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

ICAR - PROFICIENCY TESTING SCHEME CHEMICAL "ROUTINE" METHODS

RAW COW MILK

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

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International Committee for Animal Recording

ICAR Proficiency Test (PT) Chemical "Infrared Method" March 2016

1. Introduction

Dear Participant,

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

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

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

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

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

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

A) In table A you find your participation codes and the information if all the results from the samples received, have been sent to the PT provider.

B) In table B is indicated if the results have been sent on time.

C) In table C is indicated if the results have been reported in the correct unit of measurements. For example in a result excel file it was requested to report the total nitrogen and not the crude protein.

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

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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 ISO 9622 IDF 141 Infrared method. If one or more results have a result out of the limit, the cell is in red. If the repeatability is bigger it will be evaluated internally with the precision of the specific method used. You can find all the detailed information of your data in Table II in the section Statistical elaboration for each parameter.

F) In table F the results of your Z-Score$_{PT}$ (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 G the ranking of your lab will be green if the mean of difference and standard deviation of difference value are in the box of figure 2 of each parameter. Limits are only indicative and so far do not constitute standard values; they indicate what is normally reachable by labs for their self evaluation. According the results obtained the MA SC will decide eventually to revise.

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

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

<u>In the second part of the report</u> 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, the part 4 of this report, completed with the results, reference and/or routine methods, from all the ICAR countries for the parameters indicated.

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

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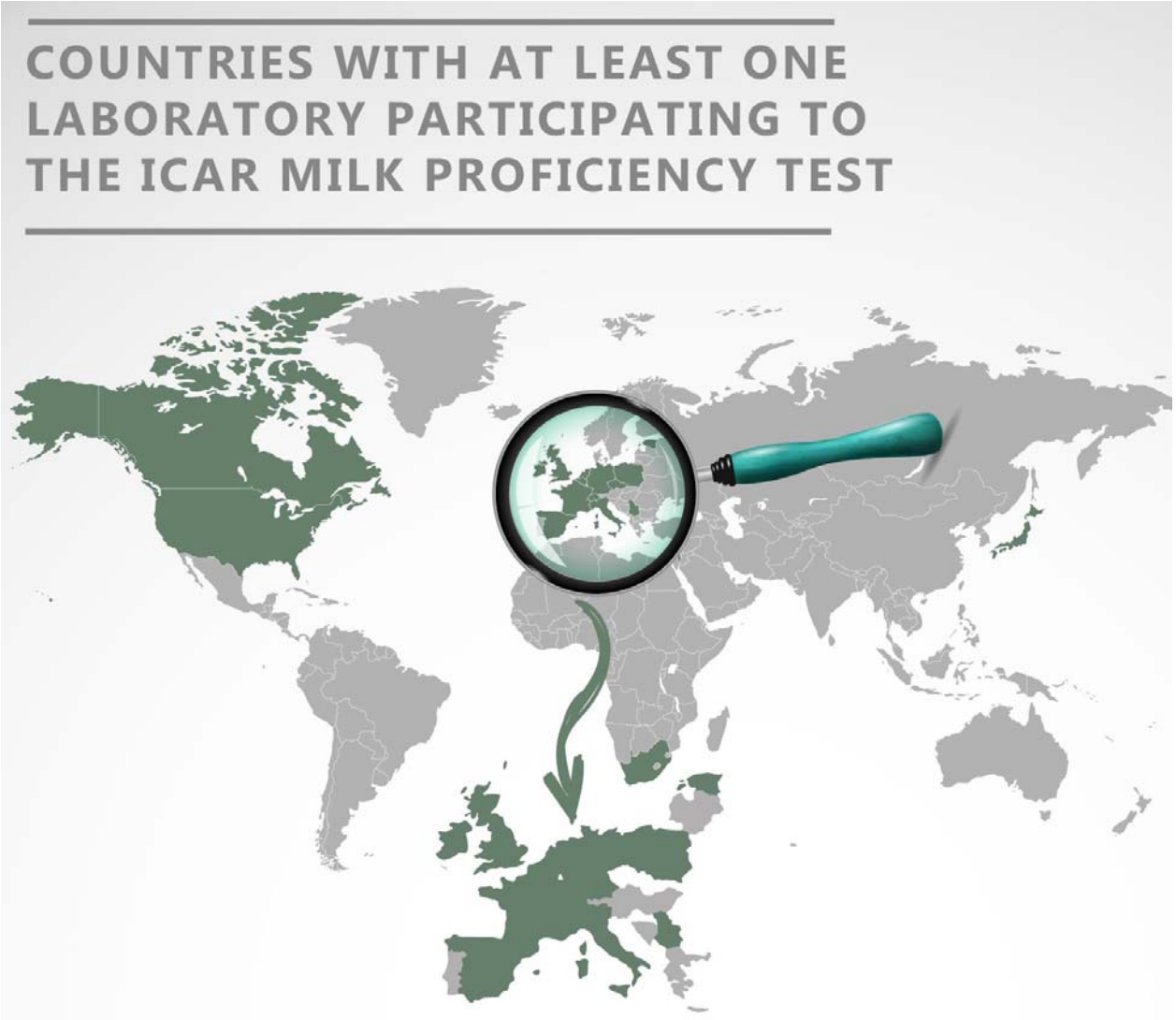
March 2016

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

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



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

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

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

Kind Regards,

ICAR Secretariat

ICAR - International Committee for Animal Recording
Via Savoia 78, sc. A, int.3,
00198 Rome, Italy

2. Your Performance Analysis and Control Charts

International Committee for Animal Recording

ICAR Proficiency Test (PT) "Infrared method" – March 2016

Laboratory Name	Laboratory Name

A – Your participation Codes

	Fat_{IR}	$Protein_{IR}$	$Lactose_{IR}$	$Urea_{IR}$
Subscription	Yes	Yes	Yes	Yes
Participation Codes				
Are all the sample results received ?	Yes	Yes	Yes	Yes

B – Data received on time

Yes	No	Deadline
		21.03.2016

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

Fat_{IR}	$Protein_{IR}$	$Lactose_{IR}$	$Urea_{IR}$
g/100g	g/100g	g/100g	mg/dl

D – Outliers

Fat_{IR}	$Protein_{IR}$	$Lactose_{IR}$	$Urea_{IR}$
g/100g	g/100g	g/100g	mg/dl
sample	sample	sample	sample

E – Repeatability

Your "r" performance

Fat_{IR}	$Protein_{IR}$	$Lactose_{IR}$	$Urea_{IR}$
g/100g	g/100g	g/100g	mg/dl

Limits

Fat_{ref}	$Protein_{ref}$	$Lactose_{ref}$	$Urea_{ref}$
g/100g	g/100g	g/100g	mg/dl
ISO 9622 IDF 141	ISO 9622 IDF 141	ISO 9622 IDF 141	ISO 14637 / IDF 195
0,040 g/100g	0,040 g/100g	0,040 g/100g	1,5 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.

F – Z-Score

Your Z-Score PT

Fat_{IR}	$Protein_{IR}$	$Lactose_{IR}$	$Urea_{IR}$

Your FIX Z-Score

Fat_{IR}	$Protein_{IR}$	$Lactose_{IR}$	$Urea_{IR}$

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

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

G – Ranking of your lab

Mean difference and standard deviation of difference

Fat_{IR}	$Protein_{IR}$	$Lactose_{IR}$	$Urea_{IR}$

Indicative Limits defined in the ICAR MA SC

Fat_{IR}	$Protein_{IR}$	$Lactose_{IR}$	$Urea_{IR}$
g/100g	g/100g	g/100g	mg/dl
ISO 1211 IDF 1D	ISO 8968 / IDF 20	ISO 22662 IDF 198	ISO IDF 195
d=0,02	d=0,025	d=0,10	d=2,5
sd=0,03	sd=0,020	sd=0,10	sd=1,5

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

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

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

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

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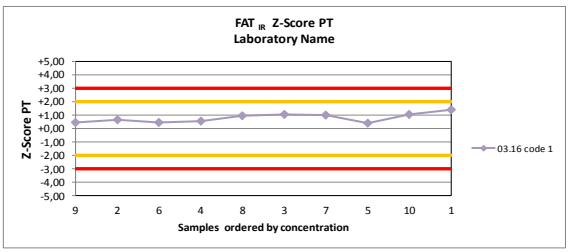
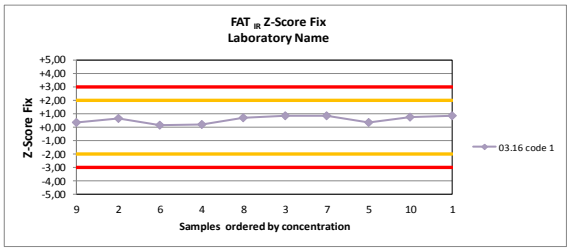
H

