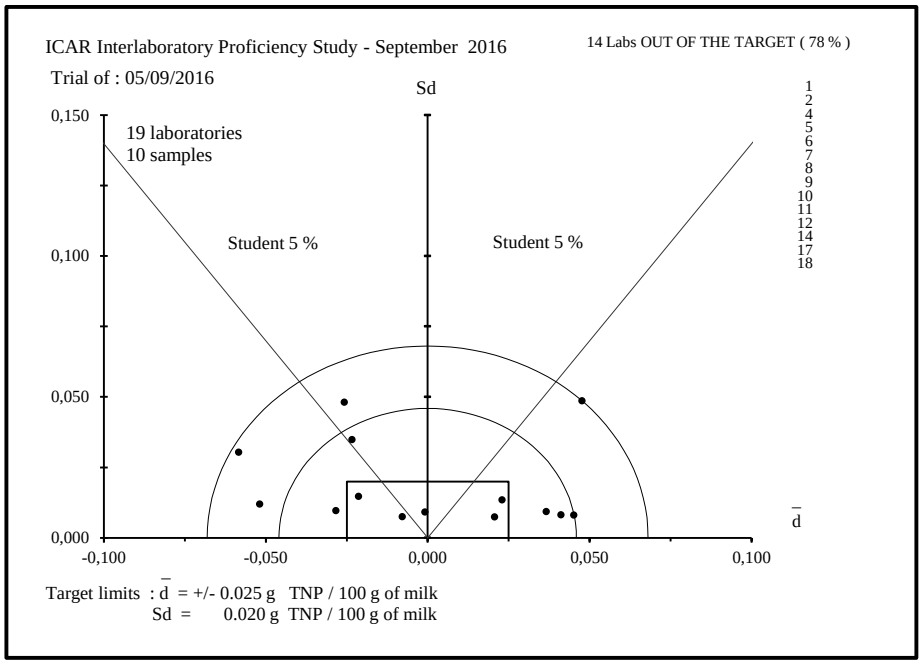


Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).

LIST OF THE PARTICIPANTS ICAR
ICAR PROFICIENCY TEST
RAW MILK

Determination of CRUDE PROTEIN CONTENT
September

Name	City	Country
AIA-LAB. STANDARD LATTE	MACARESE (ROMA)	ITALY
CATTLE INFORMATION SERVICE	TELFORD	UNITED KINGDOM
CENTRAL MILK LABORATORY - ICBA	CAESAREA	ISRAEL
COMITÉ DU LAIT ASBL	BATTICE	BELGIUM
DAIRY CATTLE RESEARCH	SHANDONG	CHINA
DELTAMUNE	PRETORIA	SOUTH AFRICA
DIR. DE L'AMELIORATION GENETIQUE	SIDI THABET	TUNISIA
EASTERN LAB SERVICES	MEDINA	USA
FED.LATTERIE SOCIALI DI BOLZANO	BOLZANO	ITALY
LAB PRO ROZBOR MLÉKA	BUSTEHRAD	CHECH REPUBLIC
LAB. OCENY MLEKA KCHZ	PARZNIEWIE	POLAND
LACTOLAB	IRENE	SOUTH AFRICA
MERIEUX NUTRISCIENCE	MIDRAND	SOUTH AFRICA
MILCHPRUFRING BADEN-WURTTEMBERG E.V.	KIRCHEIM / TECK	GERMANY
OSUUSKUNTA SATAMAITO	PORI	FINLAND
PLEMENÁRSKE SLUZBY SR S.P.	ZILINA	SLOVAKIA
SUISSE LAB AG	ZOLLIKOFEN	SWITZERLAND
UNIV. NOVI SAD LAB. POLJOPRIVREDNI	NOVI SAD	SERBIA
VETLAB AGRICULTURAL SHOWGROUNDS	LUSAKA	ZAMBIA

ICAR
PROFICIENCY TESTING SCHEME

September 2016

Raw Milk

Determination of LACTOSE CONTENT

Routine method

Sending date of statistical treatment : 29th september 2016

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

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

Nb	%	N°	d	Sd	D
1	6	16	- 0,017	0,020	0,026
2	12	6	- 0,017	0,023	0,029
3	18	14	- 0,033	0,018	0,038
4	24	11	- 0,015	0,035	0,038
5	29	8	- 0,002	0,041	0,041
6	35	10	- 0,052	0,015	0,054
7	41	9	- 0,056	0,017	0,059
8	47	2	- 0,062	0,031	0,070
9	53	4	+ 0,077	0,026	0,081
10	59	13	+ 0,084	0,029	0,089
11	65	12	+ 0,119	0,020	0,120
12	71	1	- 0,059	0,120	0,134
13	76	3	- 0,052	0,137	0,146
14	82	15	+ 0,179	0,054	0,187
15	88	5	+ 0,187	0,028	0,189
16	94	17	- 0,128	0,143	0,192
17	100	7	- 0,297	0,011	0,297

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 Idgf 141, after outlier discarging using Grubbs test at 5 % risk level

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

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

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

(D : Euclidian distance to YX-axis origin = SQUARE ROOT.(d^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}	0,006
Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)	SR_{PT}	0,116

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	0,000	0,020	0,020	0,000	0,010	0,020	0,010	0,010	0,010	0,000	0,009	20
2	0,000	0,000	0,000	0,010	0,000	0,000	0,010	0,010	0,000	0,000	0,004	20
3	0,000	0,000	0,000	0,010	0,000	0,010	0,010	0,010	0,010	0,010	0,005	20
4	0,020 *	0,000	0,010	0,000	0,000	0,010	0,000	0,010	0,010	0,000	0,006	20
5	0,000	0,000	0,000	0,011	0,000	0,010	0,000	0,011	0,011	0,000	0,005	20
6	0,000	0,010	0,010	0,010	0,000	0,010	0,010	0,010	0,000	0,000	0,005	20
7	0,010	0,020	0,030	0,020	0,020	0,010	0,020	0,000	0,020	0,050 *	0,017	20
8	0,000	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,010	0,003	20
9	0,000	0,010	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,003	20
10	0,000	0,000	0,010	0,010	0,000	0,000	0,000	0,010	0,010	0,000	0,004	20
11	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,000	0,000	0,002	20
12	0,010 *	0,010	0,000	0,000	0,010	0,000	0,000	0,010	0,000	0,010	0,005	20
13	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,000	0,000	0,010	0,003	20
14	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	20
15	0,030 *	0,010	0,010	0,000	0,010	0,000	0,010	0,010	**	0,020	0,010	18
16	0,000	0,010	0,031	0,032 *	0,031 *	0,010	0,010	0,010	0,021	0,032 *	0,016	20
17	0,040 *	0,990 *	0,010	0,000	0,010	0,010	0,000	0,000	0,000	0,020	0,222	20
Sr	0,010	0,170	0,009	0,008	0,007	0,006	0,006	0,005	0,007	0,012		338
NE	34	34	34	34	34	34	34	34	32	34		
L	0,000	0,024	0,035	0,021	0,020	0,023	0,022	0,021	0,025	0,024		

