



THE GLOBAL STANDARD
FOR LIVESTOCK DATA

Network. Guidelines. Certification.

ICAR PROFICIENCY TEST - SEPTEMBER 2019

Raw cow milk “Reference” Methods





THE GLOBAL STANDARD
FOR LIVESTOCK DATA

Network. Guidelines. Certification.

FRAME OF ACTIVITY :

ICAR MILK ANALYSES SUB-COMMITTEE (MA SC)

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 2019 !

This is the eight round that ICAR organized since 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. Its 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, 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) It is the ranking of your laboratory. The values of table 1 for each parameter are reported. 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. During the meeting of Milk Analyses Sub Committee held in Copenhagen in June 2016 the experts decided to update the limit of the box to evaluate the accuracy.
- E) Here are reported the samples that resulted outlier for your participation code for Cochran and/or Grubbs test
- F) The evaluation of repeatability of the results should be one of the first controls before communication of the data. In table F 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.

- G) In table G 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 $-2 < \text{Z-Score}$ 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.

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

In the second part of the report the statistical elaboration followed the template approved by ICAR's Milk Analyses Sub Committee. You find the statistical elaboration for all the ICAR interested parameters, fat, protein, lactose, urea and somatic cell.

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

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

ICAR would like to see, in the next years, 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 2019 with at least one parameter is reported below and upload on ICAR website (available [here](#))

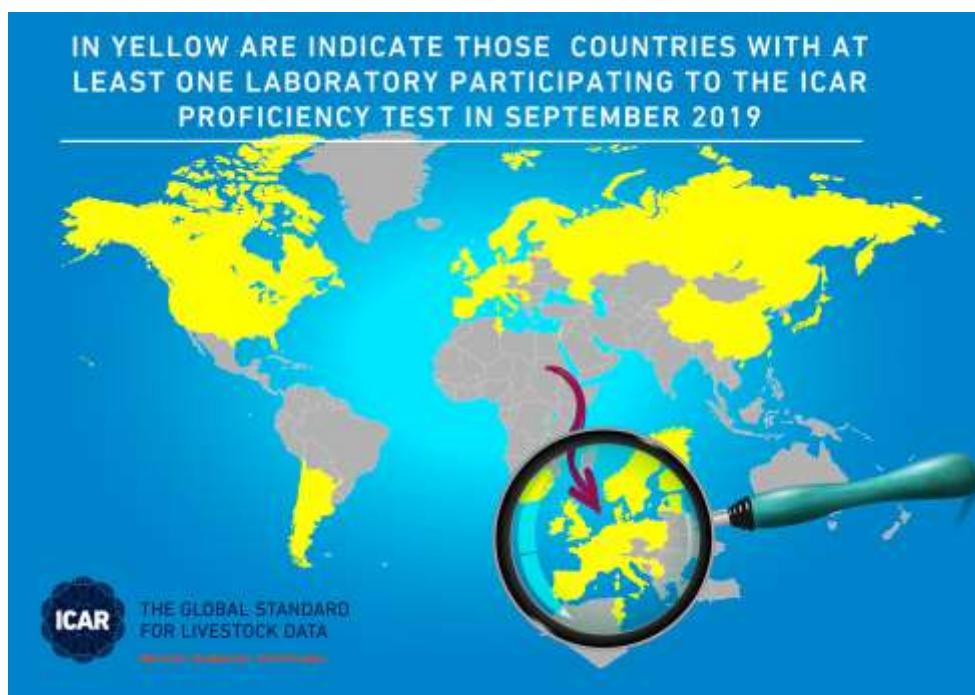


Table 1. Participating milk laboratories to the ICAR Proficiency Test (September 2019)

Country	Laboratory
Argentina	INTI (Instituto Nacional de Tecnología Industrial)
Belgium	Laboratory of milk analysis of the Valorisation of Agricultural Products, Department of Agricultural products of Walloon Agricultural Research Centre
Canada	Horizon Lab Ltd
Canada	Lactanet Canada - Centre d'Expertise en Production Laitière du Québec
Chile	COOPRINSEM
Croatia	Croatian Agency for Agriculture and Food, Central Laboratory for Milk Quality Control (Centar za kontrolu kvalitete stočarskih proizvoda)
Cyprus	Epta Laboratories LTD
Czech Republic	Laborator pro rozbor mléka Brno, Ceskomoravská společnost chovatelů a.s.
Czech Republic	MILCOM a.s Dairy Research Institute
Denmark	ChemoMetec A/S
Denmark	DNA Diagnostic
Denmark	Eurofins Steins Laboratorium A/S
Denmark	LVK
Finland	Osuuskunta Satamaito, Laboratorio Kati Järvinen
Finland	Thermo Fisher Scientific
Finland	Valio Ltd, Regional laboratory
France	ACTALIA / ACTILAIT / CECALAIT
France	IDvet Genetics
France	Thermo Fisher Scientific Lab Service International
Germany	Milchprüfing Baden-Württemberg e.V., Zentrallabor Kirchheim
Ireland	Teagasc, Technical Services Laboratory
Israel	Central Milk Laboratory – ICBA
Italy	Associazione Italiana Allevatori, Laboratorio Standard Latte (LSL-AIA)
Japan	Japan Dairy Technical Association
Lithuania	Pieno Tyrimai, State Laboratory for Milk Control
Norway	Tine Ramelklaboratoriet Heimdal
Poland	Laboratorium Oceny Mleka, Krajowego Centrum Hodowli Zwierząt (KCHZ), Laboratorium Referencyjne z siedziba w Parzniewie
Russia	Ao Agropfen
Serbia	Laboratorija za ispitivanje kvaliteta mleka, Poljoprivredni fakultet Novi Sad
Slovak Republic	Plemenárske služby SR, š.p., Centrálné laboratórium rozboru mlieka (Milk Laboratory, Slovak Agricultural Research Centre)



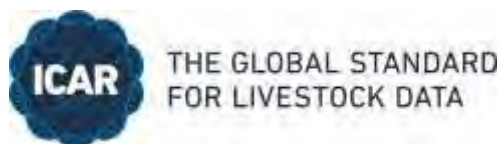
Country	Laboratory
Slovenia	KGZS Zavod Ptuj
Slovenia	University of Ljubljana, Biotechnical Faculty, Zootech. Dept., Laboratory for Dairying
South Africa	Deltamune Pty Ltd.
South Africa	Mérieux NutriSciences South Africa
South Africa	University of Pretoria, Department of Production Animal Studies, Faculty of Veterinary Science
South Korea	Korea Animal Improvement Association
Spain	Ministero de Agricultura, Pesca y Alimentacion Laboratorio Agroalimentario de Santander Cantabria
Sweden	Eurofins Steins Laboratory A/B
Switzerland	Agroscope Institute for food Sciences IFS
Switzerland	Suisselab AG
Taiwan	Council of Agriculture, Executive Yuan, Taiwan Animal Germplasm Center of TLRI
The Netherlands	Qlip B.V.
Tunisia	Office de l'Elevage et des Pâturages, Laboratoire de Contrôle Laitier, Direction de l'Amélioration Génétique
UK	Nationl Milk Laboratory
United Kingdom	CIS
USA	Eastern Laboratory Services
USA	NorthStar Cooperative

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





Chemical Reference Methods Laboratory participation codes and Performance analyses

ICAR PT
RF0919

Laboratory Name						
-----------------	--	--	--	--	--	--

A	Your participation Codes					
	Subscription	Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		Yes	Yes	Yes	Yes	Yes
	Participation Codes	7	7	6	5	8
	Are all the sample results received?	Yes	Yes	Yes	Yes	Yes

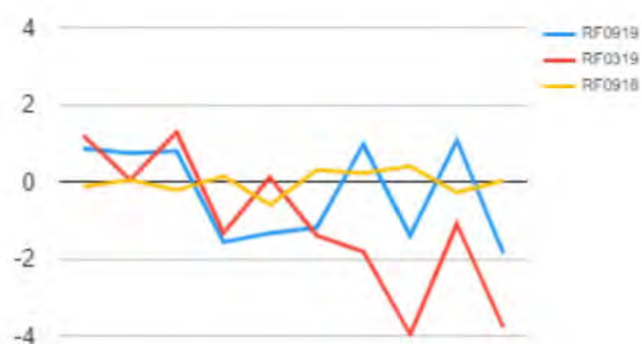
B	Data results received on time	
	Yes	13-09-2019

C	Have you sent the data with the correct units of measurements?					
		Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
		Yes	Yes	Yes	Yes	Yes
* It was requested to report the value in total nitrogen						

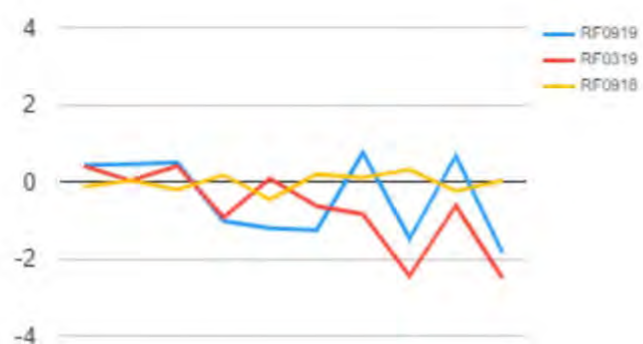
D	Ranking of your lab					
		Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
	Code	7	7	6	5	8
	%	79	42	29	67	7%
	d	0.008	0.008	0.044	0.908	0
	Sd	0.021	0.011	0.018	0.785	3%
	D	0.022	0.013	0.047	1.200	3%
	Method	ISO 1211 IDF 1	ISO 8968-1 IDF 20-1	ISO 22662 / IDF 198	Continuous flow analyzer	ISO 13366-2 IDF 148-2
	Limits					
d	0.020	0.025	0.10	2.5	10%	
Sd	0.030	0.020	0.10	1.5	10%	

E	Outliers					
		Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
	Sample 1					
	Sample 2					
	Sample 3					
	Sample 4					
	Sample 5					
	Sample 6					
	Sample 7					
	Sample 8					
	Sample 9					
Sample 10						

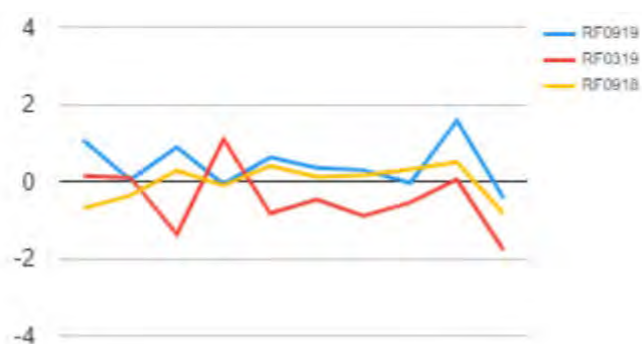
ZSCORE-PT - fat_ref



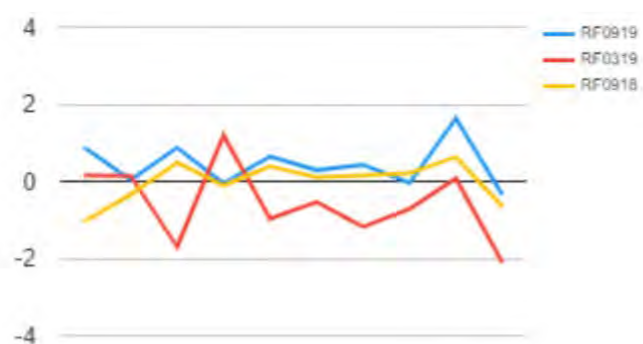
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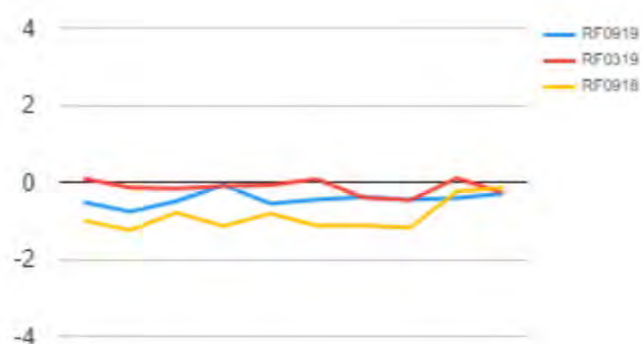
ZSCORE-PT - protein_ref