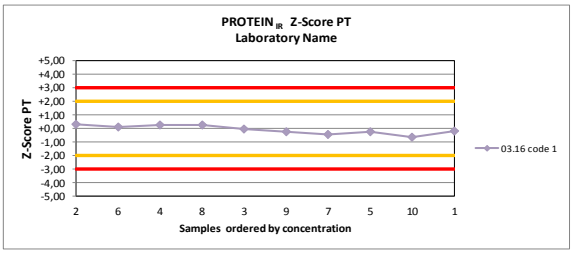
Control Charts

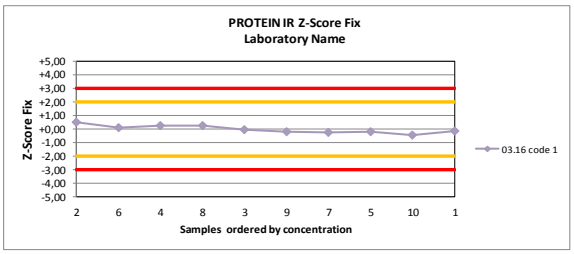
Control Charts Fat





Control Charts Protein





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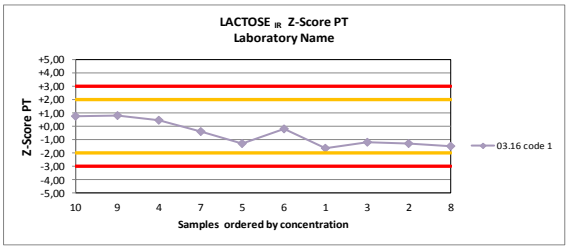
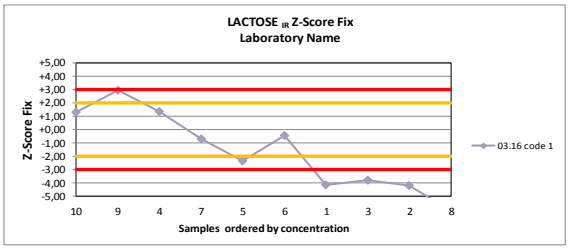
Control Charts Lactose





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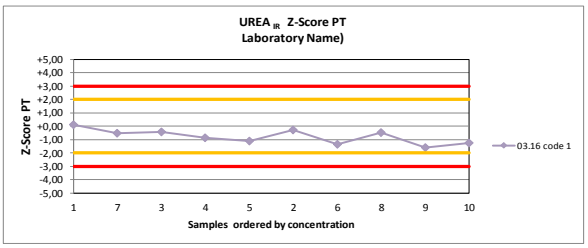
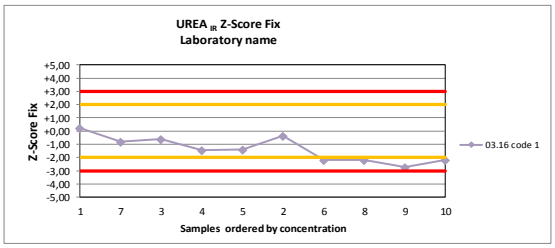
ICAR Proficiency Test (PT) "Infrared method" – March 2016

Control Charts Urea





3. ICAR Statistical elaboration

ICAR
PROFICIENCY TESTING SCHEME

March 2016

Raw Milk

Determination of FAT CONTENT
Routine method

Sending date of statistical treatment : 19th april 2016

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

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

Nb	%	N°	d	Sd	D	Method
1	8	7	- 0,000	0,012	0,012	IR
2	17	12	- 0,006	0,011	0,013	IR
3	25	4	+ 0,007	0,012	0,014	IR
4	33	11	- 0,007	0,018	0,019	IR
5	42	9	- 0,012	0,022	0,025	IR
6	50	1	+ 0,023	0,011	0,026	IR
7	58	6	- 0,021	0,016	0,026	IR
8	67	2	+ 0,017	0,021	0,027	IR
9	75	10	+ 0,025	0,022	0,033	IR
10	83	3	+ 0,030	0,025	0,039	IR
11	92	5	- 0,061	0,026	0,067	IR
12	100	8	- 2,590	1,595	3,042	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 12 sets of results send by 10 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}	0,009
Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)	SR_{PT}	0,029

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	0,020	0,010	0,010	0,010	0,010	0,007	20
2	0,000	0,030 *	0,020	0,010	0,020	0,000	0,000	0,010	0,020	0,000	0,011	20
3	0,000	0,010	0,010	0,000	0,010	0,000	0,000	0,010	0,010	0,000	0,005	20
4	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,000	0,000	0,000	0,002	20
5	0,000	0,000	0,010	0,000	0,000	0,000	0,010	0,000	0,010	0,010	0,004	20
6	0,020	0,000	0,000	0,000	0,010	0,010	0,000	0,010	0,000	0,000	0,006	20
7	0,000	0,000	0,010	0,000	0,000	0,000	0,000	0,000	0,000	0,010	0,003	20
8	0,030	0,121 *	**	**	**	**	**	**	**	**	0,062	4
9	0,010	0,000	0,000	0,000	0,000	0,010	0,020	0,010	0,010	0,020	0,008	20
10	0,010	0,010	**	0,010	0,010	0,010	**	0,000	0,000	**	0,006	14
11	0,020	0,010	0,010	0,010	0,010	0,020	0,020	0,000	0,020	0,020	0,011	20
12	0,010	0,000	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,003	20
Sr	0,009	0,026	0,006	0,005	0,006	0,007	0,007	0,005	0,007	0,007		218
NE	24	24	20	22	22	22	20	22	22	20		
L	0,034	0,016	0,022	0,017	0,021	0,025	0,026	0,017	0,026	0,026		

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	4,765	2,165	3,520	2,835	4,170	2,440	3,855	3,135	1,495	4,525
2	4,740	2,135	3,510	2,835	4,210	2,450	3,850	3,125	1,460	4,530
3	4,750	2,195	3,525	2,860	4,175	2,450	3,860	3,145	1,545	4,470
4	4,720	2,130	3,490	2,825	4,170	2,450	3,840	3,110	1,490	4,520
5	4,610 *	2,080	3,415	2,760 *	4,090	2,410	3,765	3,040	1,435	4,455
6	4,680	2,140	3,460	2,810	4,115	2,425	3,800	3,095	1,470	4,470
7	4,730	2,140	3,495	2,830	4,160	2,420	3,830	3,110	1,450	4,505
8	4,705	2,070								
9	4,725	2,160	3,470	2,810	4,150	2,445	3,770	3,075	1,485	4,460
10	4,755	2,205		2,835	4,175	2,435		3,120	1,520	
11	4,740	2,115	3,475	2,815	4,155	2,420	3,820	3,100	1,440	4,520
12	4,715	2,140	3,480	2,820	4,140	2,420	3,815	3,090	1,500	4,490
M	4,730	2,140	3,484	2,828	4,155	2,433	3,821	3,104	1,481	4,495
REF.	4,731	2,140	3,487	2,826	4,157	2,433	3,821	3,106	1,480	4,495
SD	0,024	0,040	0,032	0,015	0,032	0,015	0,034	0,029	0,034	0,029

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 12 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		2;8								
Outlier Grubbs	5			5						
sr	0,010	0,004	0,006	0,005	0,006	0,007	0,007	0,005	0,007	0,007
SR	0,025	0,037	0,033	0,016	0,032	0,016	0,034	0,030	0,034	0,029

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	+ 0,034	+ 0,025	+ 0,033	+ 0,009	+ 0,013	+ 0,007	+ 0,035	+ 0,029	+ 0,015	+ 0,031	+ 0,023	0,011	6,65
2	+ 0,009	- 0,005	+ 0,023	+ 0,009	+ 0,053	+ 0,017	+ 0,030	+ 0,019	- 0,020	+ 0,035	+ 0,017	0,021	2,61
3	+ 0,019	+ 0,055	+ 0,038	+ 0,034	+ 0,018	+ 0,017	+ 0,039	+ 0,039	+ 0,065	- 0,025	+ 0,030	0,025	3,84
4	- 0,011	- 0,010	+ 0,003	- 0,001	+ 0,013	+ 0,017	+ 0,019	+ 0,004	+ 0,010	+ 0,025	+ 0,007	0,012	1,83
5	- 0,121	- 0,060	- 0,072	- 0,066	- 0,067	- 0,023	- 0,056	- 0,066	- 0,045	- 0,040	- 0,061	0,026	7,56
6	- 0,051	+ 0,000	- 0,027	- 0,016	- 0,042	- 0,008	- 0,021	- 0,011	- 0,010	- 0,025	- 0,021	0,016	4,23
7	- 0,001	+ 0,000	+ 0,008	+ 0,004	+ 0,003	- 0,013	+ 0,010	+ 0,004	- 0,030	+ 0,010	- 0,000	0,012	0,12
8	- 0,026	- 0,070									- 0,048	0,031	2,16
9	- 0,006	+ 0,020	- 0,017	- 0,016	- 0,007	+ 0,012	- 0,051	- 0,031	+ 0,005	- 0,035	- 0,012	0,022	1,79
10	+ 0,024	+ 0,065		+ 0,009	+ 0,018	+ 0,002		+ 0,014	+ 0,040		+ 0,025	0,022	3,00
11	+ 0,009	- 0,025	- 0,012	- 0,011	- 0,002	- 0,013	- 0,000	- 0,006	- 0,040	+ 0,025	- 0,007	0,018	1,32
12	- 0,016	+ 0,000	- 0,007	- 0,006	- 0,017	- 0,013	- 0,006	- 0,016	+ 0,020	- 0,005	- 0,006	0,011	1,86
d	- 0,001	- 0,000	- 0,003	+ 0,001	- 0,001	+ 0,000	- 0,000	- 0,002	+ 0,001	- 0,000	- 0,002	0,031	
Sd	0,024	0,040	0,032	0,015	0,032	0,015	0,034	0,029	0,034	0,029	0,030		