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	4,800	4,780	4,730	4,720	4,695	4,860	4,815	4,775	4,745	4,710
2	4,820	5,020	4,630	4,605	4,710	4,740	4,665	4,885	4,960	4,560
3	4,870	4,800	4,740	4,685	4,640	4,955	4,855	4,775	4,715	4,665
4	4,940	5,160	4,795	4,760	4,850	4,865	4,810	5,015	5,085	4,710
5	5,063	5,284	4,884	4,848	4,947	4,984	4,916	5,132	5,195	4,842
6	4,860	5,065	4,685	4,645	4,750	4,775	4,715	4,925	4,990	4,640
7	4,565	4,740	4,425 *	4,400	4,480	4,505	4,450	4,630	4,690	4,365 *
8	4,880	5,110	4,690	4,650	4,760	4,795	4,720	4,950	5,030	4,615
9	4,820	5,015	4,650	4,620	4,710	4,740	4,675	4,880	4,940	4,610
10	4,820	5,010	4,665	4,625	4,710	4,740	4,680	4,885	4,945	4,620
11	4,870	5,080	4,670	4,640	4,745	4,770	4,700	4,940	5,010	4,640
12	4,995	5,195	4,820	4,790	4,885	4,910	4,850	5,055	5,120	4,785
13	4,960	5,180	4,780	4,745	4,840	4,880	4,810	5,030	5,100	4,735
14	4,840	5,040	4,680	4,640	4,730	4,760	4,700	4,900	4,970	4,630
15	5,055	5,315	4,855	4,810	4,925	4,980	4,885	5,155	5,230	4,800
16	4,841	5,056	4,689	4,657	4,752	4,783	4,720	4,930	4,988	4,636
17	4,750	4,505	4,625	4,590	4,685	4,705	4,660	4,870	4,940	4,610
M	4,868	5,021	4,724	4,672	4,754	4,809	4,743	4,925	4,980	4,676
REF.	4,873	5,034	4,722	4,679	4,758	4,817	4,751	4,929	4,985	4,673
SD	0,118	0,209	0,080	0,104	0,113	0,119	0,112	0,131	0,152	0,080

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	4;12 15;17	17		16	16					7;16
Outlier Grubbs			7							7
sr	0,000	0,006	0,008	0,006	0,005	0,006	0,006	0,005	0,007	0,006
SR	0,074	0,166	0,081	0,107	0,116	0,119	0,112	0,131	0,143	0,082

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	- 0,073	- 0,254	+ 0,008	+ 0,041	- 0,063	+ 0,043	+ 0,064	- 0,154	- 0,240	+ 0,037	- 0,059	0,120	1,56
2	- 0,053	- 0,014	- 0,092	- 0,074	- 0,048	- 0,077	- 0,086	- 0,044	- 0,025	- 0,113	- 0,062	0,031	6,36
3	- 0,003	- 0,234	+ 0,018	+ 0,006	- 0,118	+ 0,138	+ 0,104	- 0,154	- 0,270	- 0,008	- 0,052	0,137	1,20
4	+ 0,067	+ 0,126	+ 0,073	+ 0,081	+ 0,092	+ 0,048	+ 0,059	+ 0,086	+ 0,100	+ 0,037	+ 0,077	0,026	9,35
5	+ 0,190	+ 0,250	+ 0,162	+ 0,169	+ 0,189	+ 0,167	+ 0,165	+ 0,203	+ 0,210	+ 0,169	+ 0,187	0,028	21,43
6	- 0,013	+ 0,031	- 0,037	- 0,034	- 0,008	- 0,042	- 0,036	- 0,004	+ 0,005	- 0,033	- 0,017	0,023	2,29
7	- 0,308	- 0,294	- 0,297	- 0,279	- 0,278	- 0,312	- 0,301	- 0,299	- 0,295	- 0,308	- 0,297	0,011	82,60
8	+ 0,007	+ 0,076	- 0,032	- 0,029	+ 0,002	- 0,022	- 0,031	+ 0,021	+ 0,045	- 0,058	- 0,002	0,041	0,16
9	- 0,053	- 0,019	- 0,072	- 0,059	- 0,048	- 0,077	- 0,076	- 0,049	- 0,045	- 0,063	- 0,056	0,017	10,13
10	- 0,053	- 0,024	- 0,057	- 0,054	- 0,048	- 0,077	- 0,071	- 0,044	- 0,040	- 0,053	- 0,052	0,015	10,99
11	- 0,003	+ 0,046	- 0,052	- 0,039	- 0,013	- 0,047	- 0,051	+ 0,011	+ 0,025	- 0,033	- 0,015	0,035	1,42
12	+ 0,122	+ 0,161	+ 0,098	+ 0,111	+ 0,127	+ 0,093	+ 0,099	+ 0,126	+ 0,135	+ 0,112	+ 0,119	0,020	18,36
13	+ 0,087	+ 0,146	+ 0,058	+ 0,066	+ 0,082	+ 0,063	+ 0,059	+ 0,101	+ 0,115	+ 0,062	+ 0,084	0,029	9,12
14	- 0,033	+ 0,006	- 0,042	- 0,039	- 0,028	- 0,057	- 0,051	- 0,029	- 0,015	- 0,043	- 0,033	0,018	5,72
15	+ 0,182	+ 0,281	+ 0,133	+ 0,131	+ 0,167	+ 0,163	+ 0,134	+ 0,226	+ 0,245	+ 0,127	+ 0,179	0,054	10,43
16	- 0,032	+ 0,022	- 0,033	- 0,022	- 0,007	- 0,034	- 0,031	+ 0,001	+ 0,003	- 0,037	- 0,017	0,020	2,64
17	- 0,123	- 0,529	- 0,097	- 0,089	- 0,073	- 0,112	- 0,091	- 0,059	- 0,045	- 0,063	- 0,128	0,143	2,83
d	- 0,005	- 0,013	+ 0,002	- 0,006	- 0,005	- 0,008	- 0,008	- 0,004	- 0,005	+ 0,003	- 0,009	0,127	
Sd	0,118	0,209	0,080	0,104	0,113	0,119	0,112	0,131	0,152	0,080	0,127		