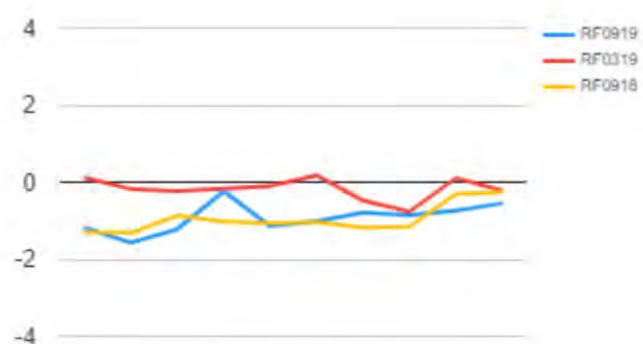
ZSCORE-FIX - protein_ref



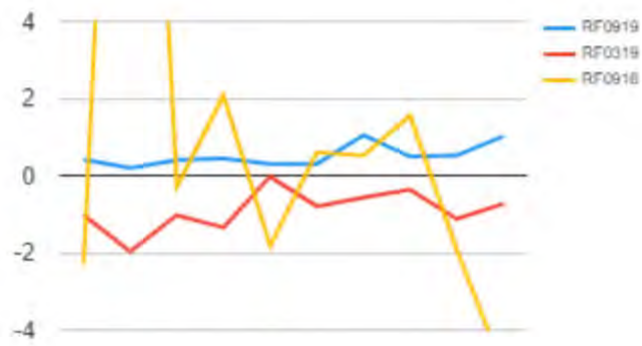
ZSCORE-PT - lactose_ref



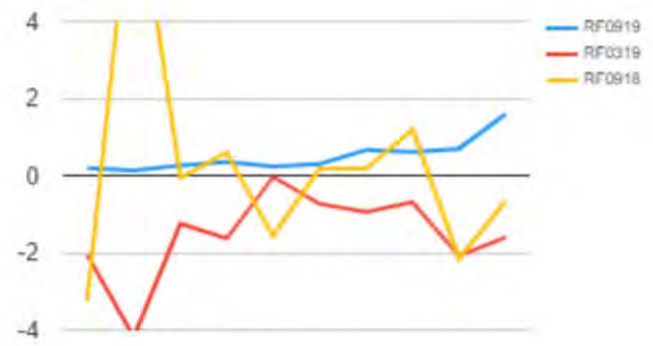
ZSCORE-FIX - lactose_ref



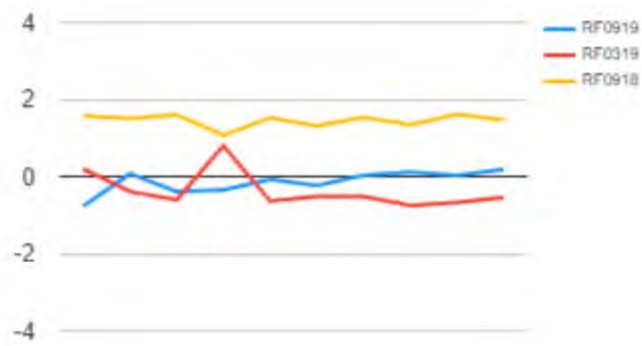
ZSCORE-PT - urea_ref



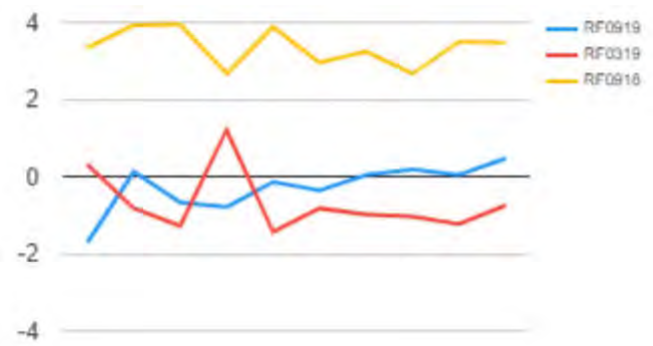
ZSCORE-FIX - urea_ref



ZSCORE-PT - scc_ref



ZSCORE-FIX - scc_ref





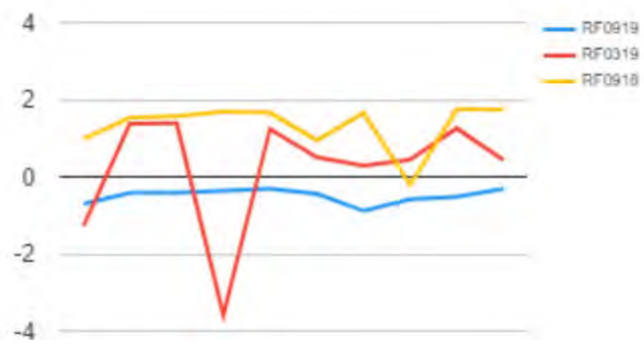
Chemical Reference Methods

Laboratory participation codes and Performance analyses

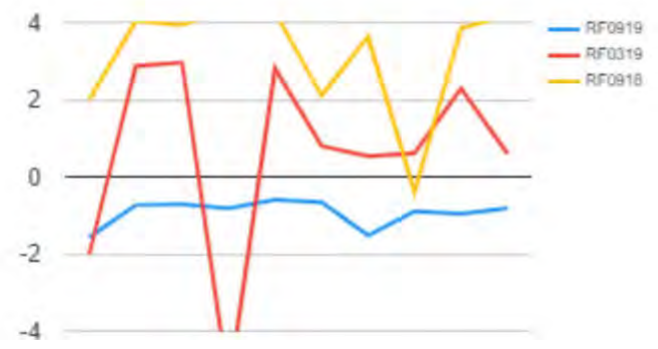
ICAR PT
RF0919

Laboratory Name						
A	Your participation Codes					
	Subscription	Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		No	No	No	No	Yes
	Participation Codes					32
	Are all the sample results received?	No	No	No	No	Yes
B	Data results received on time					
	Yes	13-09-2019				
C	Have you sent the data with the correct units of measurements?					
		Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
						Yes
* It was requested to report the value in total nitrogen						
D	Ranking of your lab					
		Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
	Code					32
	%					45%
	d					6%
	Sd					5%
	D					8%
	Method					ISO 13366-2 IDF 148-2
	Limits					
d	0,020	0,025	0,10	2,5	10%	
Sd	0,030	0,020	0,10	1,5	10%	
E	Outliers					
		Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
	Sample 1					
	Sample 2					
	Sample 3					
	Sample 4					
	Sample 5					
	Sample 6					
	Sample 7					
	Sample 8					
Sample 9						
Sample 10						

ZSCORE-PT - scc_ref



ZSCORE-FIX - scc_ref





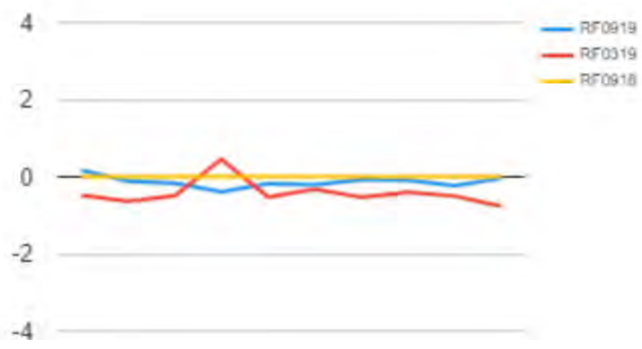
Chemical Reference Methods

Laboratory participation codes and Performance analyses

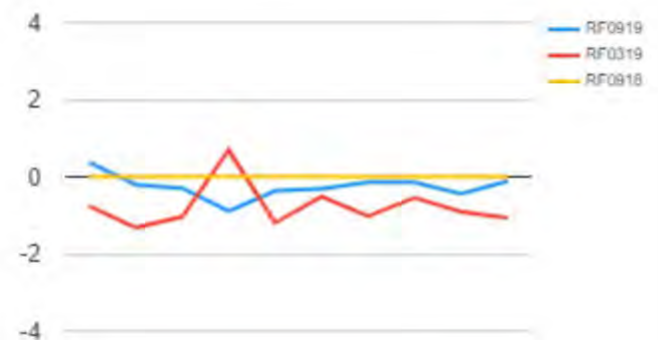
ICAR PT
RF0919

Laboratory Name						
A	Your participation Codes					
	Subscription	Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		No	No	No	No	Yes
	Participation Codes					35
	Are all the sample results received?	No	No	No	No	Yes
B	Data results received on time					
	Yes	13-09-2019				
C	Have you sent the data with the correct units of measurements?					
		Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
						Yes
* It was requested to report the value in total nitrogen						
D	Ranking of your lab					
		Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
	Code					35
	%					2%
	d					2%
	Sd					2%
	D					3%
	Method					ISO 13366-2 IDF 148-2
	Limits					
d	0,020	0,025	0,10	2,5	10%	
Sd	0,030	0,020	0,10	1,5	10%	
E	Outliers					
		Fat _{ref}	Protein _{ref}	Lactose _{ref}	Urea _{ref}	SCC _{ref/alt}
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
	Sample 1					
	Sample 2					
	Sample 3					
	Sample 4					
	Sample 5					
	Sample 6					
	Sample 7					
	Sample 8					
Sample 9						
Sample 10						

ZSCORE-PT - scc_ref



ZSCORE-FIX - scc_ref





ICAR
PROFICIENCY TESTING SCHEME

September 2019

Raw Milk

Determination of FAT CONTENT

Röse Gottlieb method

Sending date of statistical treatment : 3th October 2019

Frame of activity :	ICAR Milk Analyses Sub Committee (MA SC)		
ICAR Staff	Silvia Orlandini	pt@icar.org	silvia@icar.org

Proficiency test accredited ISO 17043



ACCREDITATION
N° 1 2473
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Table I : **Ranking of the laboratories** Units : g / 100 g

Nb	%	N°	d	Sd	D	Method
1	5	15	+ 0,002	0,002	0,003	A
2	11	3	+ 0,005	0,004	0,006	A
3	16	2	- 0,001	0,007	0,007	A
4	21	1	+ 0,001	0,007	0,007	A
5	26	9	- 0,001	0,007	0,007	A
6	32	13	+ 0,007	0,005	0,008	A
7	37	12	+ 0,008	0,005	0,009	A
8	42	4	- 0,001	0,010	0,010	A
9	47	10	+ 0,004	0,009	0,010	A
10	53	8	- 0,009	0,010	0,013	A
11	58	6	- 0,011	0,009	0,014	A
12	63	17	- 0,002	0,017	0,017	A
13	68	11	+ 0,009	0,019	0,021	A
14	74	5	+ 0,002	0,022	0,022	B
15	79	7	- 0,008	0,021	0,022	A
16	84	14	- 0,029	0,012	0,031	A
17	89	18	- 0,013	0,032	0,035	A
18	95	16	+ 0,004	0,037	0,038	A
19	100	19	+ 0,060	0,051	0,079	C

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 17 laboratories using the reference method ISO 1211|IDF 1, after outliers discarding using Grubbs test at 5% risk level,

A ISO 1211|IDF 1 Röse Gottlieb Method
B ISO 2446|IDF 226 Gerber method
C Not specified

(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.(d² + Sd²))

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} 0,016

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	0,005	0,017	0,008	0,002	0,008	0,010	0,008	0,002	0,007	0,000	0,006	20
2	0,012	0,007	0,001	0,005	0,010	0,025	0,000	0,004	0,001	0,004	0,007	20
3	0,000	0,007	0,009	0,006	0,002	0,007	0,001	0,011	0,007	0,020	0,006	20
4	0,000	0,000	0,020	0,000	0,000	0,000	0,020	0,010	0,010	0,000	0,007	20
5	0,040	0,030	0,020	0,020	0,020	0,020	0,020	0,020	0,000	0,030	0,017	20
6	0,024	0,013	0,005	0,002	0,019	0,007	0,013	0,007	0,008	0,022	0,010	20
7	0,003	0,004	0,003	0,007	0,004	0,005	0,018	0,018	0,019	0,001	0,008	20
8	0,004	0,008	0,002	0,004	0,001	0,008	0,001	0,011	0,003	0,001	0,004	20
9	0,011	0,011	0,008	0,017	0,004	0,011	0,010	0,002	0,002	0,019	0,008	20
10	0,009	0,015	0,015	0,010	0,006	0,005	0,006	0,005	0,020	0,004	0,008	20
11	0,006	0,001	0,006	0,002	0,005	0,017	0,003	0,002	0,004	0,002	0,005	20
12	0,008	0,005	0,002	0,003	0,007	0,003	0,001	0,003	0,007	0,007	0,004	20
13	0,011	0,001	0,004	0,006	0,013	0,023	0,005	0,001	0,010	0,001	0,007	20
14	0,003	0,000	0,015	0,013	0,018	0,013	0,011	0,007	0,008	**	0,008	18
15	**	0,000	**	0,020	**	**	**	0,010	**	0,000	0,008	8
16	0,010	0,010	0,000	0,010	0,000	0,010	0,000	0,010	0,010	0,010	0,006	20
17	0,015	0,000	0,005	**	0,002	0,001	0,000	0,024	0,009	0,002	0,007	18
18	0,015	0,018	0,017	**	0,027	0,004	0,009	0,034	0,002	0,005	0,013	18
19	0,030	0,040 *	0,040 *	0,110 *	0,070 *	0,100	0,090 *	0,020	0,010	0,050	0,046	20
Sr	0,010	0,008	0,007	0,007	0,007	0,009	0,008	0,007	0,007	0,009		286
NE	28	30	28	30	28	28	28	30	28	28		
L	0,042	0,031	0,028	0,027	0,032	0,033	0,027	0,038	0,025	0,032		

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

NL : number of measurements per laboratory

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

SE : repeatability standard deviation per sample

NE : number of measurements per sample

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

** : missing data

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

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

Sample Lab code	1	2	3	4	5	6	7	8	9	10
1	4,635	4,082	3,453	2,789	2,075	4,443	3,806	3,092	2,423	1,442
2	4,631	4,080	3,458	2,791	2,069	4,427	3,816	3,081	2,437	1,438
3	4,639	4,080	3,465	2,786	2,078	4,444	3,809	3,090	2,450	1,442
4	4,630	4,070	3,460	2,780	2,050	4,440	3,810	3,095	2,435	1,450
5	4,680	4,045	3,460	2,780	2,050	4,460	3,800	3,100	2,450	1,425
6	4,619	4,050	3,446	2,782	2,050	4,434	3,808	3,081	2,431	1,421
7	4,599	4,046	3,431	2,768	2,079	4,456	3,821	3,058	2,449	1,449
8	4,643	4,068	3,444	2,764	2,060	4,439	3,783	3,079	2,434	1,435
9	4,621	4,074	3,452	2,791	2,067	4,441	3,804	3,095	2,442	1,435
10	4,646	4,079	3,462	2,800	2,076	4,457	3,818	3,083	2,435	1,424
11	4,637	4,068	3,453	2,799	2,076	4,502 *	3,806	3,094	2,443	1,441
12	4,645	4,080	3,464	2,796	2,077	4,451	3,821	3,098	2,444	1,441
13	4,640	4,078	3,462	2,797	2,071	4,451	3,820	3,099	2,444	1,440
14	4,624	4,041	3,419	2,772	2,021 *	4,414	3,768	3,063	2,412	
15		4,080		2,790				3,085		1,440
16	4,675	4,105	3,490	2,815	1,980 *	4,455	3,800	3,085	2,435	1,435
17	4,655	4,082	3,476		2,068	4,436	3,783	3,051	2,441	1,440
18	4,622	4,086	3,418		2,083	4,376 *	3,785	3,036	2,459	1,463
19	4,655	4,130	3,500	2,885 *	2,045	4,610 *	3,865 *	3,160 *	2,465	1,515 *
M	4,638	4,075	3,456	2,787	2,067	4,443	3,803	3,081	2,440	1,439
REF.	4,635	4,076	3,456	2,788	2,070	4,442	3,806	3,082	2,439	1,440
SD	0,020	0,021	0,021	0,013	0,012	0,013	0,016	0,018	0,012	0,010

M = mean per sample

REF. = reference values

SD = standard deviation per sample

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

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 17 laboratories using the reference method ISO 1211 | IDF 1, after outliers discarding using Grubbs test at 5 % risk level.