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	+1,41	+0,63	+1,03	+0,58	+0,41	+0,46	+1,03	+0,98	+0,44	+1,05
2	+0,38	-0,12	+0,72	+0,58	+1,66	+1,14	+0,88	+0,64	-0,59	+1,22
3	+0,79	+1,38	+1,19	+2,23	+0,57	+1,14	+1,17	+1,32	+1,91	-0,84
4	-0,44	-0,25	+0,10	-0,08	+0,41	+1,14	+0,58	+0,13	+0,29	+0,88
5	-4,96	-1,50	-2,21	-4,38	-2,07	-1,58	-1,65	-2,26	-1,32	-1,36
6	-2,09	+0,00	-0,82	-1,07	-1,29	-0,56	-0,61	-0,38	-0,29	-0,84
7	-0,03	+0,00	+0,26	+0,25	+0,10	-0,90	+0,28	+0,13	-0,88	+0,36
8	-1,06	-1,76								
9	-0,24	+0,51	-0,51	-1,07	-0,21	+0,80	-1,50	-1,07	+0,15	-1,19
10	+0,99	+1,63		+0,58	+0,57	+0,12		+0,47	+1,18	
11	+0,38	-0,62	-0,36	-0,74	-0,05	-0,90	-0,01	-0,21	-1,17	+0,88
12	-0,65	+0,00	-0,20	-0,41	-0,52	-0,90	-0,16	-0,56	+0,59	-0,16

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

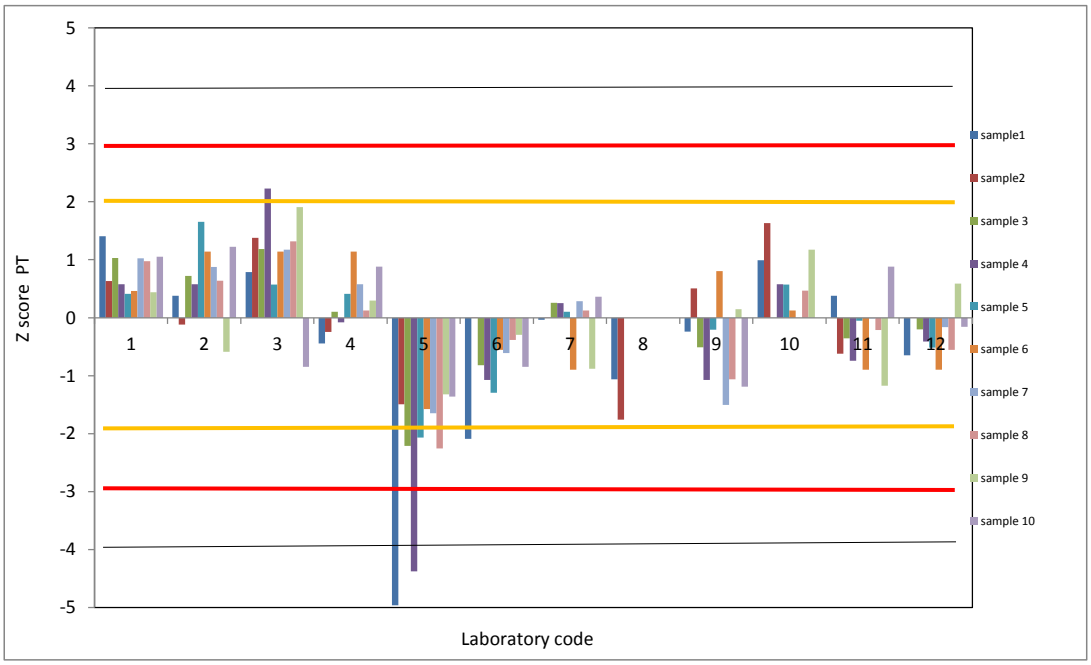

Table VII : **Zscore of the different laboratories for each sample.**
ZS calculated on the standard deviation of reproducibility of the method

Sample / Lab code	1	2	3	4	5	6	7	8	9	10
1	+0,85	+0,63	+0,83	+0,22	+0,33	+0,17	+0,86	+0,72	+0,38	+0,76
2	+0,23	-0,12	+0,58	+0,22	+1,33	+0,42	+0,74	+0,47	-0,50	+0,89
3	+0,48	+1,38	+0,96	+0,84	+0,46	+0,42	+0,99	+0,97	+1,63	-0,61
4	-0,27	-0,25	+0,08	-0,03	+0,33	+0,42	+0,49	+0,09	+0,25	+0,64
5	-3,02	-1,50	-1,79	-1,66	-1,67	-0,58	-1,39	-1,66	-1,12	-0,99
6	-1,27	+0,00	-0,67	-0,41	-1,04	-0,20	-0,51	-0,28	-0,25	-0,61
7	-0,02	+0,00	+0,21	+0,09	+0,08	-0,33	+0,24	+0,09	-0,75	+0,26
8	-0,65	-1,76								
9	-0,15	+0,50	-0,42	-0,41	-0,17	+0,30	-1,26	-0,78	+0,13	-0,86
10	+0,60	+1,63		+0,22	+0,46	+0,05		+0,34	+1,00	
11	+0,23	-0,62	-0,29	-0,28	-0,04	-0,33	-0,01	-0,16	-1,00	+0,64
12	-0,40	+0,00	-0,17	-0,16	-0,42	-0,33	-0,14	-0,41	+0,50	-0,11

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

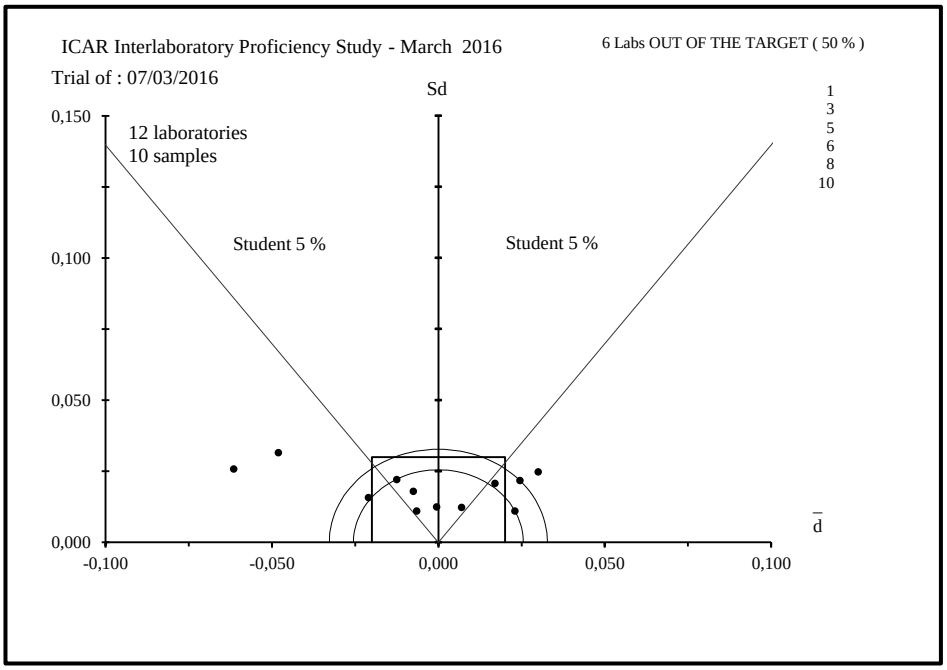

Figure 1 : **ACCURACY - Evaluation of the individual performances (to see table I).**

LIST OF THE PARTICIPANTS ICAR

ICAR PROFICIENCY TEST
RAW MILK
FAT CONTENT Infrared method
March 2016

Name	City	Country
AIA-LAB. STANDARD LATTE	MACARESE (ROMA)	ITALY
ALLATTENYESZTESI TELJESITMENYVIZSGALO KFT	GODOLLO	HUNGARY
CATTLE INFORMATION SERVICE	TELFORD	UNITED KINGDOM
EASTERN LAB SERVICES	MEDINA	USA
FED.LATTERIE SOCIALI DI BOLZANO	BOLZANO	ITALY
LACTOLAB	IRENE	SOUTH AFRICA
LOM KCHZ LABORATORIUM REFERENCYJNE z/s w	PARZNIEWIE	POLAND
SUISSE LAB AG	ZOLLIKOFEN	SWITZERLAND
UNI.NOVOM SADU MLEKA	MLEKA	SERBIA
VALACTA	STE ANNE DE BELLEVUE	CANADA

ICAR
PROFICIENCY TESTING SCHEME

March 2016

Raw Milk

Determination of CRUDE PROTEIN CONTENT
Routine method

Sending date of statistical treatment : 19th april 2016

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

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

Nb	%	N°	d	Sd	D	Method
1	8	1	- 0,000	0,012	0,012	IR
2	17	9	- 0,001	0,014	0,014	IR
3	25	6	- 0,013	0,006	0,014	IR
4	33	12	- 0,001	0,018	0,018	IR
5	42	4	+ 0,005	0,020	0,020	IR
6	50	2	+ 0,009	0,023	0,024	IR
7	58	3	- 0,024	0,014	0,028	IR
8	67	5	- 0,023	0,034	0,041	IR
9	75	11	+ 0,042	0,011	0,044	IR
10	83	7	+ 0,058	0,004	0,058	IR
11	92	10	- 0,086	0,053	0,101	IR
12	100	8	- 0,436	1,207	1,283	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 12 sets of results send by 10 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.