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	-0,62	-1,22	+0,10	+0,40	-0,56	+0,36	+0,57	-1,18	-1,57	+0,46
2	-0,45	-0,07	-1,14	-0,71	-0,43	-0,65	-0,76	-0,34	-0,16	-1,41
3	-0,02	-1,12	+0,23	+0,06	-1,05	+1,16	+0,93	-1,18	-1,77	-0,10
4	+0,57	+0,60	+0,91	+0,78	+0,81	+0,40	+0,53	+0,66	+0,66	+0,46
5	+1,62	+1,20	+2,02	+1,62	+1,67	+1,41	+1,47	+1,55	+1,38	+2,11
6	-0,11	+0,15	-0,46	-0,33	-0,08	-0,35	-0,32	-0,03	+0,04	-0,41
7	-2,61	-1,41	-3,69	-2,68	-2,47	-2,63	-2,68	-2,29	-1,93	-3,85
8	+0,06	+0,36	-0,40	-0,28	+0,01	-0,19	-0,27	+0,16	+0,30	-0,72
9	-0,45	-0,09	-0,89	-0,57	-0,43	-0,65	-0,67	-0,37	-0,29	-0,79
10	-0,45	-0,12	-0,71	-0,52	-0,43	-0,65	-0,63	-0,34	-0,26	-0,66
11	-0,02	+0,22	-0,65	-0,37	-0,12	-0,40	-0,45	+0,08	+0,17	-0,41
12	+1,04	+0,77	+1,22	+1,07	+1,12	+0,78	+0,89	+0,96	+0,89	+1,40
13	+0,74	+0,70	+0,72	+0,64	+0,72	+0,53	+0,53	+0,77	+0,76	+0,78
14	-0,28	+0,03	-0,52	-0,37	-0,25	-0,48	-0,45	-0,22	-0,10	-0,54
15	+1,55	+1,35	+1,66	+1,26	+1,48	+1,37	+1,20	+1,73	+1,61	+1,59
16	-0,27	+0,10	-0,42	-0,21	-0,06	-0,29	-0,27	+0,01	+0,02	-0,46
17	-1,04	-2,53	-1,21	-0,85	-0,65	-0,94	-0,81	-0,45	-0,29	-0,79