Table IV : Outlier identification

Sample	1	2	3	4	5	6	7	8	9	10
Outliers Cochran		19	19	19	19		19			
Outlier Grubbs				19	14;16	11;18;19	19	19		19
sr	0,011	0,008	0,007	0,007	0,008	0,009	0,007	0,009	0,007	0,008
SR	0,021	0,018	0,019	0,014	0,013	0,014	0,016	0,019	0,013	0,012

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	- 0,001	+ 0,006	- 0,003	+ 0,001	+ 0,005	+ 0,001	- 0,000	+ 0,010	- 0,016	+ 0,002	+ 0,001	0,007	0,23
2	- 0,004	+ 0,004	+ 0,002	+ 0,002	- 0,001	- 0,016	+ 0,010	- 0,001	- 0,002	- 0,002	- 0,001	0,007	0,38
3	+ 0,004	+ 0,004	+ 0,009	- 0,002	+ 0,008	+ 0,001	+ 0,002	+ 0,007	+ 0,011	+ 0,002	+ 0,005	0,004	3,69
4	- 0,005	- 0,006	+ 0,004	- 0,008	- 0,020	- 0,002	+ 0,004	+ 0,013	- 0,004	+ 0,010	- 0,001	0,010	0,45
5	+ 0,045	- 0,031	+ 0,004	- 0,008	- 0,020	+ 0,018	- 0,006	+ 0,018	+ 0,011	- 0,015	+ 0,002	0,022	0,24
6	- 0,016	- 0,026	- 0,010	- 0,006	- 0,020	- 0,009	+ 0,001	- 0,002	- 0,008	- 0,019	- 0,011	0,009	4,14
7	- 0,037	- 0,030	- 0,025	- 0,021	+ 0,009	+ 0,013	+ 0,015	- 0,024	+ 0,010	+ 0,008	- 0,008	0,021	1,23
8	+ 0,008	- 0,008	- 0,012	- 0,024	- 0,010	- 0,003	- 0,024	- 0,004	- 0,005	- 0,006	- 0,009	0,010	2,88
9	- 0,015	- 0,002	- 0,004	+ 0,002	- 0,003	- 0,002	- 0,002	+ 0,013	+ 0,003	- 0,006	- 0,001	0,007	0,63
10	+ 0,010	+ 0,003	+ 0,006	+ 0,012	+ 0,006	+ 0,014	+ 0,012	+ 0,000	- 0,004	- 0,016	+ 0,004	0,009	1,52
11	+ 0,002	- 0,008	- 0,003	+ 0,011	+ 0,006	+ 0,060	- 0,000	+ 0,011	+ 0,005	+ 0,001	+ 0,009	0,019	1,43
12	+ 0,010	+ 0,004	+ 0,008	+ 0,007	+ 0,007	+ 0,008	+ 0,014	+ 0,015	+ 0,005	+ 0,000	+ 0,008	0,005	5,57
13	+ 0,004	+ 0,003	+ 0,006	+ 0,009	+ 0,002	+ 0,008	+ 0,013	+ 0,016	+ 0,005	- 0,001	+ 0,007	0,005	3,95
14	- 0,012	- 0,035	- 0,037	- 0,017	- 0,049	- 0,029	- 0,039	- 0,020	- 0,027		- 0,029	0,012	7,36
15		+ 0,004		+ 0,002				+ 0,003		- 0,000	+ 0,002	0,002	2,49
16	+ 0,040	+ 0,029	+ 0,034	+ 0,027	- 0,090	+ 0,013	- 0,006	+ 0,003	- 0,004	- 0,005	+ 0,004	0,037	0,35
17	+ 0,019	+ 0,006	+ 0,020		- 0,002	- 0,007	- 0,023	- 0,031	+ 0,002	- 0,000	- 0,002	0,017	0,30
18	- 0,014	+ 0,010	- 0,038		+ 0,013	- 0,066	- 0,022	- 0,046	+ 0,020	+ 0,022	- 0,013	0,032	1,25
19	+ 0,020	+ 0,054	+ 0,044	+ 0,097	- 0,025	+ 0,168	+ 0,059	+ 0,078	+ 0,026	+ 0,075	+ 0,060	0,051	3,67
d	+ 0,003	- 0,001	+ 0,000	- 0,001	- 0,003	+ 0,001	- 0,003	- 0,001	+ 0,002	- 0,001	+ 0,002	0,025	
Sd	0,020	0,021	0,021	0,013	0,012	0,013	0,016	0,018	0,012	0,010	0,016		

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

Upper limits : $\bar{d} = \pm 0,02 \text{ g / 100 g}$ Sd = 0,03 g / 100g**ISO 1211 | IDF 1 : Precision of the method :**

Sr = 0.016 g / 100 g

SR = 0.020 g / 100 g

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

Sample/Lab code	1	2	3	4	5	6	7	8	9	10
1	-0,04	+0,29	-0,13	+0,07	+0,42	+0,07	-0,00	+0,55	-1,31	+0,20
2	-0,22	+0,19	+0,08	+0,19	-0,07	-1,24	+0,64	-0,06	-0,18	-0,20
3	+0,19	+0,19	+0,42	-0,16	+0,66	+0,11	+0,16	+0,41	+0,88	+0,20
4	-0,27	-0,26	+0,20	-0,61	-1,61	-0,17	+0,25	+0,71	-0,30	+1,00
5	+2,26	-1,45	+0,20	-0,61	-1,61	+1,43	-0,39	+0,99	+0,92	-1,50
6	-0,82	-1,24	-0,49	-0,46	-1,65	-0,69	+0,09	-0,09	-0,62	-1,90
7	-1,86	-1,41	-1,20	-1,57	+0,74	+1,07	+0,96	-1,34	+0,80	+0,85
8	+0,39	-0,36	-0,56	-1,84	-0,84	-0,25	-1,52	-0,20	-0,42	-0,55
9	-0,75	-0,10	-0,18	+0,19	-0,23	-0,13	-0,13	+0,71	+0,27	-0,55
10	+0,52	+0,14	+0,27	+0,91	+0,50	+1,15	+0,77	+0,02	-0,30	-1,60
11	+0,10	-0,38	-0,14	+0,85	+0,52	+4,77	-0,02	+0,64	+0,38	+0,13
12	+0,49	+0,19	+0,39	+0,57	+0,54	+0,7	+0,9	+0,85	+0,4	+0,05
13	+0,21	+0,12	+0,28	+0,65	+0,13	+0,67	+0,87	+0,91	+0,43	-0,05
14	-0,60	-1,64	-1,77	-1,26	-3,97	-2,28	-2,48	-1,09	-2,16	
15		+0,21		+0,15				+0,16		-0,00
16	+2,01	+1,41	+1,63	+2,06	-7,30	+1,03	-0,39	+0,16	-0,30	-0,50
17	+0,97	+0,31	+0,94		-0,15	-0,53	-1,49	-1,73	+0,15	-0,00
18	-0,70	+0,50	-1,82		+1,02	-5,27	-1,39	-2,56	+1,65	+2,26
19	+1,00	+2,60	+2,10	+7,40	-2,02	+13,38	+3,80	+4,32	+2,13	+7,52

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

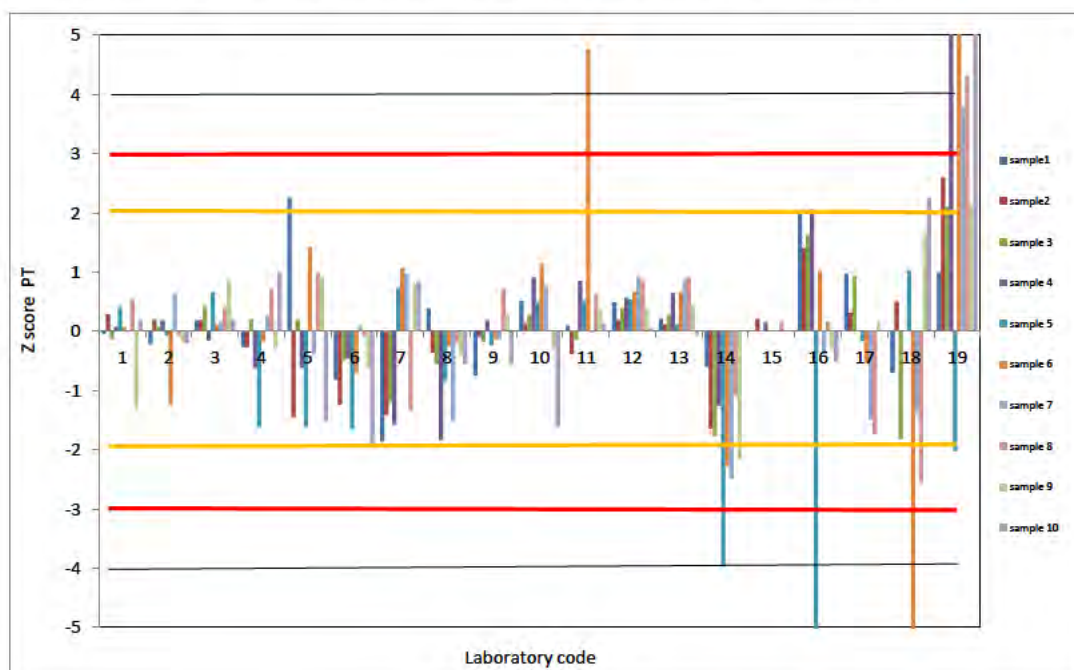


Table VII : Zscore of the different laboratories for each sample.
ZS calculated on the standard deviation of reproducibility of the method

Sample/Lab code	1	2	3	4	5	6	7	8	9	10
1	-0,04	+0,30	-0,14	+0,05	+0,26	+0,04	-0,00	+0,49	-0,81	+0,10
2	-0,21	+0,20	+0,09	+0,12	-0,04	-0,78	+0,50	-0,06	-0,11	-0,10
3	+0,19	+0,20	+0,44	-0,10	+0,41	+0,07	+0,12	+0,37	+0,54	+0,10
4	-0,26	-0,28	+0,21	-0,40	-0,99	-0,11	+0,20	+0,64	-0,18	+0,50
5	+2,24	-1,53	+0,21	-0,40	-0,99	+0,89	-0,30	+0,89	+0,57	-0,75
6	-0,81	-1,30	-0,51	-0,30	-1,02	-0,43	+0,07	-0,08	-0,38	-0,95
7	-1,84	-1,48	-1,26	-1,03	+0,46	+0,67	+0,75	-1,21	+0,49	+0,42
8	+0,39	-0,38	-0,59	-1,20	-0,52	-0,16	-1,18	-0,18	-0,26	-0,28
9	-0,74	-0,10	-0,19	+0,12	-0,14	-0,08	-0,10	+0,64	+0,17	-0,28
10	+0,51	+0,15	+0,29	+0,60	+0,31	+0,72	+0,60	+0,02	-0,18	-0,80
11	+0,09	-0,40	-0,15	+0,56	+0,32	+2,99	-0,01	+0,57	+0,23	+0,07
12	+0,49	+0,20	+0,41	+0,37	+0,33	+0,42	+0,7	+0,77	+0,24	+0,02
13	+0,21	+0,13	+0,29	+0,43	+0,08	+0,42	+0,67	+0,82	+0,27	-0,03
14	-0,59	-1,73	-1,86	-0,83	-2,44	-1,43	-1,93	-0,98	-1,33	
15		+0,22		+0,10				+0,14		-0,00
16	+1,99	+1,47	+1,71	+1,35	-4,49	+0,64	-0,30	+0,14	-0,18	-0,25
17	+0,96	+0,32	+0,99		-0,09	-0,33	-1,15	-1,56	+0,09	-0,00
18	-0,69	+0,52	-1,91		+0,63	-3,31	-1,08	-2,31	+1,02	+1,12
19	+0,99	+2,72	+2,21	+4,85	-1,24	+8,39	+2,95	+3,89	+1,32	+3,75