eatability standard deviation of this ICAR proficiency test (after Cochran elimination at 5 %)	Sr_{PT}	0,006
oducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)	SR_{PT}	0,037

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	0,000	0,010	0,000	0,010	0,000	0,000	0,000	0,004	20
2	0,000	0,010	0,000	0,010	0,010	0,010	0,000	0,010	0,000	0,020	0,007	20
3	0,000	0,000	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,010	0,003	20
4	0,010	0,010	0,000	0,010	0,010	0,000	0,010	0,000	0,010	0,000	0,005	20
5	0,000	0,000	0,010	0,000	0,010	0,010	0,000	0,000	0,020	0,000	0,006	20
6	0,010	0,000	0,020	0,000	0,000	0,000	0,010	0,000	0,000	0,000	0,005	20
7	0,010	0,010	0,000	0,000	0,000	0,010	0,010	0,000	0,010	0,000	0,005	20
8	0,009	0,007	0,093 *	0,060 *	** *	0,211 *	** *	0,038 *	0,026	**	0,065	14
9	0,010	0,000	0,010	0,010	0,000	0,000	0,000	0,010	0,010	0,010	0,005	20
10	0,000	0,010	**	0,000	0,000	0,000	**	0,000	0,000	**	0,003	14
11	0,010	0,000	0,000	0,010	0,010	0,010	0,020	0,000	0,020	0,010	0,008	20
12	0,000	0,010	0,010	0,000	0,010	0,000	0,000	0,010	0,000	0,010	0,005	20
Sr	0,005	0,005	0,021	0,013	0,005	0,043	0,007	0,009	0,009	0,006		228
NE	24	24	22	24	22	24	20	24	24	20		
L	0,019	0,017	0,022	0,015	0,018	0,015	0,023	0,013	0,031	0,022		

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	3,713	2,718	3,233	2,888	3,513	2,808	3,413	3,078	3,238	3,598
2	3,690	2,745	3,240	2,905	3,515	2,825	3,420	3,085	3,270	3,600
3	3,710	2,660	3,210	2,850	3,510	2,750	3,405	3,040	3,220	3,605
4	3,695	2,735	3,240	2,905	3,505	2,820	3,415	3,080	3,255	3,600
5	3,740	2,630	3,215	2,820	3,525	2,735	3,420	3,030	3,230	3,630
6	3,705	2,680	3,220	2,870	3,510	2,780	3,415	3,050	3,240	3,600
7	3,775	2,755	3,290	2,940	3,580	2,855	3,475	3,130	3,305	3,680
8	3,712	2,684	3,206	2,850		2,849		3,046	3,230	
9	3,695	2,720	3,235	2,885	3,510	2,810	3,420	3,075	3,245	3,595
10	3,700	2,545		2,780	3,470	2,650 *		2,980	3,200	
11	3,755	2,760	3,270	2,935	3,555	2,835	3,460	3,120	3,290	3,645
12	3,740	2,675	3,235	2,870	3,535	2,770	3,430	3,065	3,240	3,635
M	3,719	2,692	3,236	2,875	3,521	2,803	3,427	3,065	3,247	3,619
REF.	3,718	2,698	3,234	2,877	3,520	2,803	3,423	3,067	3,246	3,616
SD	0,027	0,062	0,025	0,046	0,029	0,040	0,022	0,040	0,029	0,028

M = mean per sample REF. = reference values

SD = standard deviation per sample *: discarded data using the test of Grubbs 5 %

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 12 laboratories 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			8	8		8		8		
Outlier Grubbs						10				
sr	0,005	0,005	0,006	0,004	0,005	0,004	0,007	0,004	0,009	0,006
SR	0,027	0,062	0,025	0,048	0,029	0,039	0,023	0,042	0,030	0,028

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	+ 0,020	- 0,001	+ 0,011	- 0,007	+ 0,005	- 0,010	+ 0,011	- 0,008	- 0,018	- 0,000	0,012	0,06
2	- 0,028	+ 0,047	+ 0,006	+ 0,028	- 0,005	+ 0,022	- 0,003	+ 0,018	+ 0,024	- 0,016	+ 0,009	0,023	1,30
3	- 0,008	- 0,038	- 0,024	- 0,027	- 0,010	- 0,053	- 0,018	- 0,027	- 0,026	- 0,011	- 0,024	0,014	5,49
4	- 0,023	+ 0,037	+ 0,006	+ 0,028	- 0,015	+ 0,017	- 0,008	+ 0,013	+ 0,009	- 0,016	+ 0,005	0,020	0,76
5	+ 0,022	- 0,068	- 0,019	- 0,057	+ 0,005	- 0,068	- 0,003	- 0,037	- 0,016	+ 0,014	- 0,023	0,034	2,14
6	- 0,013	- 0,018	- 0,014	- 0,007	- 0,010	- 0,023	- 0,008	- 0,017	- 0,006	- 0,016	- 0,013	0,006	7,41
7	+ 0,057	+ 0,057	+ 0,056	+ 0,063	+ 0,060	+ 0,052	+ 0,052	+ 0,063	+ 0,059	+ 0,064	+ 0,058	0,004	42,00
8	- 0,006	- 0,014	- 0,028	- 0,027		+ 0,045		- 0,021	- 0,016		- 0,010	0,025	1,00
9	- 0,023	+ 0,022	+ 0,001	+ 0,008	- 0,010	+ 0,007	- 0,003	+ 0,008	- 0,001	- 0,021	- 0,001	0,014	0,28
10	- 0,018	- 0,153		- 0,097	- 0,050	- 0,153		- 0,087	- 0,046		- 0,086	0,053	4,32
11	+ 0,037	+ 0,062	+ 0,036	+ 0,058	+ 0,035	+ 0,032	+ 0,037	+ 0,053	+ 0,044	+ 0,029	+ 0,042	0,011	11,63
12	+ 0,022	- 0,023	+ 0,001	- 0,007	+ 0,015	- 0,033	+ 0,007	- 0,002	- 0,006	+ 0,019	- 0,001	0,018	0,13
d	+ 0,001	- 0,006	+ 0,002	- 0,002	+ 0,001	- 0,000	+ 0,004	- 0,002	+ 0,001	+ 0,003	- 0,001	0,038	
Sd	0,027	0,062	0,025	0,046	0,029	0,040	0,022	0,040	0,029	0,028	0,037		

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	-0,18	+0,32	-0,04	+0,24	-0,24	+0,11	-0,44	+0,28	-0,27	-0,65
2	-1,03	+0,76	+0,23	+0,61	-0,17	+0,54	-0,13	+0,45	+0,82	-0,58
3	-0,29	-0,62	-0,95	-0,59	-0,34	-1,35	-0,80	-0,67	-0,88	-0,40
4	-0,85	+0,60	+0,23	+0,61	-0,52	+0,42	-0,35	+0,33	+0,31	-0,58
5	+0,82	-1,11	-0,76	-1,24	+0,18	-1,73	-0,13	-0,92	-0,54	+0,49
6	-0,48	-0,30	-0,56	-0,15	-0,34	-0,59	-0,35	-0,42	-0,20	-0,58
7	+2,12	+0,92	+2,22	+1,37	+2,11	+1,30	+2,32	+1,57	+2,01	+2,28
8	-0,22	-0,23	-1,10	-0,59		+1,15		-0,52	-0,55	
9	-0,85	+0,35	+0,04	+0,17	-0,34	+0,16	-0,13	+0,20	-0,03	-0,76
10	-0,66	-2,49		-2,11	-1,74	-3,87		-2,16	-1,56	
11	+1,38	+1,00	+1,42	+1,26	+1,23	+0,79	+1,66	+1,32	+1,50	+1,03
12	+0,82	-0,38	+0,04	-0,15	+0,53	-0,84	+0,32	-0,05	-0,20	+0,67

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

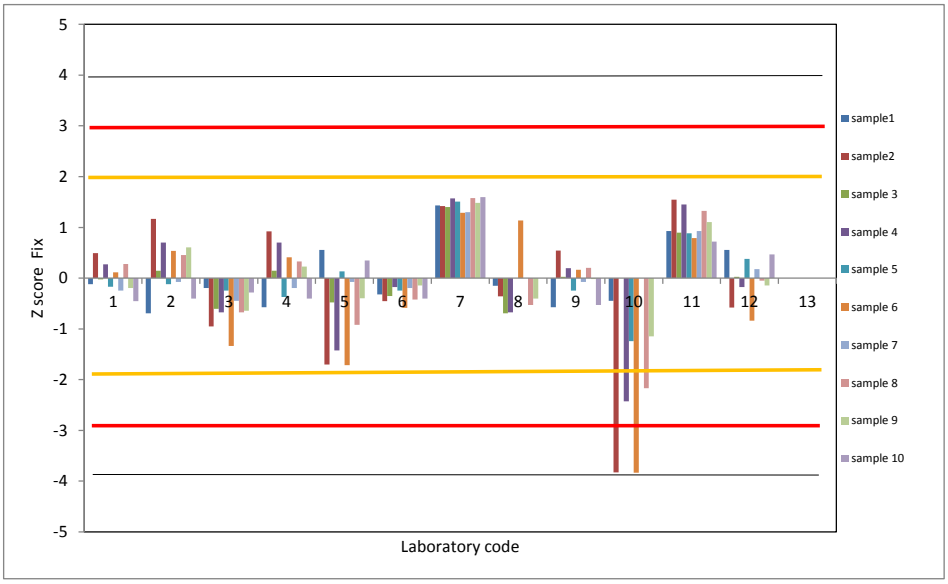
Table VII : **Zscore of the different laboratories for each sample.**
ZS calculated on the standard deviation of reproducibility of the method