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

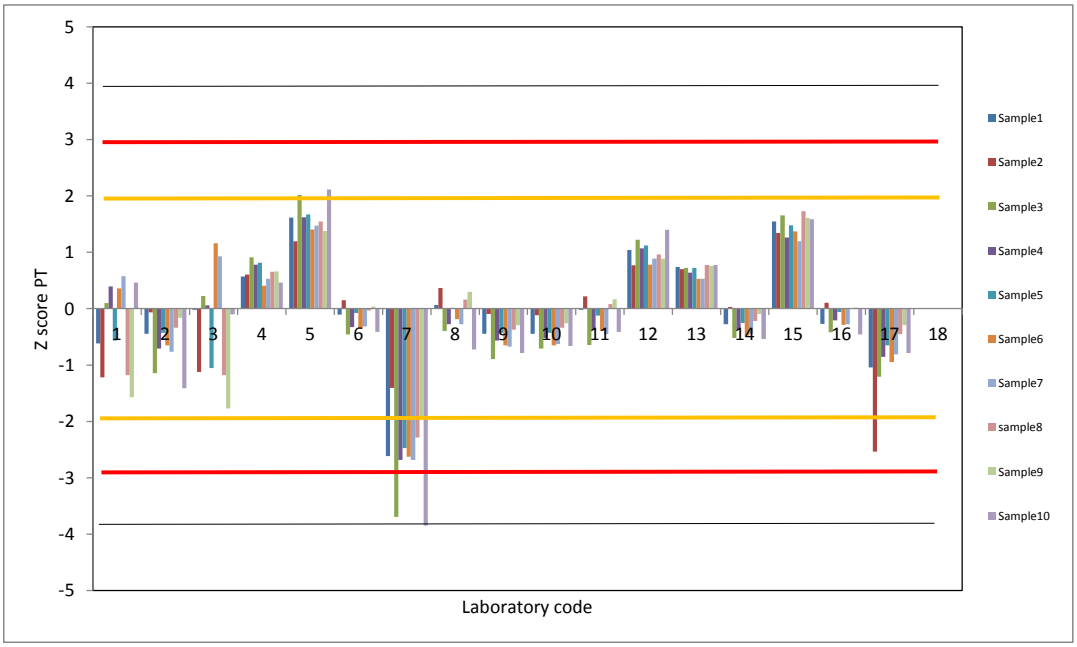


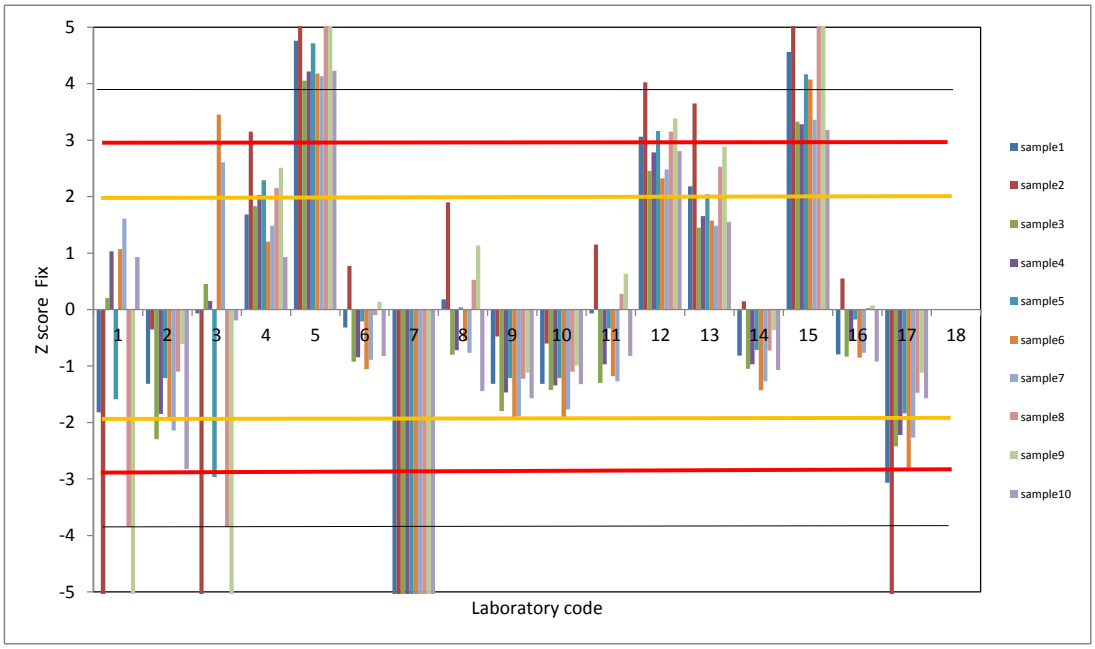
Table VII : **Zscore of the different laboratories for each sample.**
ZS calculated on the standard deviation of reproducibility of the method

Sample/Lab code	1	2	3	4	5	6	7	8	9	10
1	-1,82	-6,35	+0,20	+1,03	-1,59	+1,07	+1,61	-3,85	-5,99	+0,93
2	-1,32	-0,35	-2,30	-1,84	-1,21	-1,93	-2,14	-1,10	-0,61	-2,82
3	-0,07	-5,85	+0,45	+0,16	-2,96	+3,45	+2,61	-3,85	-6,74	-0,20
4	+1,68	+3,15	+1,83	+2,03	+2,29	+1,20	+1,48	+2,15	+2,51	+0,93
5	+4,76	+6,25	+4,05	+4,22	+4,71	+4,17	+4,13	+5,06	+5,25	+4,23
6	-0,32	+0,77	-0,92	-0,84	-0,21	-1,05	-0,89	-0,10	+0,14	-0,82
7	-7,69	-7,35	-7,42	-6,97	-6,96	-7,80	-7,52	-7,47	-7,36	-7,70
8	+0,18	+1,90	-0,80	-0,72	+0,04	-0,55	-0,77	+0,53	+1,14	-1,45
9	-1,32	-0,48	-1,80	-1,47	-1,21	-1,93	-1,89	-1,22	-1,11	-1,57
10	-1,32	-0,60	-1,42	-1,34	-1,21	-1,93	-1,77	-1,10	-0,99	-1,32
11	-0,07	+1,15	-1,30	-0,97	-0,34	-1,18	-1,27	+0,28	+0,64	-0,82
12	+3,06	+4,02	+2,45	+2,78	+3,16	+2,32	+2,48	+3,15	+3,39	+2,80
13	+2,18	+3,65	+1,45	+1,66	+2,04	+1,57	+1,48	+2,53	+2,89	+1,55
14	-0,82	+0,15	-1,05	-0,97	-0,71	-1,43	-1,27	-0,72	-0,36	-1,07
15	+4,56	+7,02	+3,33	+3,28	+4,16	+4,07	+3,36	+5,65	+6,14	+3,18
16	-0,79	+0,55	-0,83	-0,54	-0,17	-0,85	-0,77	+0,03	+0,07	-0,92
17	-3,07	-13,23	-2,42	-2,22	-1,84	-2,80	-2,27	-1,47	-1,11	-1,57

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



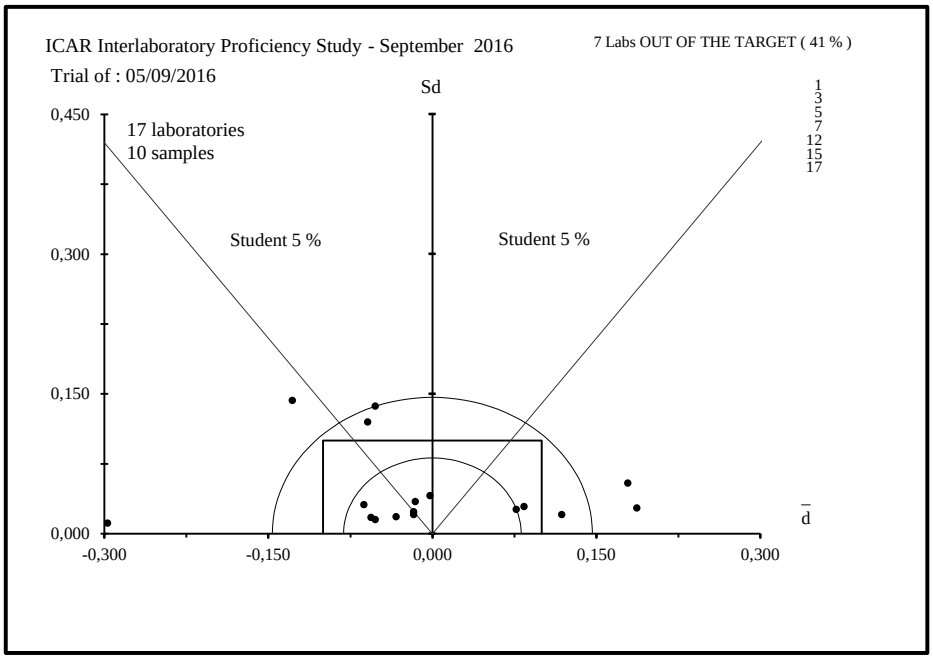


Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).