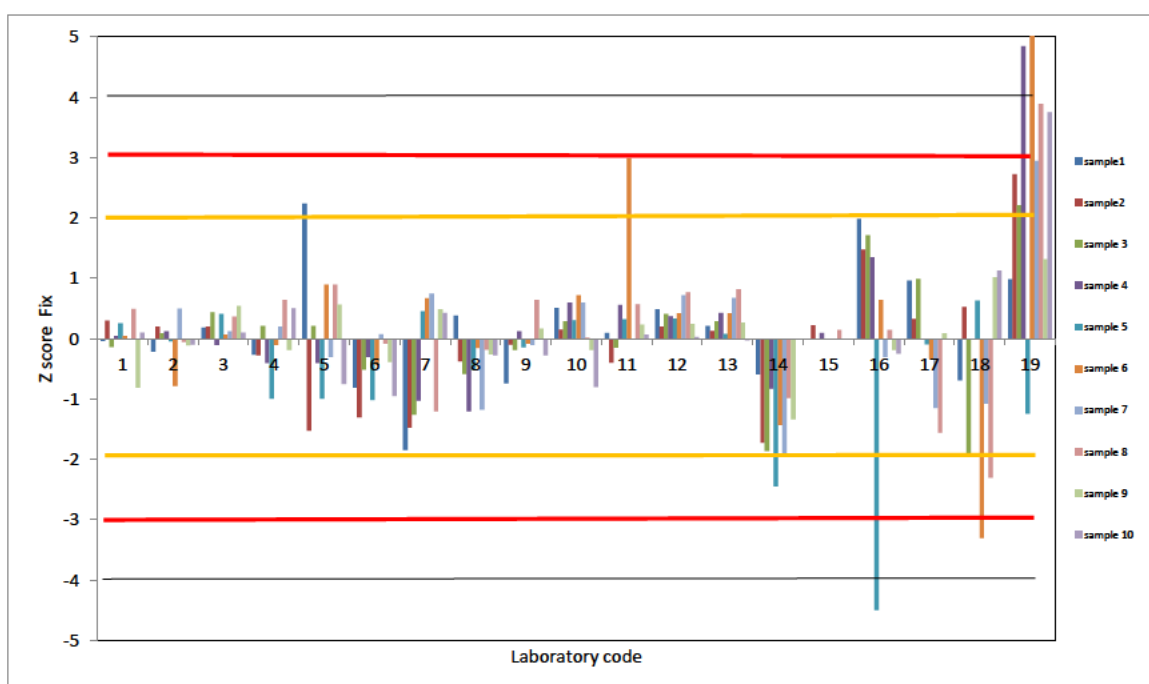
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=0,02

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

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

Figure 3 :

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



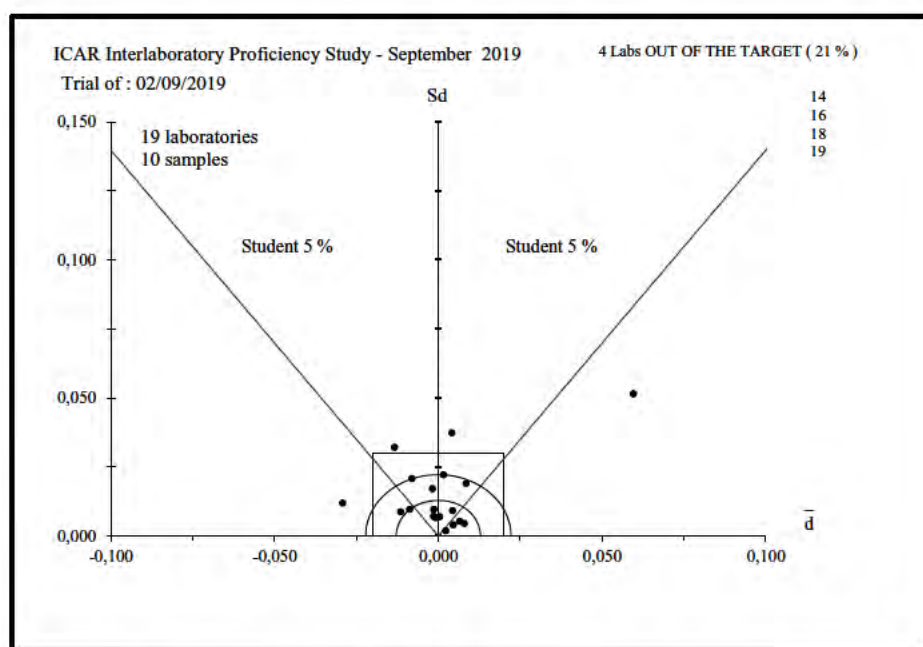


Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).

**LIST OF THE PARTICIPANTS ICAR
ICAR PROFICIENCY TEST
RAW MILK
FAT CONTENT Röse Göttlieb
March 2019**

Name	City	Country
Actalia	Poligny	France
Agroscope Institute for food Sciences IFS	Bern-Liebefeld	Switzerland
Central Milk Lab ICBA	Caesarea	Israel
Deltamune	Centurion 0140 Pretoria	South Africa
Department Valorisation des productions Agricoles	Gembloux	Belgium
EPTA Laboratories LTD	Agios Dometios- Nicosia	Cyprus
Int. National de tecnologia Industrial (INTI)	Rafaela Santa Fe	Argentina
Japan Dairy Technical Association	Tokyo	Japan
Lab Agroalimentario de Santander	Santander Cantabria	Spain
Laboratorio Standard Latte	Maccarese (Roma)	Italy
KCHZ Laboratorium Referencyjne z/s w Parzniewie	Pruszkow	Poland
Milchprüfing Baden-Württemberg e.V.	Kirchheim unter Teck	Germany
Pieno Tyrimai	Kaunas	Lithuania
Qlip B.V.,	Zutphen	NL
Teagasc Food research Center	Cork	IR
Univ. of Ljubljana	Domzale	Slovenia
Lactanet-Québec	Quebec	Canada
Valio Ltd, Regional laboratory	Seinäjoki	Finland



**ICAR
PROFICIENCY TESTING SCHEME**

September 2019

Raw Milk

**Determination of CRUDE PROTEIN CONTENT
KJELDAHL Method**

Sending date of statistical treatment : 3th October 2019

Frame of activity :	ICAR Milk Analyses Sub Committee (MA SC)
ICAR Staff	Silvia Orlandini pt@icar.org silvia@icar.org

Proficiency test accredited ISO 17043



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

Nb	%	N°	d	Sd	D
1	5	2	- 0,002	0,005	0,005
2	11	1	+ 0,003	0,007	0,008
3	16	12	- 0,007	0,006	0,009
4	21	17	+ 0,002	0,010	0,010
5	26	6	+ 0,001	0,010	0,010
6	32	8	- 0,010	0,007	0,012
7	37	9	+ 0,009	0,008	0,012
8	42	7	+ 0,008	0,011	0,013
9	47	18	+ 0,013	0,005	0,014
10	53	5	- 0,014	0,008	0,016
11	58	15	+ 0,016	0,006	0,017
12	63	16	- 0,014	0,011	0,017
13	68	11	- 0,015	0,009	0,018
14	74	19	+ 0,017	0,007	0,018
15	79	14	+ 0,018	0,008	0,019
16	84	10	+ 0,004	0,020	0,021
17	89	13	+ 0,021	0,015	0,026
18	95	3	- 0,030	0,005	0,030
19	100	4	- 0,041	0,020	0,046

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

$$\pm 0,025 \text{ g / 100 g for } \bar{d} \text{ and } 0,020 \text{ g / 100 g for Sd}$$

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 19 laboratories using the reference method (ISO 8968-1|IDF 20-1), after outlier discarding using Grubbs test at 5% risk level

N.B. : N° 10 and N° 12 : ISO 8968-3|IDF 20-3

(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.(d² + Sd²))

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} 0,008

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

SR_{PT} 0,018

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

Sample Lab code	1	2	3	4	5	6	7	8	9	10	Sr	NL
1	0,001	0,000	0,000	0,013	0,007	0,010	0,001	0,007	0,015	0,023	0,008	20
2	0,006	0,000	0,000	0,000	0,000	0,000	0,013	0,006	0,000	0,000	0,003	20
3	0,001	0,004	0,001	0,003	0,000	0,012	0,006	0,006	0,012	0,001	0,004	20
4	0,027	0,005	**	0,007	0,020	**	0,002	0,003	**	0,006	0,009	14
5	0,003	0,003	0,004	0,020	0,011	0,009	0,004	0,017	0,006	0,001	0,007	20
6	0,004	0,034 *	0,017	0,015	0,013	0,010	0,015	0,004	0,001	0,001	0,011	20
7	0,022	0,001	0,020	0,010	0,021	0,011	0,001	0,028	0,010	0,006	0,011	20
8	0,001	0,006	0,004	0,005	0,003	0,004	0,004	0,004	0,001	0,001	0,003	20
9	0,005	0,002	0,009	0,003	0,004	0,003	0,005	0,006	0,006	0,006	0,004	20
10	0,003	0,001	0,003	0,013	0,004	0,006	0,019	0,006	0,004	0,002	0,006	20
11	0,004	0,013	0,008	0,006	0,000	0,003	0,028	0,009	0,029	0,013	0,010	20
12	0,013	0,013	0,006	0,013	0,013	0,006	0,000	0,013	0,019	0,013	0,008	20
13	0,008	0,080 *	0,022	0,006	0,054 *	0,031 *	0,028	0,001	0,007	0,004	0,024	20
14	0,004	0,003	0,000	0,020	0,002	0,016	0,017	0,005	0,015	0,004	0,008	20
15	**	0,007	**	0,013	**	**	**	0,013	**	0,003	0,007	8
16	0,013	0,006	0,004	0,007	0,004	0,004	0,013	0,003	0,020	0,012	0,007	20
17	0,030	0,000	0,010	0,010	0,000	0,010	0,010	0,010	0,010	0,010	0,009	20
18	0,020	0,003	0,008	0,005	0,008	0,003	0,004	0,011	0,000	0,000	0,006	20
19	0,031	0,015	0,001	0,001	0,001	0,004	0,004	0,007	0,000	0,013	0,008	20
Sr	0,011	0,015	0,007	0,007	0,011	0,008	0,009	0,007	0,009	0,006		362
NE	36	38	34	38	36	34	36	38	34	38		
L	0 041	0 018	0 026	0 029	0 026	0 022	0 036	0 029	0 033	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 between two replicates=0,040 according ISO 8968-1 | IDF 20-1

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

Sample Lab code	1	2	3	4	5	6	7	8	9	10
1	3,799	3,567	3,314	2,935	2,739	3,690	3,505	3,121	2,837	3,314
2	3,780	3,573	3,292	2,935	2,737	3,675	3,496	3,117	2,839	3,324
3	3,752	3,540	3,277	2,908	2,712	3,653	3,462	3,095	2,807	3,283
4	3,716 *	3,529	3,287	2,908	2,708	3,658	3,446	3,097	2,802	3,246 *
5	3,765	3,560	3,282	2,936	2,712	3,674	3,482	3,113	2,820	3,305
6	3,799	3,565	3,294	2,926	2,741	3,679	3,498	3,134	2,845	3,320
7	3,779	3,572	3,316	2,954	2,753	3,710	3,504	3,119	2,833	3,327
8	3,769	3,579	3,287	2,925	2,730	3,669	3,482	3,113	2,823	3,314
9	3,785	3,589	3,314	2,945	2,741	3,685	3,503	3,120	2,857	3,339
10	3,790	3,577	3,305	2,905	2,730	3,681	3,545	3,121	2,845	3,327
11	3,770	3,558	3,293	2,939	2,725	3,669	3,460	3,100	2,819	3,303
12	3,783	3,566	3,308	2,935	2,737	3,672	3,483	3,107	2,817	3,311
13	3,796	3,580	3,344	2,964	2,741	3,698	3,548	3,136	2,858	3,335
14	3,805	3,597	3,332	2,961	2,760	3,703	3,502	3,133	2,836	3,334
15		3,592		2,954				3,142		3,329
16	3,772	3,578	3,282	2,927	2,733	3,645	3,478	3,115	2,816	3,306
17	3,805	3,570	3,305	2,935	2,740	3,675	3,515	3,115	2,835	3,315
18	3,788	3,587	3,313	2,948	2,750	3,697	3,506	3,133	2,854	3,341
19	3,791	3,587	3,327	2,959	2,757	3,712	3,509	3,135	2,841	3,338
M	3,784	3,572	3,305	2,937	2,736	3,680	3,496	3,119	2,832	3,320
REF.	3,786	3,573	3,304	2,939	2,737	3,680	3,496	3,120	2,832	3,321
SD	0,015	0,017	0,019	0,018	0,015	0,019	0,026	0,014	0,017	0,015

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 19 laboratories using the reference method ISO 8968-1 | IDF 20-1, after outliers discarding using Grubbs test at 5 % risk level.