Sample / Lab code	1	2	3	4	5	6	7	8	9	10
1	-0,12	+0,50	-0,03	+0,27	-0,17	+0,11	-0,25	+0,28	-0,20	-0,46
2	-0,70	+1,17	+0,15	+0,70	-0,12	+0,54	-0,07	+0,45	+0,60	-0,41
3	-0,20	-0,95	-0,60	-0,68	-0,24	-1,34	-0,45	-0,67	-0,65	-0,28
4	-0,57	+0,92	+0,15	+0,70	-0,37	+0,41	-0,20	+0,33	+0,23	-0,41
5	+0,55	-1,70	-0,48	-1,43	+0,13	-1,71	-0,07	-0,92	-0,40	+0,34
6	-0,32	-0,45	-0,35	-0,18	-0,24	-0,59	-0,20	-0,42	-0,15	-0,41
7	+1,43	+1,42	+1,40	+1,57	+1,51	+1,29	+1,30	+1,58	+1,48	+1,59
8	-0,15	-0,36	-0,70	-0,68		+1,14		-0,53	-0,41	
9	-0,57	+0,55	+0,02	+0,20	-0,24	+0,16	-0,07	+0,20	-0,02	-0,53
10	-0,45	-3,83		-2,43	-1,24	-3,84		-2,17	-1,15	
11	+0,93	+1,55	+0,90	+1,45	+0,88	+0,79	+0,93	+1,33	+1,10	+0,72
12	+0,55	-0,58	+0,02	-0,18	+0,38	-0,84	+0,18	-0,05	-0,15	+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=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



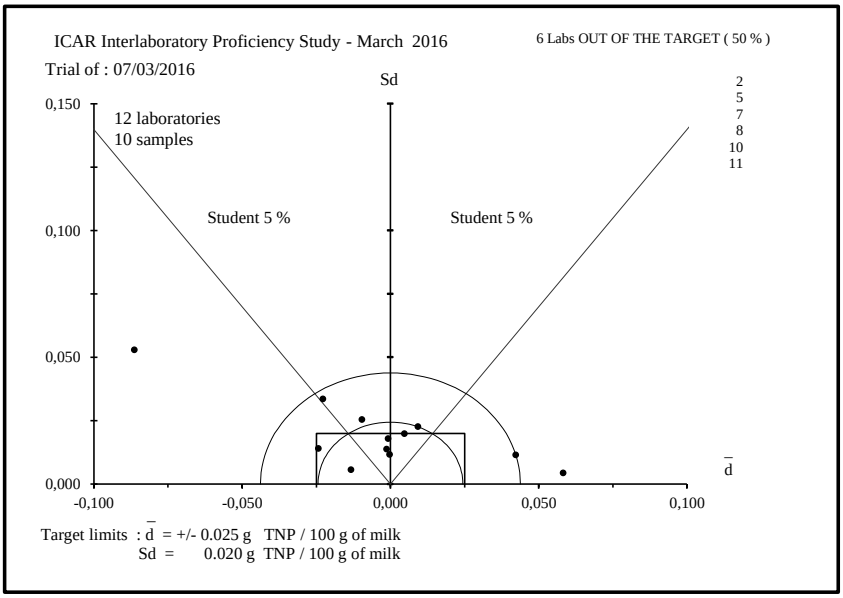


Figure 1 : **ACCURACY - Evaluation of the individual performances (to see table I).**

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EASTERN LAB SERVICES	MEDINA	USA
FED.LATTERIE SOCIALI DI BOLZANO	BOLZANO	ITALY
LACTOLAB	IRENE	SOUTH AFRICA
LOM KCHZ LABORATORIUM REFERENCYJNE z/s w	PARZNIEWIE	POLAND
SUISSE LAB AG	ZOLLIKOFEN	SWITZERLAND
UNI.NOVOM SADU MLEKA	MLEKA	SERBIA
VALACTA	STE ANNE DE BELLEVUE	CANADA

ICAR
PROFICIENCY TESTING SCHEME

March 2016

Raw Milk

Determination of LACTOSE CONTENT
Routine method

Sending date of statistical treatment : 19th april 2016

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

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

Nb	%	N°	d	Sd	D
1	9	5	- 0,026	0,028	0,038
2	18	9	- 0,036	0,027	0,045
3	27	11	- 0,036	0,028	0,046
4	36	8	- 0,034	0,038	0,051
5	45	3	+ 0,038	0,044	0,058
6	55	4	- 0,003	0,074	0,074
7	64	6	+ 0,113	0,045	0,121
8	73	10	+ 0,121	0,020	0,123
9	82	1	- 0,064	0,118	0,134
10	91	2	+ 0,028	0,145	0,148
11	100	7	- 0,761	1,445	1,634

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

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

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 11 sets of results send by 9 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}	0,007
Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %)	SR_{PT}	0,099

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	0,010	0,020	0,010	0,000	0,000	0,010	0,000	0,000	0,007	20
2	0,010	0,010	0,000	0,000	0,000	0,010	0,010	0,010	0,000	0,000	0,005	20
3	0,000	0,030	0,000	0,000	0,020	0,010	0,020	0,000	0,010	0,000	0,010	20
4	0,010	0,010	0,010	0,010	0,000	0,020	0,000	0,000	0,000	0,000	0,006	20
5	0,010	0,000	0,000	0,000	0,000	0,000	0,010	0,020	0,010	0,000	0,006	20
6	0,010	0,000	0,010	0,010	0,010	0,020	0,000	0,010	0,010	0,020	0,008	20
7	**	0,060 *	0,060 *	0,020	0,100 *	0,050 *	0,040 *	0,070 *	0,020 *	0,020	0,039	18
8	0,000	0,000	0,000	0,000	0,000	0,020	0,000	0,000	0,000	0,000	0,004	20
9	0,000	0,000	0,000	0,000	**	**	**	0,000	0,000	0,000	0,000	14
10	0,010	0,000	0,010	0,000	0,010	0,010	0,010	0,000	0,000	0,000	0,005	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
Sr	0,005	0,015	0,013	0,007	0,023	0,014	0,011	0,016	0,006	0,006		212
NE	20	22	22	22	20	20	20	22	22	22		
L	0,017	0,030	0,016	0,024	0,023	0,031	0,021	0,021	0,013	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	4,710	4,850	4,785	4,820	4,745	4,830	4,760	4,805	4,860	4,730
2	4,735	5,005	4,850	4,930	4,790	4,985	4,815	4,895	5,040	4,770
3	4,940	5,085	5,020	4,790	4,870	4,835	4,810	5,160	4,755	4,650
4	4,905	5,085	5,005	4,705	4,830	4,790	4,730	5,180	4,670	4,600
5	4,865	5,010	4,930	4,710	4,810	4,790	4,735	5,070	4,685	4,670
6	5,005	5,170	5,095	4,845	4,945	4,920	4,880	5,245	4,815	4,740
7		4,710	4,650	4,470	4,520 *	4,515 *	4,490 *	4,755	4,450	4,360 *
8	4,870	5,010	4,940	4,710	4,800	4,770	4,720	5,080	4,680	4,610
9	4,840	4,990	4,930	4,710				5,050	4,680	4,610
10	5,015	5,150	5,075	4,890	4,955	4,935	4,895	5,200	4,850	4,780
11	4,860	5,000	4,930	4,710	4,795	4,770	4,740	5,060	4,680	4,625
M	4,875	5,006	4,928	4,754	4,838	4,847	4,787	5,045	4,742	4,679
REF.	4,875	5,018	4,937	4,765	4,838	4,847	4,787	5,048	4,742	4,679
SD	0,100	0,131	0,130	0,124	0,072	0,080	0,066	0,162	0,150	0,070

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 11 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		7	7		7	7	7	7	7	
Outlier Grubbs					7	7	7			7
sr	0,005	0,009	0,004	0,007	0,007	0,009	0,006	0,006	0,004	0,005
SR	0,100	0,092	0,096	0,124	0,072	0,080	0,066	0,137	0,121	0,070