LIST OF THE PARTICIPANTS ICAR
ICAR PROFICIENCY TEST
RAW MILK
LACTOSE CONTENT Infrared method
´September 2016

Name	City	Country
AIA-LAB. STANDARD LATTE	MACCARESE (ROMA)	ITALY
CATTLE INFORMATION SERVICE	TELFORD	UNITED KINGDOM
CENTRAL MILK LABORATORY - ICBA	CAESAREA	ISRAEL
DAIRY CATTLE RESEARCH	SHANDONG	CHINA
DELTAMUNE	PRETORIA	SOUTH AFRICA
EASTERN LAB SERVICES	MEDINA	USA
FED.LATTERIE SOCIALI DI BOLZANO	BOLZANO	ITALY
LAB PRO ROZBOR MLÉKA	BUSTEHRAD	CHECH REPUBLIC
LAB. OCENY MLEKA KCHZ	PARZNIEWIE	POLAND
LACTOLAB	IRENE	SOUTH AFRICA
MERIEUX NUTRISCIENCE	MIDRAND	SOUTH AFRICA
MILCHPRUFRING BADEN-WURTTEMBERG E.V.	KIRCHEIM / TECK	GERMANY
PLEMENÁRSKE SLUZBY SR S.P.	ZILINA	SLOVAKIA
SUISSE LAB AG	ZOLLIKOFEN	SWITZERLAND
UNIV. NOVI SAD LAB. POLJOPRIVREDNI	NOVI SAD	SERBIA
UNIV. OF ZAGREB	ZAGREB	CROATIA
VETLAB AGRICULTURAL SHOWGROUNDS	LUSAKA	ZAMBIA

ICAR
PROFICIENCY TESTING SCHEME

September 2016

Raw Milk

Determination of UREA CONTENT
Routine method

Sending date of statistical treatment : 29th september 2016

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

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

Nb	%	N°	d	Sd	D
1	6	13	+ 0,33	0,83	0,89
2	13	10	- 0,27	0,92	0,96
3	19	5	- 0,35	1,32	1,37
4	25	4	- 1,32	0,43	1,39
5	31	16	+ 1,55	0,32	1,58
6	38	8	+ 1,14	1,36	1,78
7	44	3	- 0,99	1,56	1,85
8	50	12	+ 1,64	1,42	2,17
9	56	1	- 2,03	0,85	2,20
10	63	11	+ 1,62	1,71	2,35
11	69	14	+ 2,63	0,78	2,75
12	75	2	- 2,12	1,88	2,83
13	81	6	- 4,28	1,44	4,52
14	88	9	+ 4,28	1,99	4,72
15	94	15	- 6,83	1,36	6,96
16	100	7	+ 10,24	4,50	11,18

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 :

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

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 16 sets of results send by 16 laboratories using routine method ISO 9622 Idgf 141, after outlier discarging using Grubbs test at 5 % risk level

(NC : OUT of RANKING because of insufficient data number)
(Nb : laboratory rank; % : relative rank)
(N° : laboratory identification number)
(d et Sd : mean and standard deviation of the differences (laboratory -reference))
(D : Euclidian distance to YX-axis origin = SQUARE ROOT.(d^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}	1,09
Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)	SR_{PT}	3,50

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	0,43	0,00	1,71	0,43	0,43	0,64	0,43	0,64	0,43	0,43	0,49	20
2	0,00	1,00	1,00	1,00	2,00	3,00	2,00	1,00	1,00	0,00	1,05	20
3	0,00	0,00	0,10	0,00	0,10	0,10	0,00	0,10	0,00	0,10	0,05	20
4	0,00	0,00	3,00	0,00	1,00	1,00	0,00	3,00	1,00	1,00	1,05	20
5	1,10	1,20	0,30	1,70	0,90	1,68	1,80	2,90	0,30	0,50	1,03	20
6	4,30	0,70	1,80	0,30	4,20	1,00	1,00	2,30	0,00	1,20	1,56	20
7	5,20	4,50	1,80	1,40	1,40	2,30	0,30	0,40	0,90	0,30	1,75	20
8	3,10	2,70	1,70	0,20	0,80	3,40	5,70 *	7,20 *	1,50	0,90	2,44	20
9	2,00	3,00	3,00	2,00	2,00	4,00	3,00	2,00	3,00	0,00	1,84	20
10	0,40	0,80	0,20	0,20	1,10	0,30	0,70	0,30	0,00	0,10	0,37	20
11	2,08	**	2,08	2,08	0,00	0,00	2,08	2,08	2,08	2,08	1,30	18
12	1,10	3,00	1,30	0,10	1,90	1,20	0,90	0,80	3,20	0,10	1,20	20
13	0,30	0,90	0,30	0,30	1,40	0,30	0,20	0,50	0,10	0,10	0,42	20
14	0,40	0,70	0,00	0,00	1,60	0,90	0,60	0,60	0,00	1,20	0,56	20
15	0,50	1,60	0,20	1,20	0,20	0,50	0,00	1,40	1,60	0,60	0,69	20
16	0,40	0,70	0,80	0,40	0,60	0,40	1,00	0,30	0,10	0,20	0,39	20
Sr	1,45	1,33	1,09	0,71	1,12	1,25	1,33	1,66	0,99	0,56		318
NE	32	30	32	32	32	32	32	32	32	32		
L	5,50	5,02	4,16	2,71	4,25	4,75	3,40	4,12	3,77	2,12		