Table IV : Outlier identification

Sample	1	2	3	4	5	6	7	8	9	10
Outliers Cochran		6;13			13	13				
Outlier Grubbs	4									4
sr	0,010	0,005	0,007	0,007	0,007	0,006	0,009	0,007	0,009	0,006
SR	0,016	0,018	0,019	0,019	0,016	0,019	0,027	0,015	0,016	0,016

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	+ 0,013	- 0,006	+ 0,010	- 0,003	+ 0,001	+ 0,010	+ 0,010	+ 0,001	+ 0,004	- 0,007	+ 0,003	0,007	1,45
2	- 0,005	- 0,000	- 0,012	- 0,004	- 0,000	- 0,005	+ 0,000	- 0,003	+ 0,007	+ 0,003	- 0,002	0,005	1,26
3	- 0,033	- 0,033	- 0,027	- 0,031	- 0,025	- 0,027	- 0,034	- 0,025	- 0,026	- 0,038	- 0,030	0,005	20,78
4	- 0,069	- 0,044	- 0,030	- 0,029	- 0,022	- 0,050	- 0,023	- 0,030	- 0,075	- 0,041	0,020	6,30	
5	- 0,021	- 0,013	- 0,022	- 0,003	- 0,026	- 0,006	- 0,014	- 0,006	- 0,012	- 0,016	- 0,014	0,008	5,76
6	+ 0,014	- 0,008	- 0,010	- 0,012	+ 0,003	- 0,002	+ 0,002	+ 0,014	+ 0,013	- 0,001	+ 0,001	0,010	0,42
7	- 0,006	+ 0,001	+ 0,012	+ 0,016	+ 0,016	+ 0,029	+ 0,008	- 0,001	+ 0,001	+ 0,005	+ 0,008	0,011	2,33
8	- 0,017	+ 0,006	- 0,017	- 0,014	- 0,008	- 0,011	- 0,014	- 0,006	- 0,010	- 0,007	- 0,010	0,007	4,61
9	- 0,000	+ 0,016	+ 0,010	+ 0,006	+ 0,003	+ 0,005	+ 0,007	+ 0,001	+ 0,025	+ 0,018	+ 0,009	0,008	3,56
10	+ 0,004	+ 0,004	+ 0,001	- 0,034	- 0,007	+ 0,001	+ 0,049	+ 0,002	+ 0,012	+ 0,006	+ 0,004	0,020	0,60
11	- 0,015	- 0,015	- 0,011	+ 0,000	- 0,012	- 0,011	- 0,036	- 0,020	- 0,014	- 0,018	- 0,015	0,009	5,20
12	- 0,002	- 0,007	+ 0,004	- 0,004	- 0,000	- 0,009	- 0,012	- 0,013	- 0,016	- 0,010	- 0,007	0,006	3,49
13	+ 0,011	+ 0,007	+ 0,040	+ 0,026	+ 0,003	+ 0,017	+ 0,052	+ 0,016	+ 0,026	+ 0,014	+ 0,021	0,015	4,43
14	+ 0,020	+ 0,024	+ 0,028	+ 0,023	+ 0,022	+ 0,022	+ 0,006	+ 0,014	+ 0,004	+ 0,013	+ 0,018	0,008	6,94
15	+ 0,019	+ 0,019	+ 0,016	+ 0,016	+ 0,016	+ 0,016	+ 0,022	+ 0,022	+ 0,008	+ 0,016	+ 0,006	5,44	
16	- 0,014	+ 0,005	- 0,022	- 0,012	- 0,005	- 0,035	- 0,018	- 0,005	- 0,016	- 0,015	- 0,014	0,011	3,99
17	+ 0,019	- 0,003	+ 0,001	- 0,004	+ 0,003	- 0,005	+ 0,019	- 0,005	+ 0,003	- 0,006	+ 0,002	0,010	0,73
18	+ 0,003	+ 0,014	+ 0,009	+ 0,009	+ 0,013	+ 0,017	+ 0,010	+ 0,013	+ 0,021	+ 0,019	+ 0,013	0,005	7,39
19	+ 0,005	+ 0,014	+ 0,023	+ 0,021	+ 0,020	+ 0,031	+ 0,013	+ 0,016	+ 0,009	+ 0,016	+ 0,017	0,007	7,28
d	- 0,001	- 0,001	+ 0,001	- 0,002	- 0,002	+ 0,000	- 0,000	- 0,000	- 0,000	- 0,001	- 0,001	0,019	
Sd	0,015	0,017	0,019	0,018	0,015	0,019	0,026	0,014	0,017	0,015	0,018		

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

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

ISO 8968-1 | IDF 20-1 : Precision of the method : Sr = 0.014 g / 100 g
SR = 0.018 g / 100 g

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

Sample Lab code	1	2	3	4	5	6	7	8	9	10
1	+0,88	-0,36	+0,52	-0,18	+0,08	+0,53	+0,36	+0,09	+0,25	-0,45
2	-0,36	-0,02	-0,63	-0,21	-0,03	-0,29	+0,01	-0,22	+0,40	+0,18
3	-2,22	-1,93	-1,41	-1,74	-1,70	-1,47	-1,28	-1,80	-1,54	-2,49
4	-4,63	-2,58		-1,70	-1,96	-1,18	-1,91	-1,66	-1,82	-4,96
5	-1,39	-0,76	-1,19	-0,14	-1,74	-0,32	-0,53	-0,45	-0,73	-1,04
6	+0,92	-0,45	-0,51	-0,70	+0,21	-0,08	+0,09	+1,02	+0,77	-0,09
7	-0,43	-0,04	+0,62	+0,88	+1,07	+1,57	+0,29	-0,08	+0,04	+0,35
8	-1,11	+0,33	-0,92	-0,79	-0,52	-0,60	-0,54	-0,45	-0,59	-0,45
9	-0,02	+0,93	+0,55	+0,36	+0,23	+0,28	+0,25	+0,04	+1,48	+1,17
10	+0,30	+0,22	+0,03	-1,90	-0,50	+0,05	+1,86	+0,13	+0,75	+0,41
11	-1,01	-0,86	-0,57	+0,00	-0,81	-0,58	-1,37	-1,46	-0,81	-1,19
12	-0,15	-0,39	+0,21	-0,21	-0,03	-0,46	-0,47	-0,92	-0,94	-0,66
13	+0,73	+0,42	+2,14	+1,44	+0,23	+0,92	+1,97	+1,18	+1,54	+0,92
14	+1,33	+1,41	+1,47	+1,28	+1,50	+1,20	+0,24	+1,00	+0,23	+0,83
15		+1,11		+0,88				+1,60		+0,54
16	-0,93	+0,27	-1,18	-0,65	-0,33	-1,88	-0,66	-0,34	-0,98	-1,00
17	+1,30	-0,18	+0,05	-0,20	+0,17	-0,28	+0,72	-0,34	+0,16	-0,41
18	+0,19	+0,80	+0,47	+0,50	+0,88	+0,91	+0,38	+0,97	+1,29	+1,27
19	+0,36	+0,83	+1,20	+1,17	+1,35	+1,68	+0,51	+1,16	+0,52	+1,08

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	+0,73	-0,34	+0,54	-0,17	+0,07	+0,55	+0,53	+0,07	+0,23	-0,38
2	-0,30	-0,02	-0,66	-0,21	-0,02	-0,30	+0,02	-0,16	+0,37	+0,15
3	-1,84	-1,85	-1,48	-1,72	-1,40	-1,52	-1,88	-1,37	-1,42	-2,10
4	-3,84	-2,47		-1,68	-1,62	-1,22	-2,80	-1,26	-1,68	-4,17
5	-1,15	-0,73	-1,25	-0,14	-1,44	-0,33	-0,78	-0,34	-0,67	-0,88
6	+0,76	-0,43	-0,54	-0,69	+0,17	-0,09	+0,13	+0,77	+0,71	-0,08
7	-0,35	-0,04	+0,65	+0,87	+0,88	+1,63	+0,43	-0,06	+0,04	+0,29
8	-0,92	+0,32	-0,96	-0,78	-0,43	-0,62	-0,80	-0,34	-0,55	-0,38
9	-0,02	+0,88	+0,58	+0,36	+0,19	+0,29	+0,37	+0,03	+1,36	+0,98
10	+0,25	+0,21	+0,03	-1,88	-0,41	+0,06	+2,73	+0,10	+0,69	+0,35
11	-0,84	-0,82	-0,59	+0,00	-0,67	-0,60	-2,02	-1,11	-0,75	-1,00
12	-0,12	-0,37	+0,22	-0,21	-0,02	-0,48	-0,69	-0,70	-0,87	-0,56
13	+0,60	+0,41	+2,24	+1,42	+0,19	+0,96	+2,89	+0,90	+1,42	+0,77
14	+1,10	+1,34	+1,54	+1,26	+1,24	+1,24	+0,36	+0,76	+0,21	+0,70
15		+1,06		+0,87				+1,22		+0,45
16	-0,77	+0,26	-1,24	-0,64	-0,27	-1,95	-0,97	-0,26	-0,90	-0,84
17	+1,08	-0,18	+0,06	-0,20	+0,14	-0,29	+1,06	-0,26	+0,14	-0,35
18	+0,16	+0,76	+0,49	+0,50	+0,72	+0,94	+0,55	+0,74	+1,19	+1,07
19	+0,30	+0,79	+1,25	+1,15	+1,11	+1,74	+0,75	+0,88	+0,48	+0,91

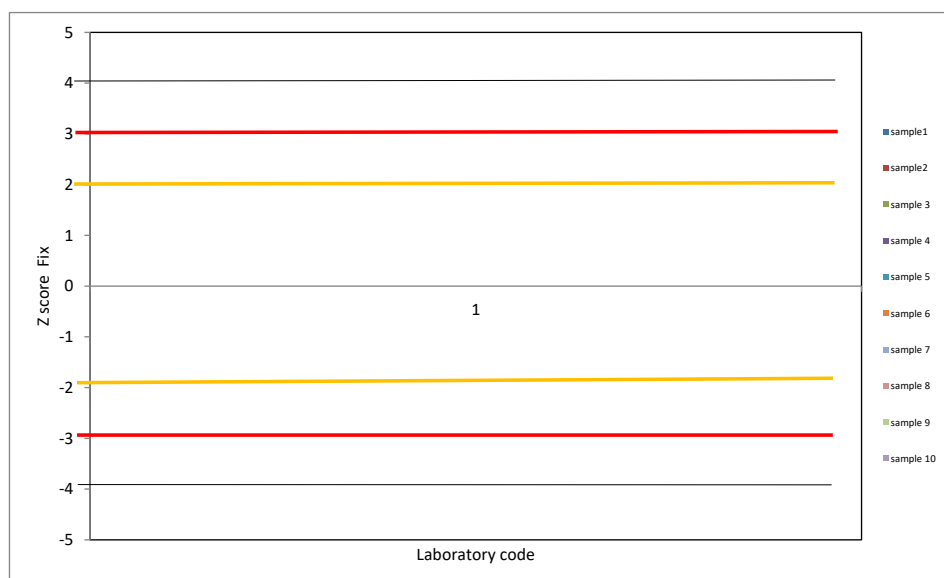
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=0,018

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

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

Figure 3 :

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



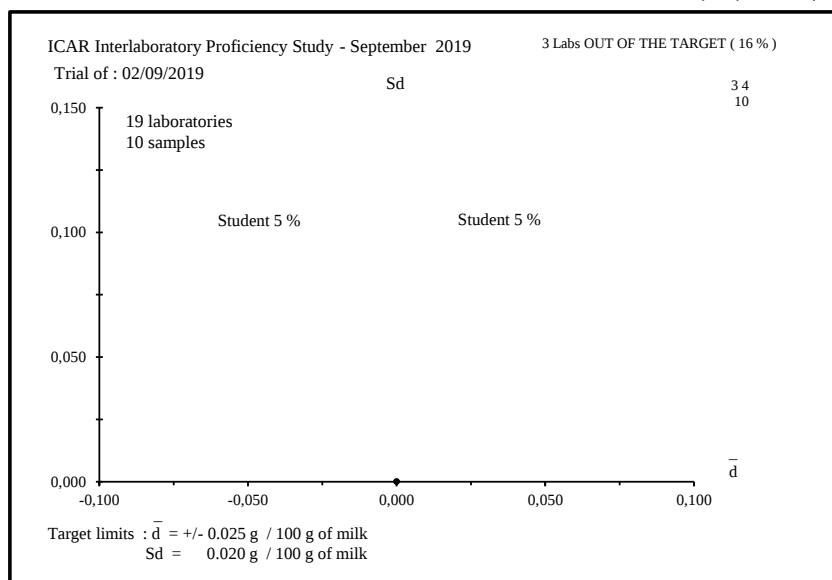


Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).

Table VIII : Relative recovery of nitrogen on pure solutions

N°	TRYP	GLY	SO4
1	100,0	99,9	100,0
2	98,3	99,9	100,3
3	98,9	99,8	99,9
4		99,2	98,9
5	99,4	99,9	99,8
6	99,9	100,4	100,4
7	99,6	100,1	100,4
8	99,6	99,4	99,5
9	98,6	98,5	99,8
10	99,7	99,9	100,3
11	96,0	99,3	102,2
12	99,5	100,6	100,7
13	100,0	100,2	100,8
14	99,9	100,1	100,3
15			
16			
17			
18	100,8	101,1	100,5
19	97,9	100,4	100,6

TRY = Tryptophan solution to 5,60 g N/l

GLY = Glycine solution to 5.60 g N/l

SO4 = Ammonium sulfate solution to 5.60 g N/l

TRYP : recovery 97 à 101 %

GLY : recovery 99 à 101 %

SO4 : recovery 99 à 101 %

**LIST OF THE PARTICIPANTS ICAR
ICAR PROFICIENCY TEST
RAW MILK
PROTEIN CONTENT KJELDAHL
Sep 19**

Name

Actalia
Agroscope Institute for food Sciences IFS
Central Milk Lab ICBA
Deltamune
Department Valorisation des productions Agricoles
Int. National de tecnologia Industrial (INTI)
Japan Dairy Technical Association
Lab Agroalimentario de Santander
Laboratorio Standard Latte
Laboratorium Oceny Mleka KCHZ Laboratorium Referecyjne z/s w Parzniewie
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Quebec
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Kaunas
Zutphen
Cork
Domzale
Quebec

Country

France
Switzerland
Israel
South Africa
Belgium
Argentina
Japan
Spain
Italy
Poland
Canada
Germany
Czech Republic

Lithuania
NL
IR
Slovenia
Canada



ICAR
PROFICIENCY TESTING SCHEME

September 2019

Raw Milk

Determination of LACTOSE CONTENT

Sending date of statistical treatment : 3th October 2019

Frame of activity :	ICAR Milk Analyses Sub Committee (MA SC)		
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Table I : **Ranking of the laboratories** Units : g / 100 g

Nb	%	N°	d	Sd	D	Method
1	7	1	- 0,026	0,009	0,028	In-house Method
2	14	5	+ 0,024	0,017	0,029	ISO 22662 / IDF 198
3	21	2	- 0,023	0,021	0,031	Polarimeter
4	29	6	- 0,044	0,018	0,047	ISO 22662 / IDF 198
5	36	7	- 0,052	0,019	0,055	ISO 26462/IDF 214
6	43	4	+ 0,030	0,050	0,058	ISO 26462/IDF 214
7	50	10	- 0,009	0,065	0,066	continuous flow analysis
8	57	3	+ 0,065	0,025	0,070	Enzymatic method
9	64	11	+ 0,082	0,022	0,085	ISO 22662 / IDF 198
10	71	9	+ 0,098	0,034	0,103	Lane-Eynon
11	79	8	- 0,132	0,020	0,134	ISO 26462/IDF 214
12	86	12	+ 0,166	0,033	0,170	Enzymatic in house
13	93	13	- 0,173	0,025	0,175	ISO 26462/IDF 214
14	100	14	- 0,140	0,271	0,305	In-house HPLC RID

(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.(d² + Sd²))

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

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

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 14 laboratories , after outliers discarding using Grubbs test at 5 % risk level.