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	- 0,165	- 0,168	- 0,152	+ 0,055	- 0,093	- 0,017	- 0,027	- 0,243	+ 0,118	+ 0,051	- 0,064	0,118	1,71
2	- 0,140	- 0,013	- 0,087	+ 0,165	- 0,048	+ 0,138	+ 0,028	- 0,153	+ 0,298	+ 0,091	+ 0,028	0,145	0,61
3	+ 0,066	+ 0,067	+ 0,083	+ 0,025	+ 0,032	- 0,012	+ 0,023	+ 0,112	+ 0,013	- 0,029	+ 0,038	0,044	2,76
4	+ 0,031	+ 0,067	+ 0,068	- 0,060	- 0,008	- 0,057	- 0,057	+ 0,132	- 0,072	- 0,079	- 0,003	0,074	0,15
5	- 0,010	- 0,008	- 0,007	- 0,055	- 0,028	- 0,057	- 0,052	+ 0,022	- 0,057	- 0,009	- 0,026	0,028	2,93
6	+ 0,131	+ 0,152	+ 0,158	+ 0,080	+ 0,107	+ 0,073	+ 0,093	+ 0,197	+ 0,073	+ 0,061	+ 0,113	0,045	7,89
7		- 0,308	- 0,287	- 0,295	- 0,318	- 0,332	- 0,297	- 0,293	- 0,292	- 0,319	- 0,304	0,015	59,83
8	- 0,005	- 0,008	+ 0,003	- 0,055	- 0,038	- 0,077	- 0,067	+ 0,032	- 0,062	- 0,069	- 0,034	0,038	2,90
9	- 0,035	- 0,028	- 0,007	- 0,055				+ 0,002	- 0,062	- 0,069	- 0,036	0,027	3,50
10	+ 0,140	+ 0,132	+ 0,138	+ 0,125	+ 0,117	+ 0,088	+ 0,108	+ 0,152	+ 0,108	+ 0,102	+ 0,121	0,020	19,17
11	- 0,015	- 0,018	- 0,007	- 0,055	- 0,043	- 0,077	- 0,047	+ 0,012	- 0,062	- 0,054	- 0,036	0,028	4,09
d	- 0,000	- 0,012	- 0,009	- 0,012	+ 0,000	+ 0,000	- 0,000	- 0,002	+ 0,001	- 0,000	- 0,016	0,124	
Sd	0,100	0,131	0,130	0,124	0,072	0,080	0,066	0,162	0,150	0,070	0,114		

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	-1,64	-1,28	-1,18	+0,44	-1,29	-0,21	-0,41	-1,50	+0,79	+0,73
2	-1,39	-0,10	-0,67	+1,33	-0,67	+1,73	+0,42	-0,94	+1,99	+1,30
3	+0,65	+0,51	+0,64	+0,20	+0,45	-0,15	+0,34	+0,69	+0,09	-0,41
4	+0,30	+0,51	+0,52	-0,49	-0,11	-0,71	-0,87	+0,82	-0,48	-1,12
5	-0,09	-0,06	-0,06	-0,45	-0,39	-0,71	-0,79	+0,14	-0,38	-0,12
6	+1,30	+1,16	+1,22	+0,64	+1,49	+0,92	+1,40	+1,22	+0,49	+0,88
7		-2,34	-2,22	-2,38	-4,43	-4,16	-4,49	-1,81	-1,94	-4,53
8	-0,04	-0,06	+0,02	-0,45	-0,53	-0,96	-1,02	+0,20	-0,41	-0,98
9	-0,34	-0,21	-0,06	-0,45				+0,01	-0,41	-0,98
10	+1,40	+1,01	+1,06	+1,00	+1,63	+1,11	+1,63	+0,94	+0,72	+1,45
11	-0,14	-0,13	-0,06	-0,45	-0,60	-0,96	-0,71	+0,08	-0,41	-0,76

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

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

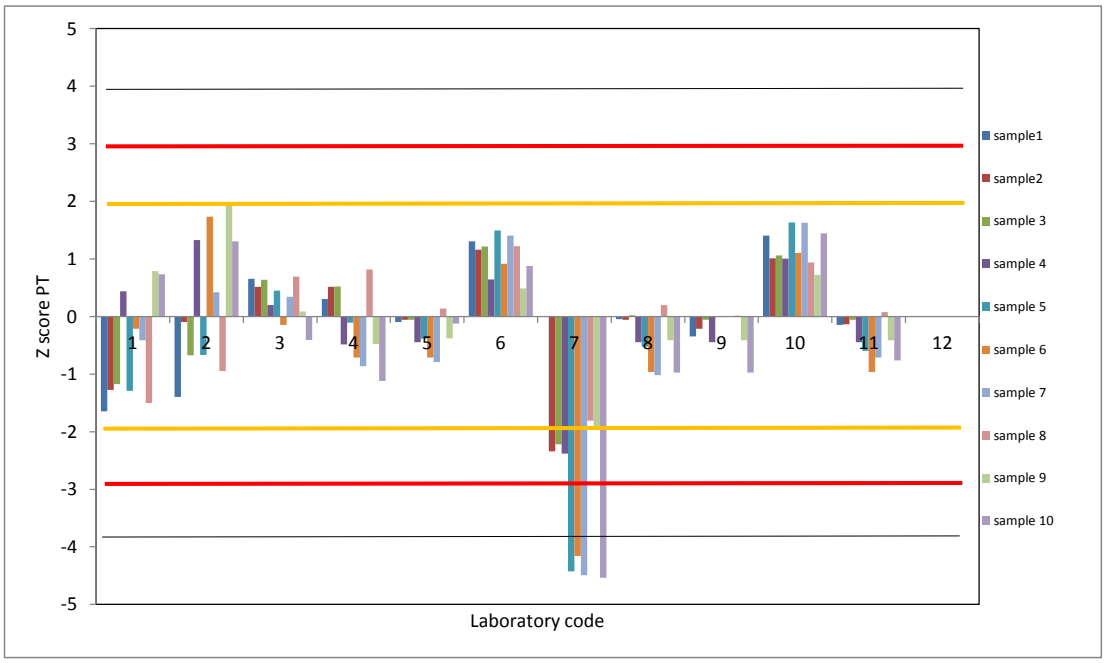


Table VII : **Zscore of the different laboratories for each sample.**
ZS calculated on the standard deviation of reproducibility of the method

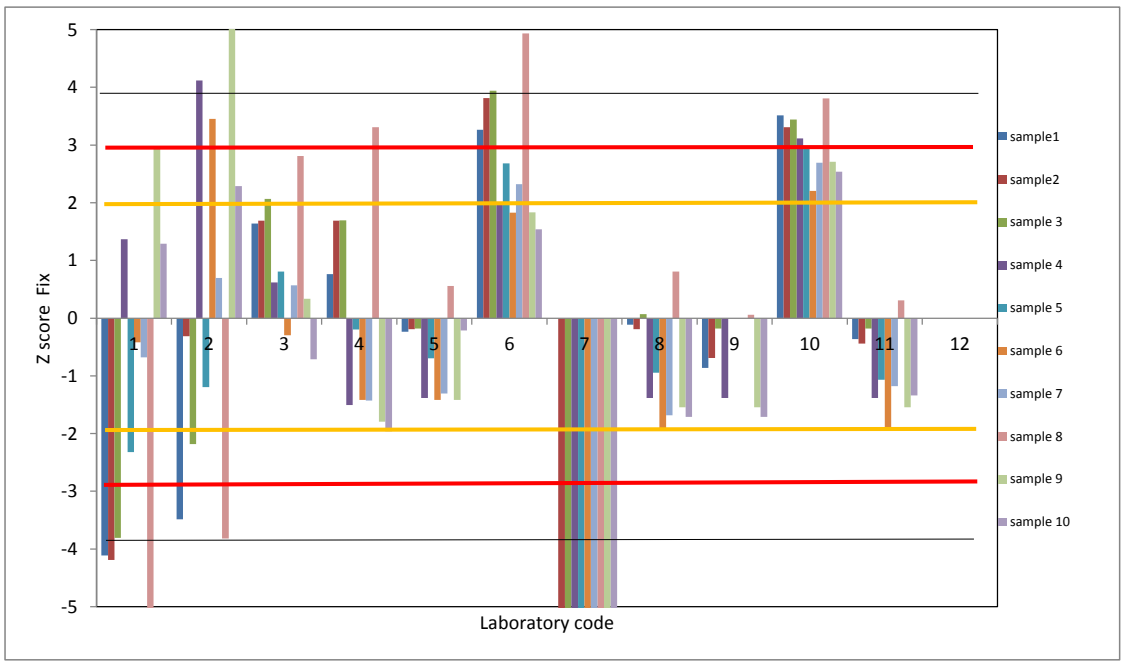
Sample / Lab code	1	2	3	4	5	6	7	8	9	10
1	-4,11	-4,19	-3,81	+1,37	-2,32	-0,42	-0,68	-6,07	+2,96	+1,29
2	-3,49	-0,31	-2,18	+4,12	-1,19	+3,46	+0,69	-3,82	+7,46	+2,29
3	+1,64	+1,69	+2,07	+0,62	+0,81	-0,29	+0,57	+2,81	+0,33	-0,71
4	+0,76	+1,69	+1,69	-1,51	-0,19	-1,42	-1,43	+3,31	-1,79	-1,96
5	-0,24	-0,19	-0,18	-1,38	-0,69	-1,42	-1,31	+0,56	-1,42	-0,21
6	+3,26	+3,81	+3,94	+1,99	+2,68	+1,83	+2,32	+4,93	+1,83	+1,54
7		-7,69	-7,18	-7,38	-7,94	-8,29	-7,43	-7,32	-7,29	-7,96
8	-0,11	-0,19	+0,07	-1,38	-0,94	-1,92	-1,68	+0,81	-1,54	-1,71
9	-0,86	-0,69	-0,18	-1,38				+0,06	-1,54	-1,71
10	+3,51	+3,31	+3,44	+3,12	+2,93	+2,21	+2,69	+3,81	+2,71	+2,54
11	-0,36	-0,44	-0,18	-1,38	-1,07	-1,92	-1,18	+0,31	-1,54	-1,34