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**

Sample Lab code	1	2	3	4	5	6	7	8	9	10
1	65,06	58,21	54,14	49,86	47,08	41,41	37,66	31,99	26,96	23,54
2	61,00	57,50	52,50	49,50	45,00	41,50	38,00	33,50	30,50	26,00
3	66,80	58,80	56,65	52,00	43,05	43,05	37,70	34,55	29,90	23,75
4	65,00	60,00	55,50	51,00	46,50	42,50	38,00	32,50	28,50	23,50
5	64,05	59,10	54,45	51,75	47,15	43,24	39,90	36,35	30,45	26,25
6	63,05	58,75	52,20	47,85	44,60	38,40	35,80	30,25	23,40	19,10
7	81,00 *	75,05 *	68,80	64,20 *	64,20 *	52,95	47,45	42,20	33,85	28,85
8	68,55	61,75	55,95	53,50	49,80	44,00	38,35	37,80	30,95	26,95
9	72,00	68,50	61,50	57,00	53,00	49,00	42,50	36,00	31,50	28,00
10	65,70	61,60	57,20	53,40	46,95	43,25	39,55	33,95	28,10	23,75
11	67,48	64,37	61,26	55,03	49,83	45,68	38,41	34,26	30,11	25,96
12	68,95	63,80	58,75	55,35	49,55	44,20	40,95	37,10	28,70	25,25
13	64,45	60,45	56,45	52,35	48,70	44,75	40,30	35,25	30,25	26,55
14	67,90	64,85	59,10	54,80	51,00	47,45	41,30	36,10	32,30	27,70
15	57,65	55,60	48,00	46,40	39,70	36,85	33,10	30,10	23,30	17,20
16	67,10	62,85	58,40	53,90	49,10	45,90	40,80	36,25	31,15	26,20
M	65,65	61,08	56,93	52,25	47,40	44,01	39,36	34,88	29,37	24,91
REF.	65,78	60,93	56,72	52,33	47,56	43,89	39,23	34,72	29,68	25,35
SD	3,47	3,37	4,69	2,94	3,36	3,86	3,14	3,01	2,88	3,11

M = mean per sample
SD = standard deviation per sample
REF. = reference values
*: 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 16 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							8	8		
Outlier Grubbs	7	7		7	7					
sr	1,15	1,09	1,09	0,69	1,13	1,25	0,90	1,10	0,99	0,56
SR	3,57	3,45	4,75	2,98	3,45	3,96	3,30	3,10	2,96	3,14

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	- 0,72	- 2,72	- 2,58	- 2,46	- 0,48	- 2,48	- 1,57	- 2,73	- 2,71	- 1,81	- 2,03	0,85	7,55
2	- 4,78	- 3,43	- 4,22	- 2,83	- 2,56	- 2,39	- 1,23	- 1,22	+ 0,82	+ 0,65	- 2,12	1,88	3,56
3	+ 1,02	- 2,13	- 0,07	- 0,33	- 4,51	- 0,84	- 1,53	- 0,17	+ 0,22	- 1,60	- 0,99	1,56	2,02
4	- 0,78	- 0,93	- 1,22	- 1,33	- 1,06	- 1,39	- 1,23	- 2,22	- 1,18	- 1,85	- 1,32	0,43	9,72
5	- 1,73	- 1,83	- 2,27	- 0,58	- 0,41	- 0,65	+ 0,67	+ 1,63	+ 0,77	+ 0,90	- 0,35	1,32	0,84
6	- 2,73	- 2,18	- 4,52	- 4,48	- 2,96	- 5,49	- 3,43	- 4,47	- 6,28	- 6,25	- 4,28	1,44	9,37
7	+ 15,22	+ 14,12	+ 12,08	+ 11,87	+ 16,64	+ 9,06	+ 8,22	+ 7,48	+ 4,17	+ 3,50	+ 10,24	4,50	7,19
8	+ 2,77	+ 0,82	- 0,77	+ 1,17	+ 2,24	+ 0,11	- 0,88	+ 3,08	+ 1,27	+ 1,60	+ 1,14	1,36	2,65
9	+ 6,22	+ 7,57	+ 4,78	+ 4,67	+ 5,44	+ 5,11	+ 3,27	+ 1,28	+ 1,82	+ 2,65	+ 4,28	1,99	6,79
10	- 0,08	+ 0,67	+ 0,48	+ 1,07	- 0,61	- 0,64	+ 0,32	- 0,77	- 1,58	- 1,60	- 0,27	0,92	0,94
11	+ 1,70	+ 3,44	+ 4,54	+ 2,70	+ 2,27	+ 1,79	- 0,82	- 0,46	+ 0,43	+ 0,61	+ 1,62	1,71	3,01
12	+ 3,17	+ 2,87	+ 2,03	+ 3,02	+ 1,99	+ 0,31	+ 1,72	+ 2,38	- 0,98	- 0,10	+ 1,64	1,42	3,64
13	- 1,33	- 0,48	- 0,27	+ 0,02	+ 1,14	+ 0,86	+ 1,07	+ 0,53	+ 0,57	+ 1,20	+ 0,33	0,83	1,26
14	+ 2,12	+ 3,92	+ 2,38	+ 2,47	+ 3,44	+ 3,56	+ 2,07	+ 1,38	+ 2,62	+ 2,35	+ 2,63	0,78	10,65
15	- 8,13	- 5,33	- 8,72	- 5,93	- 7,86	- 7,04	- 6,13	- 4,62	- 6,38	- 8,15	- 6,83	1,36	15,83
16	+ 1,32	+ 1,92	+ 1,68	+ 1,57	+ 1,54	+ 2,01	+ 1,57	+ 1,53	+ 1,47	+ 0,85	+ 1,55	0,32	15,31
d	- 0,13	+ 0,15	+ 0,21	- 0,08	- 0,16	+ 0,12	+ 0,13	+ 0,16	- 0,31	- 0,44	+ 0,33	4,01	
Sd	3,47	3,37	4,69	2,94	3,36	3,86	3,14	3,01	2,88	3,11	3,42		