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} 0,012

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	11	12	13	14	15	16	17	18	19	20	Sr	NL
1	0,007	0,007	0,008	0,007	0,004	0,007	0,009	0,007	0,008	0,008	0,005	20
2	0,000	0,000	0,000	0,000	0,000	0,000	0,010	0,000	0,000	0,010	0,003	20
3	0,028	0,018	0,016	0,028	0,017	0,034	0,000	0,031	0,016	0,028	0,017	20
4	0,009	0,005	0,000	0,005	0,011	0,004	0,002	0,010	0,011	0,009	0,005	20
5	0,004	0,016	0,018	0,023	0,017	0,000	0,015	0,002	0,010	0,036	0,012	20
6	0,001	0,001	0,002	0,009	0,005	0,013	0,007	0,025	0,033	0,006	0,010	20
7	0,065 *	0,075 *	0,025	0,053	0,005	0,002	0,044 *	0,006	0,036	0,000	0,029	20
8	0,017	0,010	0,001	0,009	0,001	0,017	0,013	0,009	0,005	0,014	0,008	20
9	0,027	0,002	0,000	0,010	0,028	0,021	0,015	0,013	0,011	0,011	0,012	20
10	0,000	0,010	0,072 *	0,020	0,000	0,010	0,010	0,143 *	0,031	0,000	0,037	20
11	0,003	0,010	0,008	0,012	0,008	0,004	0,009	0,030	0,001	0,018	0,009	20
12	0,027	0,038	0,002	0,003	0,013	0,026	0,026	0,016	0,015	0,047	0,018	20
13	0,004	0,018	0,019	0,023	0,033	0,014	0,002	0,031	0,028	0,016	0,015	20
14	**	**	**	**	**	**	**	**	**	**		
Sr	0,016	0,018	0,016	0,015	0,011	0,011	0,012	0,031	0,014	0,014		260
NE	26	26	26	26	26	26	26	26	26	26		
L	0,038	0,039	0,030	0,054	0,038	0,040	0,030	0,047	0,051	0,053		

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

NL : number of measurements per laboratory

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

SE : repeatability standard deviation per sample

NE : number of measurements per sample

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

** : missing data

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

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

Sample Lab code	11	12	13	14	15	16	17	18	19	20	LACT
1	5,183	5,126	5,050	4,974	4,902	4,862	4,839	4,806	4,773	4,751	4,906
2	5,200	5,130	5,050	4,990	4,920	4,880	4,845	4,800	4,760	4,715	4,910
3	5,273	5,199	5,147	5,036	5,000	4,945	4,922	4,942	4,896	4,817	4,936
4	5,177	5,203	5,146	5,084	5,030	4,861	4,932	4,858	4,818	4,716	5,167
5	5,225	5,160	5,086	5,030	4,933	4,925	4,905	4,872	4,834	4,793	4,926
6	5,174	5,113	5,036	4,966	4,891	4,860	4,827	4,772	4,728	4,723	4,931
7	5,161	5,094	5,017	4,929	4,867	4,808	4,834	4,813	4,774	4,712	4,929
8	5,079	5,007	4,938	4,874	4,806	4,788	4,742	4,687	4,664	4,621	4,924
9	5,289	5,236	5,155	5,128	5,079	5,006	4,973	4,959	4,851	4,828	4,943
10	5,156	5,079	5,048	4,951	4,869	4,813	4,905	4,869	4,833	4,910	4,879
11	5,280	5,196	5,142	5,079	5,025	5,003	4,961	4,903	4,895	4,864	4,971
12	5,374	5,282	5,244	5,161	5,107	5,103	5,033	5,039	4,946	4,902	5,005
13	5,047	5,006	4,946	4,829	4,768	4,702	4,681	4,628	4,600	4,590	4,787
14		5,230		4,520 *		4,560		4,670		4,950	4,420
M	5,201	5,147	5,077	5,002	4,938	4,865	4,877	4,830	4,798	4,778	
REF.	5,199	5,148	5,076	5,003	4,938	4,871	4,880	4,829	4,801	4,779	4,912
SD	0,088	0,083	0,088	0,097	0,103	0,135	0,096	0,116	0,096	0,107	

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 1352 of 14 laboratories, after outliers discarding using Grubbs test at 5 % risk level.

Table IV : Outlier identification

Sample	11	12	13	14	15	16	17	18	19	20	
Outliers Cochran	7	7	10				7	10			
Outlier Grubbs				14							
sr	0,011	0,011	0,008	0,015	0,011	0,011	0,008	0,013	0,014	0,014	
SR	0,092	0,086	0,091	0,097	0,104	0,107	0,100	0,116	0,097	0,100	

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

Sample Lab code	11	12	13	14	15	16	17	18	19	20	d	Sd _{lab}	t
1	- 0,017	- 0,022	- 0,026	- 0,029	- 0,036	- 0,009	- 0,042	- 0,024	- 0,028	- 0,028	- 0,026	0,009	9,07
2	+ 0,001	- 0,018	- 0,026	- 0,013	- 0,018	+ 0,009	- 0,035	- 0,029	- 0,041	- 0,064	- 0,023	0,021	3,54
3	+ 0,074	+ 0,051	+ 0,071	+ 0,033	+ 0,062	+ 0,074	+ 0,042	+ 0,112	+ 0,095	+ 0,038	+ 0,065	0,025	8,15
4	- 0,023	+ 0,055	+ 0,070	+ 0,081	+ 0,092	- 0,010	+ 0,052	+ 0,029	+ 0,016	- 0,063	+ 0,030	0,050	1,89
5	+ 0,026	+ 0,012	+ 0,010	+ 0,027	- 0,005	+ 0,054	+ 0,024	+ 0,043	+ 0,033	+ 0,014	+ 0,024	0,017	4,38
6	- 0,026	- 0,035	- 0,040	- 0,037	- 0,047	- 0,011	- 0,054	- 0,058	- 0,074	- 0,056	- 0,044	0,018	7,77
7	- 0,039	- 0,054	- 0,060	- 0,074	- 0,071	- 0,063	- 0,046	- 0,016	- 0,027	- 0,067	- 0,052	0,019	8,47
8	- 0,121	- 0,141	- 0,139	- 0,129	- 0,132	- 0,083	- 0,139	- 0,143	- 0,138	- 0,158	- 0,132	0,020	21,29
9	+ 0,089	+ 0,088	+ 0,079	+ 0,125	+ 0,141	+ 0,135	+ 0,092	+ 0,129	+ 0,049	+ 0,049	+ 0,098	0,034	9,13
10	- 0,044	- 0,069	- 0,028	- 0,052	- 0,069	- 0,058	+ 0,025	+ 0,040	+ 0,032	+ 0,131	- 0,009	0,065	0,44
11	+ 0,080	+ 0,048	+ 0,066	+ 0,076	+ 0,087	+ 0,132	+ 0,080	+ 0,074	+ 0,093	+ 0,085	+ 0,082	0,022	12,07
12	+ 0,174	+ 0,134	+ 0,168	+ 0,158	+ 0,169	+ 0,232	+ 0,153	+ 0,210	+ 0,144	+ 0,123	+ 0,166	0,033	15,82
13	- 0,153	- 0,141	- 0,131	- 0,174	- 0,170	- 0,169	- 0,200	- 0,201	- 0,202	- 0,188	- 0,173	0,025	21,51
14		+ 0,082		- 0,483		- 0,311		- 0,159		+ 0,171	- 0,140	0,271	1,15
d	+ 0,002	- 0,000	+ 0,001	- 0,001	- 0,000	- 0,006	- 0,004	+ 0,001	- 0,004	- 0,001	- 0,005	0,107	
Sd	0,088	0,083	0,088	0,097	0,103	0,135	0,096	0,116	0,096	0,107	0,102		

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

Upper limits : d = +/- 0.100 g / 100g Sd = 0.100 g / 100g

- 8, **ISO 22662 | IDF 198 : Precision of the method :** Sr = 0.022 g / 100 g
SR = 0.047 g / 100 g

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

Sample Lab code	11	12	13	14	15	16	17	18	19	20
1	-0,19	-0,26	-0,30	-0,30	-0,35	-0,07	-0,43	-0,20	-0,30	-0,26
2	+0,01	-0,21	-0,30	-0,13	-0,17	+0,07	-0,37	-0,25	-0,43	-0,59
3	+0,83	+0,62	+0,81	+0,34	+0,60	+0,55	+0,44	+0,97	+0,98	+0,36
4	-0,26	+0,66	+0,80	+0,83	+0,89	-0,07	+0,54	+0,25	+0,17	-0,59
5	+0,29	+0,15	+0,11	+0,27	-0,05	+0,40	+0,25	+0,37	+0,34	+0,13
6	-0,29	-0,42	-0,46	-0,39	-0,46	-0,08	-0,56	-0,50	-0,77	-0,52
7	-0,44	-0,65	-0,68	-0,77	-0,69	-0,46	-0,48	-0,14	-0,29	-0,62
8	-1,37	-1,69	-1,58	-1,34	-1,28	-0,62	-1,44	-1,23	-1,43	-1,47
9	+1,01	+1,06	+0,90	+1,29	+1,36	+1,00	+0,96	+1,12	+0,51	+0,46
10	-0,50	-0,82	-0,32	-0,54	-0,67	-0,43	+0,26	+0,35	+0,33	+1,23
11	+0,91	+0,58	+0,75	+0,79	+0,84	+0,98	+0,84	+0,64	+0,97	+0,80
12	+1,97	+1,61	+1,91	+1,63	+1,63	+1,72	+1,59	+1,81	+1,49	+1,15
13	-1,73	-1,70	-1,49	-1,80	-1,64	-1,25	-2,08	-1,74	-2,10	-1,75
14		+0,99		-5,00		-2,30		-1,38		+1,60

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

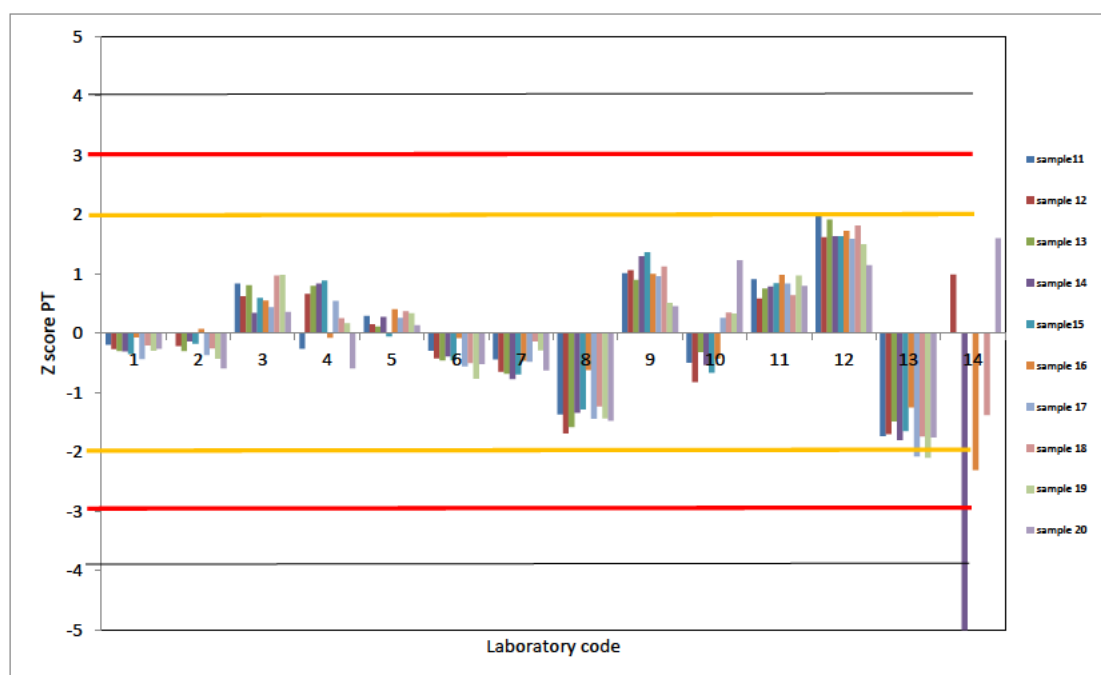


Table VII: Zscore of the different laboratories for each sample.
 ZS calculated on the standard deviation of reproducibility of the method