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



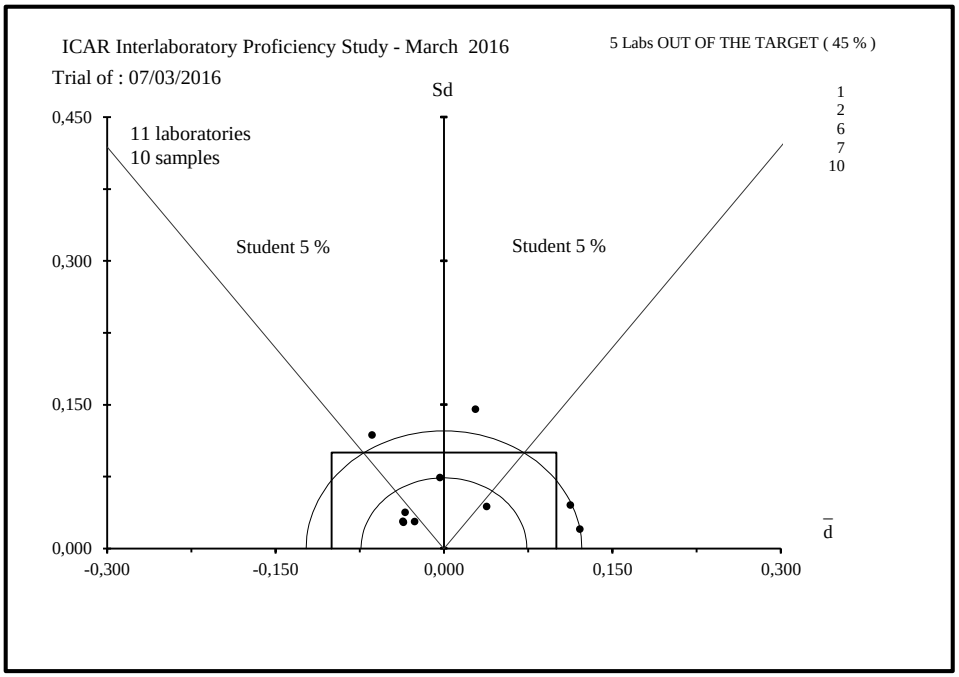


Figure 1 : **ACCURACY - Evaluation of the individual performances (to see table I).**

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

Name	City	Country
AIA-LAB. STANDARD LATTE	MACARESE (ROMA)	ITALY
ALLATTENYESZTESI TELJESITMENYVIZSGALO KFT	GODOLLO	HUNGARY
CATTLE INFORMATION SERVICE (CIS)	TELFORD	UNITED KINGDOM
CENTRAL MILK LABORATORY - ICBA	CAESAREA	ISRAEL
FED.LATTERIE SOCIALI DI BOLZANO	BOLZANO	ITALY
LACTOLAB	IRENE	SOUTH AFRICA
LOM KCHZ LABORATORIM REFERENCYJNE	PRUSZKOW	POLAND
SUISSE LAB AG	ZOLLIKOFEN	SWITZERLAND
UNI.NOVOM SADU MLEKA	MLEKA	SERBIA

ICAR
PROFICIENCY TESTING SCHEME

March 2016

Raw Milk

Determination of UREA CONTENT
Routine method

Sending date of statistical treatment : 19th april 2016

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

ACCRÉDITATION
N° 1-2473
PORTÉE
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Table I : **Ranking of the laboratories** Units : mg / 100 g

Nb	%	N°	d	Sd	D
1	13	4	- 0,49	0,78	0,92
2	25	2	- 0,32	1,91	1,94
3	38	8	+ 1,30	2,07	2,45
4	50	6	+ 2,13	1,37	2,53
5	63	1	- 2,47	1,74	3,02
6	75	5	+ 3,92	1,04	4,06
7	88	7	+ 0,15	4,93	4,93
8	100	3	- 4,17	4,70	6,28

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 8 sets of results send by 7 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} 0,88
Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) SR_{PT} 3,28

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	2,00	2,00	1,00	1,00	0,00	1,00	0,00	3,00	2,00	2,00	1,18	20
2	0,00	0,00	3,00	2,00	2,00	1,00	2,00	0,00	0,00	1,00	1,07	20
3	0,35	1,14	0,53	0,09	0,37	0,77	0,23	27,15 *	0,14	0,25	6,08	20
4	1,20	1,50	1,80	0,10	2,20	4,30 *	0,90	4,10	4,60 *	6,30 *	2,33	20
5	0,40	1,60	1,20	1,50	0,60	1,50	1,20	0,00	0,40	0,40	0,73	20
6	**	0	**	0	0	**	0	0	1	1	0,378	14
7	0,40	0,30	0,80	0,10	0,80	0,80	0,10	1,70	**	**	0,56	16
8	2,60	0,30	0,20	0,50	0,40	0,00	0,80	0,40	0,10	1,10	0,68	20
Sr	0,95	0,80	1,06	0,69	0,80	1,31	0,66	6,92	1,37	1,83		150
NE	14	16	14	16	16	14	16	16	14	14		
L	3,03	2,65	3,37	2,26	2,63	2,07	2,17	4,58	2,01	2,41		

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	21,00	45,00	29,50	33,50	38,00	46,50	25,00	52,50	56,00	61,00
2	16,00	45,00	29,50	35,00	41,00	49,50	26,00	58,00	63,00	66,50
3	17,66	41,36	28,40	33,59	38,28	47,77	23,38	39,02	58,42	63,10
4	20,50	46,35	30,50	35,85	40,30	49,95	25,25	54,25	60,20	64,65
5	23,50	48,90	34,70	40,25	44,20	52,75	30,60	61,20	64,70	71,10
6		46,00		40,00	43,00		30,00	58,00	63,50	65,50
7	19,00	44,65	27,90	32,65	38,10	55,00	23,65	67,05		
8	26,30	46,65	33,80	38,05	41,40	51,50	28,00	54,60	60,85	64,55
M	20,57	45,49	30,61	36,11	40,53	50,42	26,48	55,58	60,95	65,20
REF.	20,57	45,61	30,61	36,11	40,53	50,42	26,48	56,42	60,95	64,97
SD	3,50	2,15	2,63	2,99	2,33	2,92	2,76	8,12	3,06	3,15

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 8 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						4		3	4	4
Outlier Grubbs										
sr	0,95	0,80	1,06	0,69	0,80	0,68	0,66	1,44	0,66	0,79
SR	3,56	2,22	2,74	3,03	2,40	3,22	2,80	5,07	3,37	3,48

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	+ 0,43	- 0,61	- 1,11	- 2,61	- 2,53	- 3,92	- 1,48	- 3,92	- 4,95	- 3,97	- 2,47	1,74	4,48
2	- 4,57	- 0,61	- 1,11	- 1,11	+ 0,47	- 0,92	- 0,48	+ 1,58	+ 2,05	+ 1,53	- 0,32	1,91	0,53
3	- 2,91	- 4,25	- 2,22	- 2,53	- 2,26	- 2,66	- 3,11	- 17,41	- 2,53	- 1,88	- 4,17	4,70	2,81
4	- 0,07	+ 0,74	- 0,11	- 0,26	- 0,23	- 0,47	- 1,23	- 2,17	- 0,75	- 0,32	- 0,49	0,78	1,99
5	+ 2,94	+ 3,29	+ 4,09	+ 4,14	+ 3,67	+ 2,33	+ 4,12	+ 4,78	+ 3,75	+ 6,13	+ 3,92	1,04	11,93
6		+ 0,39		+ 3,89	+ 2,47		+ 3,52	+ 1,58	+ 2,55	+ 0,53	+ 2,13	1,37	4,13
7	- 1,57	- 0,96	- 2,71	- 3,46	- 2,43	+ 4,58	- 2,83	+ 10,63			+ 0,15	4,93	0,09
8	+ 5,74	+ 1,04	+ 3,19	+ 1,94	+ 0,87	+ 1,08	+ 1,52	- 1,82	- 0,10	- 0,42	+ 1,30	2,07	1,98
d	- 0,00	- 0,12	- 0,00	+ 0,00	+ 0,00	+ 0,00	- 0,00	- 0,85	+ 0,00	+ 0,23	- 0,07	3,55	
Sd	3,50	2,15	2,63	2,99	2,33	2,92	2,76	8,12	3,06	3,15	3,73		