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	-0,21	-0,81	-0,55	-0,84	-0,14	-0,64	-0,50	-0,91	-0,94	-0,58
2	-1,38	-1,02	-0,90	-0,96	-0,76	-0,62	-0,39	-0,41	+0,29	+0,21
3	+0,29	-0,63	-0,01	-0,11	-1,34	-0,22	-0,49	-0,06	+0,08	-0,51
4	-0,22	-0,27	-0,26	-0,45	-0,32	-0,36	-0,39	-0,74	-0,41	-0,59
5	-0,50	-0,54	-0,48	-0,20	-0,12	-0,17	+0,21	+0,54	+0,27	+0,29
6	-0,79	-0,65	-0,96	-1,52	-0,88	-1,42	-1,09	-1,49	-2,18	-2,01
7	+4,39	+4,20	+2,58	+4,04	+4,95	+2,35	+2,62	+2,49	+1,45	+1,12
8	+0,80	+0,25	-0,16	+0,40	+0,67	+0,03	-0,28	+1,02	+0,44	+0,51
9	+1,79	+2,25	+1,02	+1,59	+1,62	+1,32	+1,04	+0,42	+0,63	+0,85
10	-0,02	+0,20	+0,10	+0,37	-0,18	-0,17	+0,10	-0,26	-0,55	-0,51
11	+0,49	+1,02	+0,97	+0,92	+0,68	+0,46	-0,26	-0,15	+0,15	+0,19
12	+0,91	+0,85	+0,43	+1,03	+0,59	+0,08	+0,55	+0,79	-0,34	-0,03
13	-0,38	-0,14	-0,06	+0,01	+0,34	+0,22	+0,34	+0,18	+0,20	+0,39
14	+0,61	+1,17	+0,51	+0,84	+1,02	+0,92	+0,66	+0,46	+0,91	+0,75
15	-2,34	-1,58	-1,86	-2,02	-2,34	-1,82	-1,95	-1,54	-2,22	-2,62
16	+0,38	+0,57	+0,36	+0,54	+0,46	+0,52	+0,50	+0,51	+0,51	+0,27

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

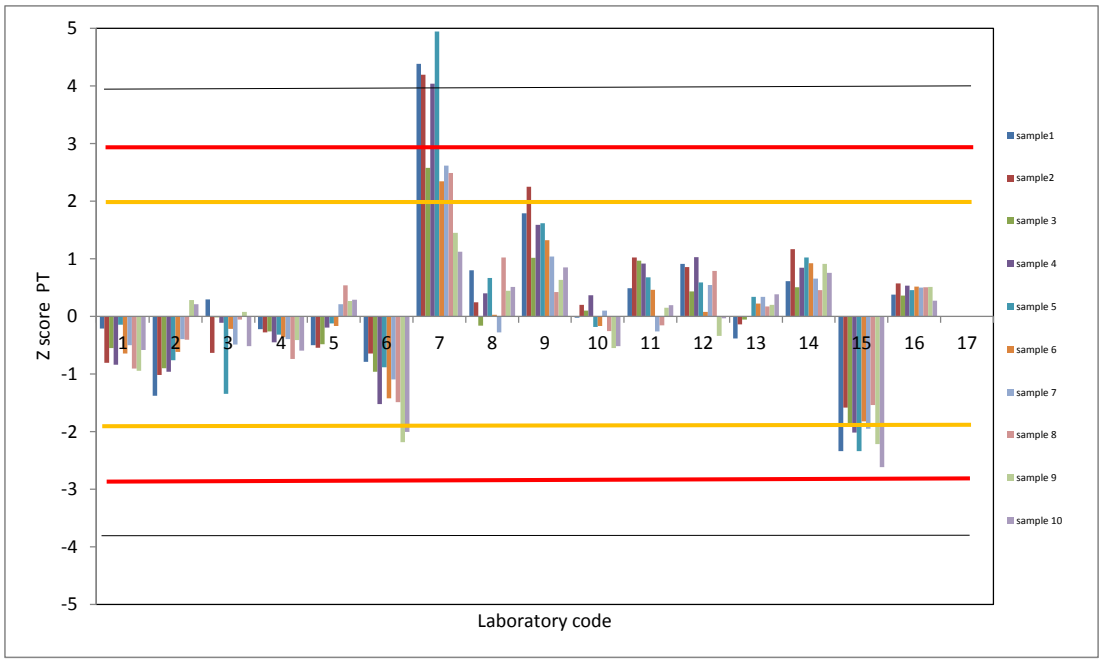


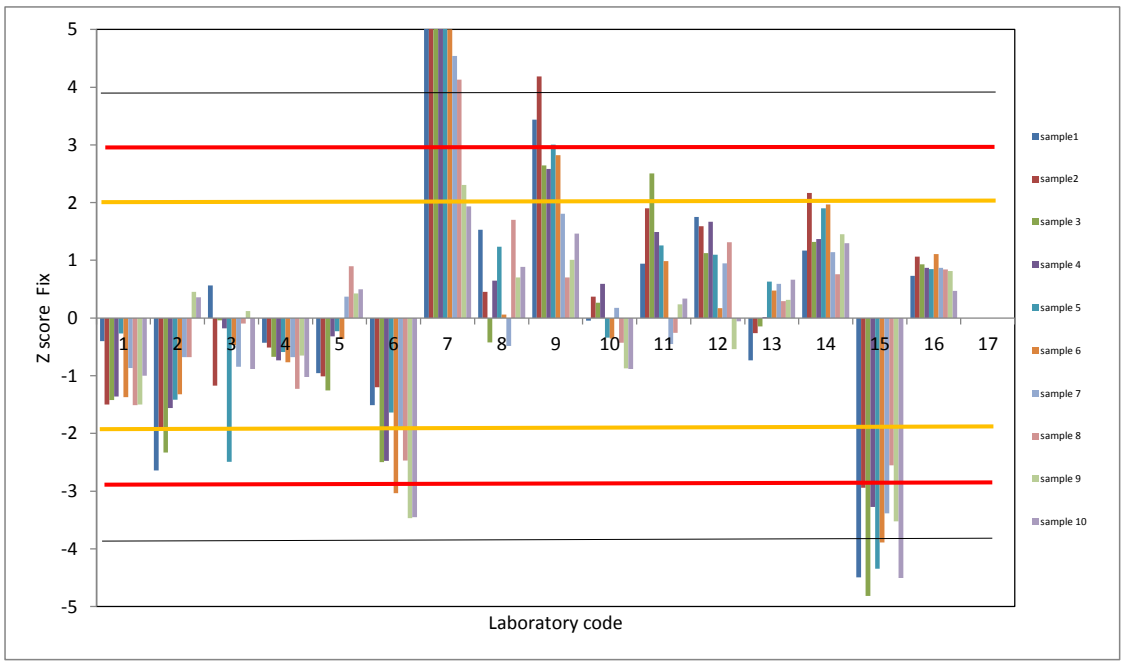
Table VII : **Zscore of the different laboratories for each sample.**
ZS calculated on the standard deviation of reproducibility of the method