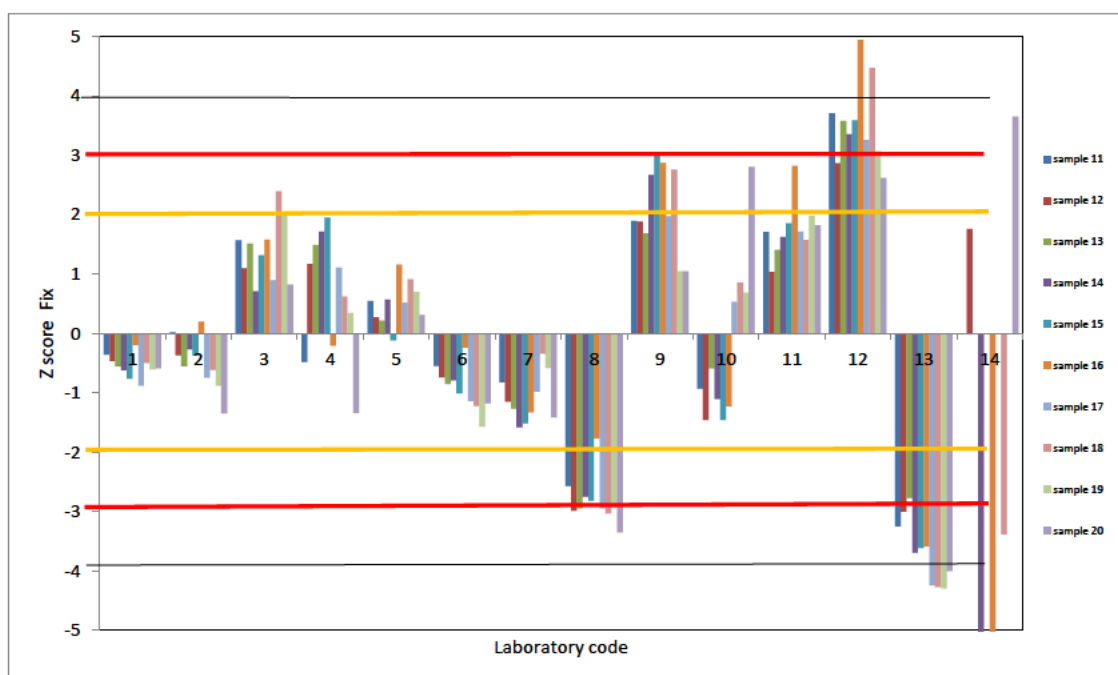
Sample Lab code	11	12	13	14	15	16	17	18	19	20
1	-0,36	-0,47	-0,56	-0,63	-0,77	-0,20	-0,89	-0,50	-0,61	-0,59
2	+0,01	-0,37	-0,56	-0,27	-0,38	+0,20	-0,75	-0,62	-0,88	-1,35
3	+1,56	+1,09	+1,51	+0,70	+1,31	+1,58	+0,89	+2,39	+2,01	+0,82
4	-0,49	+1,17	+1,49	+1,71	+1,95	-0,21	+1,10	+0,61	+0,34	-1,34
5	+0,54	+0,26	+0,21	+0,57	-0,12	+1,15	+0,52	+0,91	+0,69	+0,31
6	-0,55	-0,75	-0,85	-0,80	-1,01	-0,24	-1,14	-1,23	-1,57	-1,18
7	-0,83	-1,15	-1,27	-1,58	-1,52	-1,34	-0,98	-0,34	-0,58	-1,42
8	-2,57	-2,99	-2,95	-2,75	-2,82	-1,77	-2,95	-3,04	-2,94	-3,35
9	+1,89	+1,88	+1,68	+2,66	+3,00	+2,87	+1,96	+2,75	+1,04	+1,04
10	-0,93	-1,46	-0,59	-1,10	-1,46	-1,23	+0,53	+0,85	+0,68	+2,80
11	+1,70	+1,03	+1,40	+1,62	+1,85	+2,81	+1,71	+1,57	+1,98	+1,82
12	+3,70	+2,86	+3,57	+3,35	+3,59	+4,94	+3,25	+4,46	+3,06	+2,61
13	-3,25	-3,01	-2,78	-3,70	-3,62	-3,59	-4,25	-4,28	-4,30	-4,01
14		+1,75		-10,27		-6,61		-3,39		+3,65

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=0,047

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

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

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



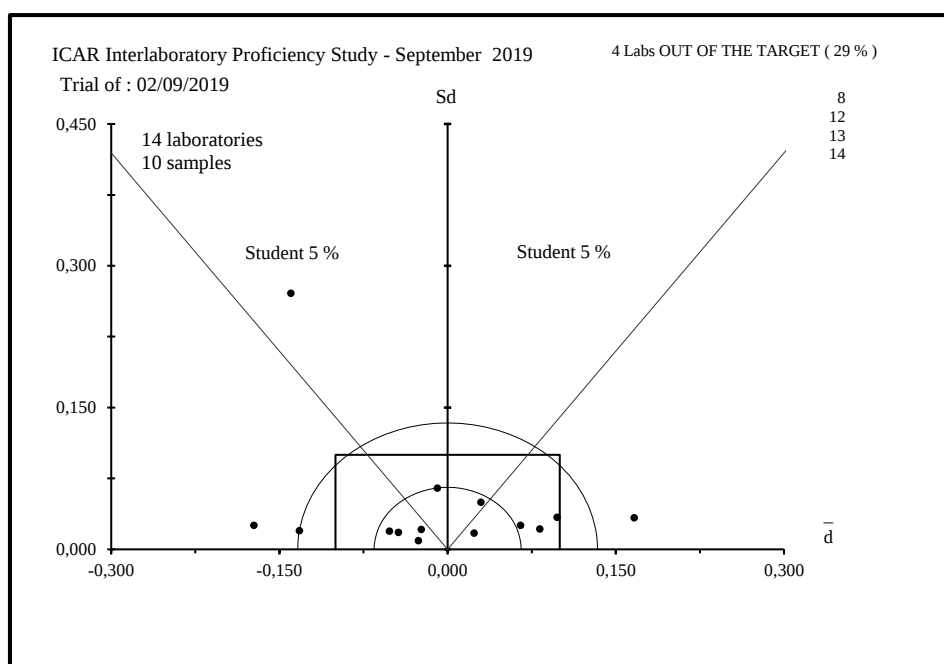


Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).

LIST OF THE PARTICIPANTS ICAR
ICAR PROFICIENCY TEST
RAW MILK
LACTOSE CONTENT
Sep 19

Name	City	Country
Actalia	Poligny	France
Agroscope Institute for food Sciences IFS	Bern-Liebefeld	Switzerland
Deltamune	Pretoria	South Africa
Department Valorisation des productions Agricoles	Gembloux	Belgium
Japan Dairy Technical Association	Tokyo	Japan
Lab Agroalimentario de Santander	Santander Cantabria	Spain
Laboratorio Standard Latte	Maccarese (Roma)	Italy
KCHZ Laboratorium Referencyjne z/s w Parzniewie	Pruszkow	Poland
Milchprüfing Baden-Württemberg e.V.	Kirchheim unter Teck	Germany
Pieno Tyrimai	Kaunas	Lithuania
Qlip B.V.,	Zutphen	Netherland
Teagasc Food research Center	Cork	Ireland
Univ. of Ljubljana	Domzale	Slovenia
Lactanet Québec	Quebec	Canada



**ICAR
PROFICIENCY TESTING SCHEME**

September 2019

Raw Milk

Determination of UREA CONTENT

Sending date of statistical treatment : 3th October 2019

Frame of activity :	ICAR Milk Analyses Sub Committee (MA SC)		
ICAR Staff	Silvia Orlandini	pt@icar.org	silvia@icar.org



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

Nb	%	N°	d	Sd	D	Method
1	11	1	+ 0,37	0,36	0,51	ISO 14637/IDF 195
2	22	2	- 0,00	0,52	0,52	Photometric
3	33	6	+ 0,84	0,56	1,01	ISO 14637/IDF 195
4	44	9	- 0,83	0,66	1,05	V 04-217
5	56	4	+ 0,54	0,96	1,10	ISO 14637/IDF 195
6	67	5	+ 0,91	0,78	1,20	Continuous flow analyzer
7	78	3	+ 1,11	0,95	1,46	ISO 14637/IDF 195
8	89	7	- 2,13	0,55	2,20	ISO 14637/IDF 195
9	100	8	+ 3,90	1,74	4,27	Continuous flow analyzer

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 6 laboratories using reference method (ISO 14637/IDF 195 or V 04-217), after outlier discarding using Grubbs test at 5% risk level

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

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

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

(D : Euclidian distance to YX-axis origin = SQUARE ROOT.(d² + Sd²))

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

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

S_{rPT} 0,43

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

S_{RPT} 1,74

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

Sample Lab code	21	22	23	24	25	26	27	28	29	30	Sr	NL
1	0,30	0,50	0,50	0,10	0,60	0,50	0,50	0,50	0,50	0,20	0,32	20
2	0,10	0,30	0,10	0,10	0,70	0,40	0,40	0,80	0,10	0,40	0,30	20
3	0,00	0,00	0,20	0,10	1,00	0,00	0,10	0,30	**	**	0,27	16
4	1,00	1,90	1,50 *	0,20	0,20	0,30	0,30	0,30	0,40	1,00	0,65	20
5	0,20	1,00	0,10	0,20	0,20	0,10	0,00	0,10	0,20	0,10	0,25	20
6	0,50	0,00	0,20	0,10	0,10	0,20	0,80	0,20	0,10	0,00	0,23	20
7	1,32	1,07	0,91 *	0,91 *	1,40	1,34	0,97	0,89	1,42	1,03	0,81	20
8	0,26	0,93	0,15	0,03	0,69	0,85	0,87	0,21	0,89	0,17	0,44	20
9	0,36	0,07	0,03	0,29	0,47	0,36	0,07	0,37	0,47	0,18	0,22	20
Sr	0,43	0,62	0,44	0,24	0,50	0,42	0,40	0,34	0,47	0,38		176
NE	18	18	18	18	18	18	18	18	16	16		
L	1,46	2,11	0,52	0,37	1,71	1,43	1,35	1,16	1,54	1,26		

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

NL : number of measurements per laboratory

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

SE : repeatability standard deviation per sample

NE : number of measurements per sample

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

** : missing data

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

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

Sample Lab code	21	22	23	24	25	26	27	28	29	30
1	43,25	48,45	38,85	33,95	62,60	53,05	29,25	24,05	58,35	19,00
2	43,65	48,05	38,35	33,45	62,65	52,60	28,60	23,60	57,85	18,30
3	43,50	48,30	38,60	33,75	63,80	55,80	30,25	25,55		
4	44,90	48,25	38,55	34,00	60,70	54,05	28,75	24,75	59,60	19,00
5	43,60	48,90	38,75	34,30	64,70	54,05	29,40	24,45	58,70	19,35
6	43,45	48,60	39,00	34,35	64,15	53,70	29,50	24,60	58,35	19,80
7	41,02	45,47	36,05	31,44	59,68	49,60	26,88	22,52	55,26	17,89
8	47,47	53,05 *	41,56	36,92	68,81	57,30	30,94	26,47	63,00	20,60
9	42,29	46,80	37,82	33,30	60,00	51,45	28,65	23,89	55,67	18,99
M	43,68	47,85	38,61	33,94	63,01	53,51	29,14	24,43	58,35	19,12
REF.	43,07	47,70	38,32	33,67	61,82	52,94	28,93	24,23	57,45	19,00
SD	1,77	1,15	1,42	1,42	2,83	2,26	1,15	1,14	2,40	0,84

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 6 laboratories using the reference method ISO 14637 | IDF 195 or V 04-2017, after outliers discarding using Grubbs test 5% risk level

Table IV : Outlier identification

Sample	21	22	23	24	25	26	27	28	29	30
Outliers										
Cochran			4;7	7						
Outlier										
Grubbs		8								
sr	0,43	0,62	0,16	0,11	0,50	0,42	0,40	0,34	0,47	0,38
SR	1,80	1,23	1,20	1,14	2,85	2,28	1,18	1,16	2,42	0,88

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

Sample Lab code	21	22	23	24	25	26	27	28	29	30	d	Sd _{lab}	t
1	+ 0,18	+ 0,75	+ 0,53	+ 0,28	+ 0,78	+ 0,11	+ 0,32	- 0,18	+ 0,90	+ 0,00	+ 0,37	0,36	3,23
2	+ 0,58	+ 0,35	+ 0,03	- 0,22	+ 0,83	- 0,34	- 0,33	- 0,63	+ 0,40	- 0,70	- 0,00	0,52	0,01
3	+ 0,43	+ 0,60	+ 0,28	+ 0,08	+ 1,98	+ 2,86	+ 1,32	+ 1,32			+ 1,11	0,95	3,29
4	+ 1,83	+ 0,55	+ 0,23	+ 0,33	- 1,12	+ 1,11	- 0,18	+ 0,52	+ 2,15	+ 0,00	+ 0,54	0,96	1,79
5	+ 0,53	+ 1,20	+ 0,43	+ 0,63	+ 2,88	+ 1,11	+ 0,47	+ 0,22	+ 1,25	+ 0,35	+ 0,91	0,78	3,66
6	+ 0,38	+ 0,90	+ 0,68	+ 0,68	+ 2,33	+ 0,76	+ 0,57	+ 0,37	+ 0,90	+ 0,80	+ 0,84	0,56	4,76
7	- 2,05	- 2,24	- 2,27	- 2,23	- 2,14	- 3,34	- 2,06	- 1,71	- 2,19	- 1,11	- 2,13	0,55	12,24
8	+ 4,40	+ 5,34	+ 3,24	+ 3,26	+ 6,98	+ 4,36	+ 2,01	+ 2,24	+ 5,55	+ 1,60	+ 3,90	1,74	7,08
9	- 0,78	- 0,91	- 0,50	- 0,36	- 1,82	- 1,49	- 0,28	- 0,33	- 1,78	- 0,01	- 0,83	0,66	3,97
d	+ 0,61	+ 0,15	+ 0,30	+ 0,27	+ 1,19	+ 0,57	+ 0,20	+ 0,20	+ 0,90	+ 0,12	+ 0,51	1,77	
Sd	1,77	1,15	1,42	1,42	2,83	2,26	1,15	1,14	2,40	0,84	1,75		