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	+0,12	-0,28	-0,42	-0,87	-1,09	-1,35	-0,54	-0,48	-1,62	-1,26
2	-1,30	-0,28	-0,42	-0,37	+0,20	-0,32	-0,18	+0,19	+0,67	+0,49
3	-0,83	-1,98	-0,84	-0,85	-0,97	-0,91	-1,13	-2,14	-0,83	-0,60
4	-0,02	+0,35	-0,04	-0,09	-0,10	-0,16	-0,45	-0,27	-0,25	-0,10
5	+0,84	+1,53	+1,55	+1,39	+1,57	+0,80	+1,49	+0,59	+1,22	+1,95
6		+0,18		+1,30	+1,06		+1,27	+0,19	+0,83	+0,17
7	-0,45	-0,45	-1,03	-1,16	-1,04	+1,57	-1,03	+1,31		
8	+1,64	+0,49	+1,21	+0,65	+0,37	+0,37	+0,55	-0,22	-0,03	-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

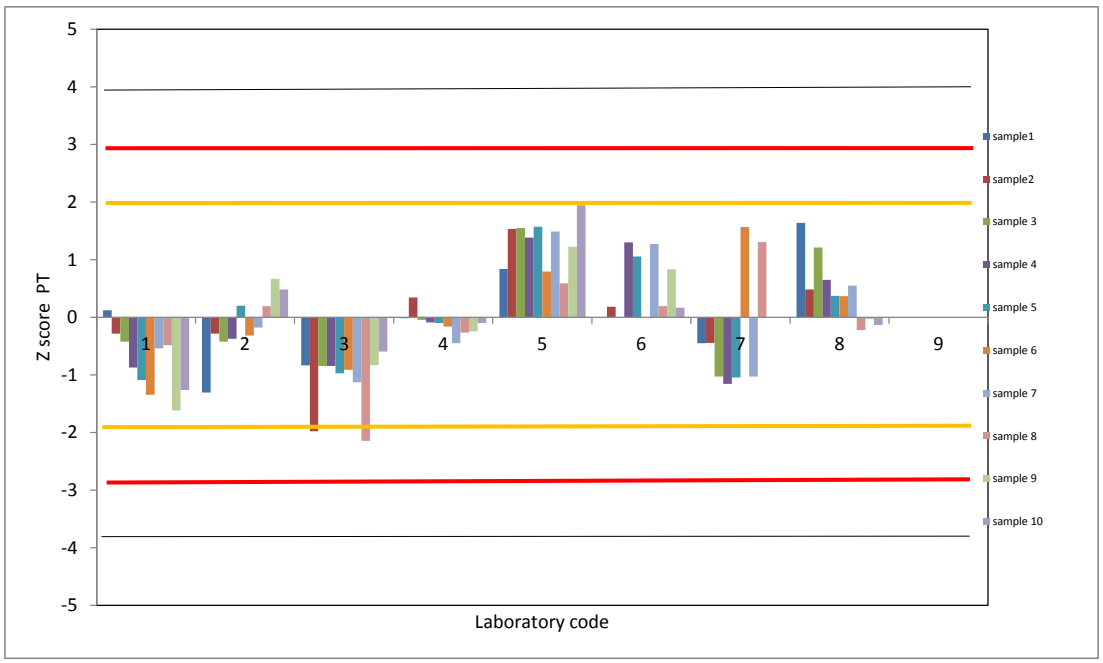


Table VII : **Zscore of the different laboratories for each sample.**
ZS calculated on the standard deviation of reproducibility of the method

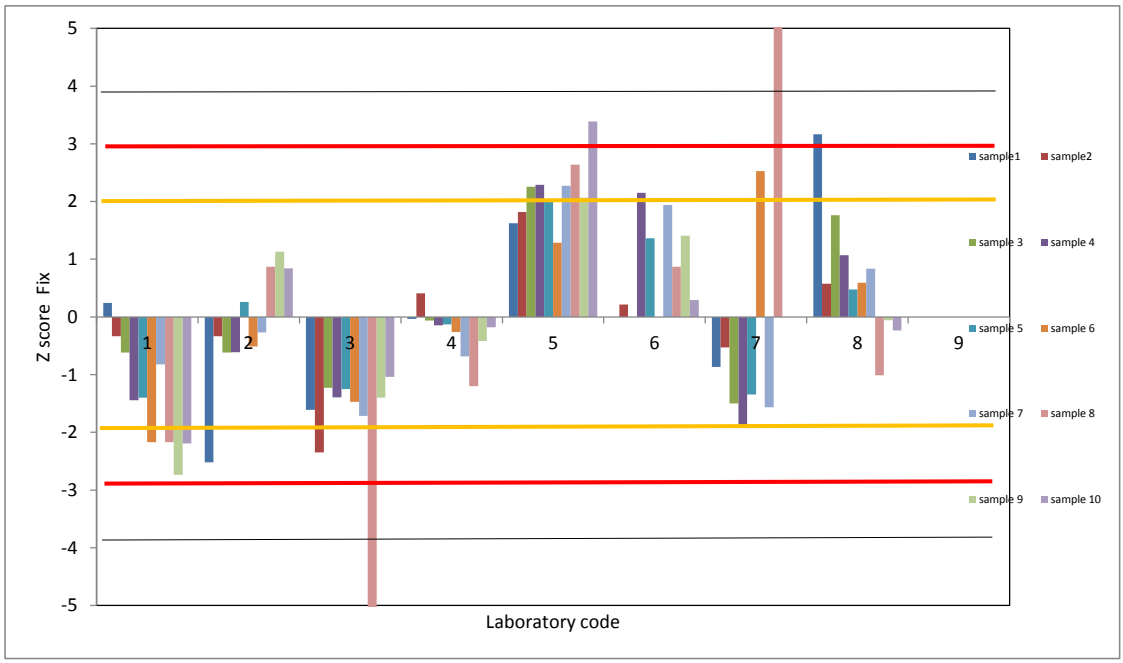
Sample / Lab code	1	2	3	4	5	6	7	8	9	10
1	+0,24	-0,34	-0,62	-1,44	-1,40	-2,17	-0,82	-2,17	-2,74	-2,19
2	-2,52	-0,34	-0,62	-0,61	+0,26	-0,51	-0,27	+0,87	+1,13	+0,84
3	-1,61	-2,35	-1,23	-1,40	-1,25	-1,47	-1,72	-9,62	-1,40	-1,04
4	-0,04	+0,41	-0,06	-0,14	-0,13	-0,26	-0,68	-1,20	-0,42	-0,18
5	+1,62	+1,82	+2,26	+2,29	+2,03	+1,29	+2,27	+2,64	+2,07	+3,39
6		+0,22		+2,15	+1,36		+1,94	+0,87	+1,41	+0,29
7	-0,86	-0,53	-1,50	-1,91	-1,34	+2,53	-1,57	+5,87		
8	+3,17	+0,58	+1,76	+1,07	+0,48	+0,59	+0,84	-1,01	-0,06	-0,23

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



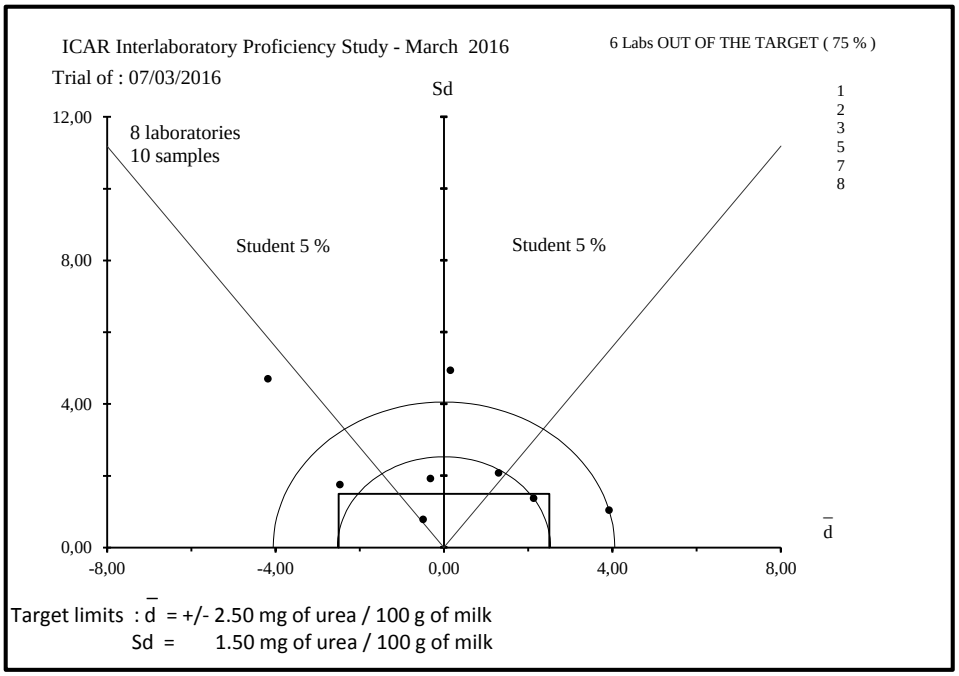


Figure 1 : **ACCURACY - Evaluation of the individual performances (to see table I).**

LIST OF THE PARTICIPANTS ICAR
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UREA CONTENT Infrared method
March 2016

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CATTLE INFORMATION SERVICE (CIS)	TELFORD	UNITED KINGDOM
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LACTOLAB	IRENE	SOUTH AFRICA
LOM KCHZ LABORATORIM REFERENCYJNE	PRUSZKOW	POLAND
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