Sample/Lab code	1	2	3	4	5	6	7	8	9	10
1	-0,40	-1,50	-1,42	-1,36	-0,27	-1,37	-0,87	-1,51	-1,50	-1,00
2	-2,64	-1,89	-2,33	-1,56	-1,42	-1,32	-0,68	-0,68	+0,45	+0,36
3	+0,56	-1,17	-0,04	-0,18	-2,49	-0,47	-0,85	-0,10	+0,12	-0,88
4	-0,43	-0,51	-0,67	-0,73	-0,59	-0,77	-0,68	-1,23	-0,65	-1,02
5	-0,96	-1,01	-1,25	-0,32	-0,23	-0,36	+0,37	+0,90	+0,43	+0,50
6	-1,51	-1,20	-2,50	-2,47	-1,64	-3,03	-1,90	-2,47	-3,47	-3,45
7	+8,41	+7,80	+6,68	+6,56	+9,19	+5,00	+4,54	+4,13	+2,31	+1,93
8	+1,53	+0,46	-0,42	+0,65	+1,24	+0,06	-0,49	+1,70	+0,70	+0,88
9	+3,44	+4,18	+2,64	+2,58	+3,00	+2,82	+1,81	+0,71	+1,01	+1,46
10	-0,04	+0,37	+0,27	+0,59	-0,34	-0,35	+0,18	-0,43	-0,87	-0,88
11	+0,94	+1,90	+2,51	+1,49	+1,26	+0,99	-0,45	-0,26	+0,24	+0,33
12	+1,75	+1,59	+1,12	+1,67	+1,10	+0,17	+0,95	+1,31	-0,54	-0,06
13	-0,73	-0,26	-0,15	+0,01	+0,63	+0,47	+0,59	+0,29	+0,32	+0,66
14	+1,17	+2,17	+1,32	+1,37	+1,90	+1,97	+1,14	+0,76	+1,45	+1,30
15	-4,49	-2,94	-4,82	-3,27	-4,34	-3,89	-3,39	-2,55	-3,52	-4,50
16	+0,73	+1,06	+0,93	+0,87	+0,85	+1,11	+0,87	+0,84	+0,81	+0,47

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

In yellow the values bigger or smaller than 2/-2 In red the values bigger or smaller than 3/-3

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



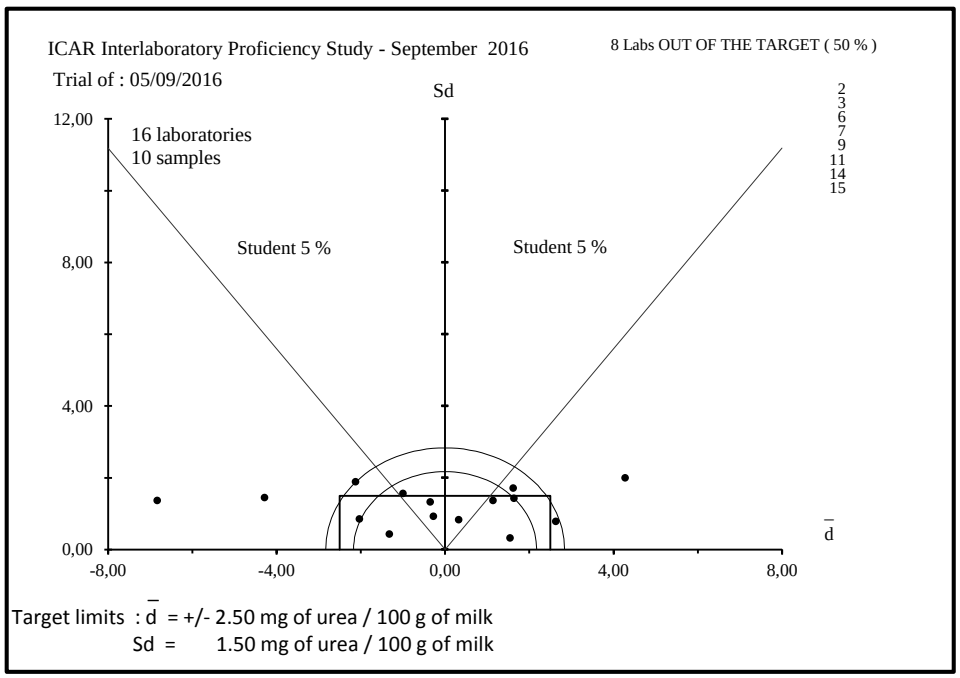


Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).

LIST OF THE PARTICIPANTS ICAR
ICAR PROFICIENCY TEST
RAW MILK
Urea IR
September

Name	City	Country
CATTLE INFORMATION SERVICE	TELFORD	UNITED KINGDOM
CENTRAL MILK LABORATORY - ICBA	CAESAREA	ISRAEL
DELTAMUNE	PRETORIA	SOUTH AFRICA
EASTERN LAB SERVICES	MEDINA	USA
EUROFINS	VEJEN	DENMARK
FED.LATTERIE SOCIALI DI BOLZANO	BOLZANO	ITALY
LAB PRO ROZBOR MLÉKA	BUSTEHRAD	CHECH REPUBLIC
LAB. OCENY MLEKA KCHZ	PARZNIEWIE	POLAND
LAB. POLJOPRIVREDNI	NOVI SAD	SERBIA
LACTOLAB	IRENE	SOUTH AFRICA
MERIEUX NUTRISCIENCE	CAPE TOWN	SOUTH AFRICA
MERIEUX NUTRISCIENCE	MIDRAND	SOUTH AFRICA
MILCHPRUFRING BADEN-WURTTEMBERG E.V.	KIRCHEIM / TECK	GERMANY
OSUUSKUNTA SATAMAITO	PORI	FINLAND
PLEMENÁRSKE SLUZBY SR S.P.	ZILINA	SLOVAKIA
SUISSE LAB AG	ZOLLIKOFEN	SWITZERLAND