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

Upper limits : $\bar{d} = \pm 2,50$ 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	21	22	23	24	25	26	27	28	29	30
1	+0,10	+0,65	+0,37	+0,20	+0,27	+0,05	+0,28	-0,16	+0,38	+0,00
2	+0,33	+0,30	+0,02	-0,15	+0,29	-0,15	-0,29	-0,55	+0,17	-0,83
3	+0,24	+0,52	+0,20	+0,06	+0,70	+1,27	+1,15	+1,16		
4	+1,03	+0,48	+0,16	+0,24	-0,40	+0,49	-0,16	+0,46	+0,90	+0,00
5	+0,30	+1,04	+0,30	+0,45	+1,02	+0,49	+0,41	+0,20	+0,52	+0,42
6	+0,22	+0,78	+0,48	+0,48	+0,82	+0,34	+0,49	+0,33	+0,38	+0,95
7	-1,16	-1,95	-1,60	-1,57	-0,76	-1,48	-1,80	-1,51	-0,91	-1,32
8	+2,48	+4,66	+2,28	+2,29	+2,47	+1,93	+1,75	+1,97	+2,31	+1,90
9	-0,44	-0,79	-0,35	-0,25	-0,64	-0,66	-0,24	-0,29	-0,74	-0,01

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

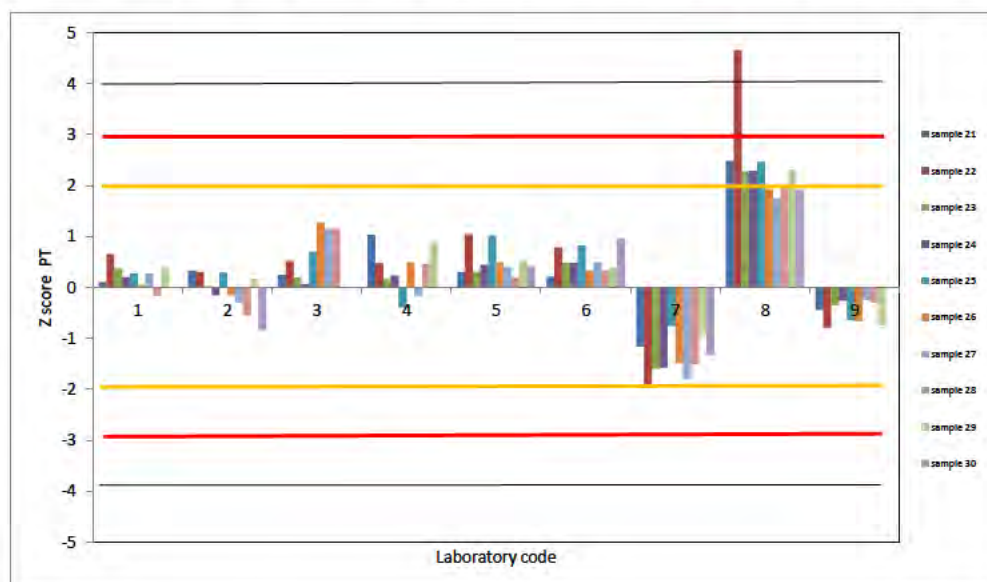


Table VII : Zscore of the different laboratories for each sample.
 ZS calculated on the standard deviation of reproducibility of the method

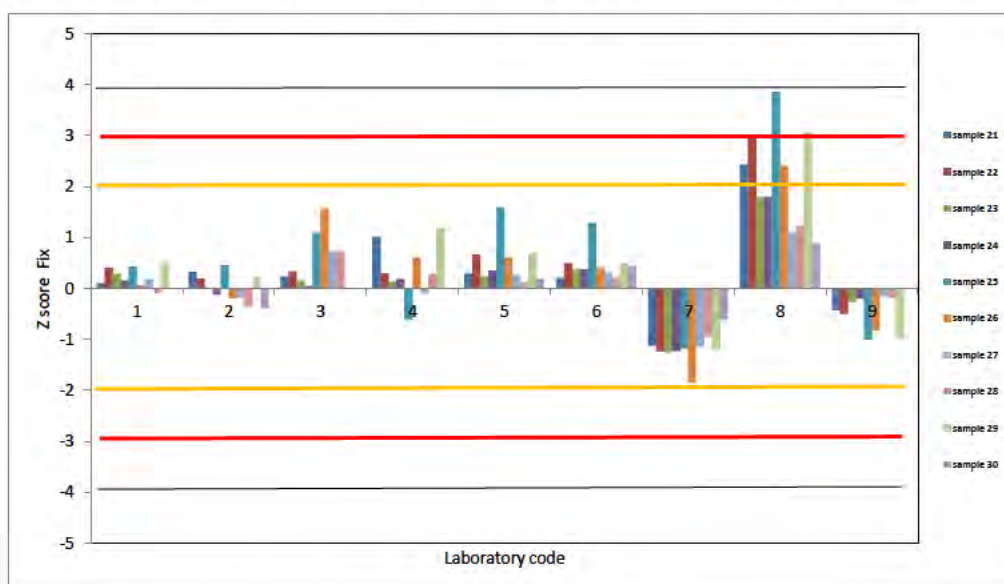
Sample lab code	21	22	23	24	25	26	27	28	29	30
1	+0,10	+0,41	+0,29	+0,16	+0,43	+0,06	+0,17	-0,10	+0,50	+0,00
2	+0,32	+0,19	+0,02	-0,12	+0,46	-0,19	-0,18	-0,35	+0,22	-0,39
3	+0,24	+0,33	+0,16	+0,05	+1,09	+1,58	+0,73	+0,73		
4	+1,01	+0,30	+0,13	+0,18	-0,62	+0,61	-0,10	+0,29	+1,19	+0,00
5	+0,29	+0,66	+0,24	+0,35	+1,59	+0,61	+0,26	+0,12	+0,69	+0,19
6	+0,21	+0,50	+0,38	+0,38	+1,29	+0,42	+0,31	+0,21	+0,50	+0,44
7	-1,13	-1,24	-1,26	-1,23	-1,18	-1,85	-1,14	-0,95	-1,21	-0,61
8	+2,43	+2,95	+1,79	+1,80	+3,86	+2,41	+1,11	+1,24	+3,07	+0,88
9	-0,43	-0,50	-0,27	-0,20	-1,01	-0,82	-0,15	-0,18	-0,98	-0,01

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

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

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

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



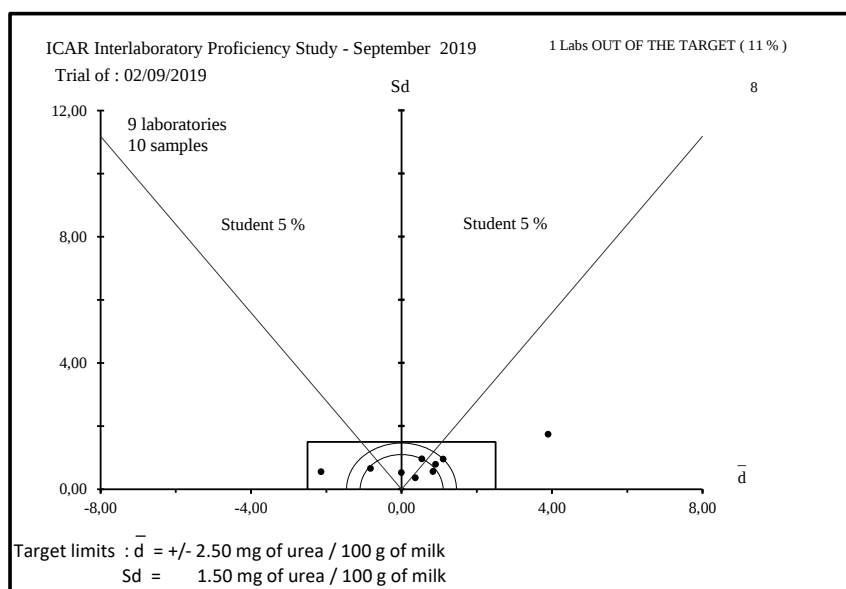


Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).

LIST OF THE PARTICIPANTS ICAR
ICAR PROFICIENCY TEST
RAW MILK
UREA CONTENT
Sep 19

Name	City	Country
Agroscope Institute for food Sciences IFS	Bern-Liebefeld	Switzerland
Central Milk Lab ICBA	Caesarea	Israel
Department Valorisation des productions Agricoles	Gembloux	Belgium
Laboratorio Standard Latte	Maccarese (Roma)	Italy
KCHZ Laboratorium Referencyjne z/s w Parzniewie	Pruszkow	Poland
Milchprüfring Baden-Württemberg e.V.	Kirchheim unter Teck	Germany
Pieno Tyrimai	Kaunas	Lithuania
Qlip B.V.,	Zutphen	Netherland
Lactanet Québec	Quebec	Canada



**ICAR
PROFICIENCY TESTING SCHEME**

September 2019

Raw Milk

Enumeration of SOMATIC CELLS

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Proficiency test accredited ISO 17043



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Table I : Ranking of the laboratories in %

Nb	%	N°	d	Sd	D	Method
1	2	35	- 2%	2%	3%	B
2	5	13	- 1%	2%	3%	B
3	7	8	- 0%	3%	3%	B
4	9	39	+ 1%	3%	3%	B
5	11	34	- 2%	2%	3%	B
6	14	12	+ 2%	2%	3%	B
7	16	26	+ 2%	4%	4%	C
8	18	22	- 0%	4%	4%	B
9	20	6	- 3%	4%	4%	B
10	23	40	- 4%	2%	5%	B
11	25	23	+ 1%	5%	5%	B
12	27	25	+ 1%	5%	5%	B
13	30	27	- 4%	3%	5%	B
14	32	42	- 0%	5%	5%	B
15	34	38	- 2%	6%	6%	B
16	36	41	+ 4%	5%	6%	C
17	39	36	- 5%	5%	7%	B
18	41	19	- 3%	7%	8%	B
19	43	37	- 5%	6%	8%	B
20	45	32	- 6%	5%	8%	B
21	48	31	- 3%	8%	9%	B
22	50	20	+ 7%	6%	9%	B
23	52	24	+ 6%	7%	9%	B
24	55	7	- 8%	6%	10%	B
25	57	14	- 9%	6%	11%	B

Nb	%	N°	d	Sd	D	Method
26	59	5	+ 5%	12%	13%	A
27	61	3	- 12%	7%	14%	B
28	64	1	- 12%	7%	14%	B
29	66	28	- 10%	11%	14%	B
30	68	30	- 7%	13%	15%	A
31	70	16	- 12%	9%	15%	B
32	73	43	- 14%	10%	17%	B
33	75	29	- 15%	10%	18%	B
34	77	18	+ 14%	12%	19%	B
35	80	21	+ 17%	9%	19%	B
36	82	11	- 2%	21%	21%	B
37	84	4	+ 16%	14%	21%	B
38	86	2	+ 14%	17%	22%	B
39	89	33	+ 19%	16%	24%	B
40	91	17	+ 27%	13%	30%	B
41	93	15	+ 25%	20%	32%	B
42	95	9	+ 35%	34%	49%	A
43	98	44	- 45%	35%	56%	B
44	100	10	+ 48%	38%	61%	B

A ISO 13366-1 | IDF 148-1
B ISO 13366-2 | IDF 148-2
C Image Cytometry

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

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 :

+/- 10% for d and 10% for Sd

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 42 laboratories using reference method ISO 13366-1|IDF 148-1 and alternative method ISO 13366-2|IDF 148-2 after outlier discarding using Grubbs test at 5% risk level

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} 16 4%
Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) SR_{PT} 66 16%

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

Sample Lab code	31	32	33	34	35	36	37	38	39	40	Sr	NL
1	5	5	18	5	6	28	53	10	6	14	15	20
2	6	2	8	5	4	15	22	3	5	4	7	20
3	20	3	4	22	9	6	14	10	1	11	8	20
4	10	6	21	30	10	25	50	7	6	7	16	20
5	12	6	50	20	83 *	92	103	4	16	2	38	20
6	2	8	20	39	34	8	11	11	5	11	13	20
7	11	1	13	2	11	9	3	5	8	10	6	20
8	3	2	2	4	39	33	56	2	13	21	18	20
9	10	3	13	26	6	25	28	5	5	11	11	20
10	2	8	7	2	5	3	5	3	3	7	4	20
11	20	4	33	30	7	15	42	14	34 *	1	17	20
12	4	1	9	13	11	8	7	6	15	8	6	20
13	5	5	29	52	4	19	19	2	2	12	15	20
14	9	3	28	7	4	7	23	3	4	5	9	20
15	1	1	21	15	3	27	15	5	6	10	9	20
16	10	10	10	20	10	0	10	0	10	10	7	20
17	16	8	2	6	2	9	4	20	10	3	7	20
18	8	1	8	3	8	4	25	1	5	3	7	20
19	10	3	1	33	2	19	31	1	9	7	12	20
20	41 *	5	64	36	1	25	28	3	1	2	21	20
21	9	3	71	37	2	51	32	10	9	1	23	20
22	16	7	22	30	50	14	31	4	4	16	17	20
23	9	3	38	1	2	32	8	1	1	17	12	20
24	5	5	2	28	3	10	5	8	4	8	7	20
25	7	3	19	6	9	38	7	6	3	32	12	20
26	0	0	9	7	6	9	10	4	1	1	4	20
27	28	7	13	55	58	67	92	22	15	13	33	20
28	5	1	3	6	7	4	21	1	1	11	6	20
29	1	9	10	14	44	32	32	1	4	4	15	20
30	39 *	2	32	16	10	19	50	10	4	29	18	20

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

Sample Lab code	31	32	33	34	35	36	37	38	39	40	Sr	NL
31	4	2	**	3	28	12	25	4	**	**	11	14
32	4	1	12	51	4	17	10	3	0	4	13	20
33	6	11	10	78	33	42	51	19	2	20	25	20
34	5	3	43	36	50	51	14	13	9	7	21	20
35	11	3	31	23	14	29	0	3	13	4	12	20
36	6	7	13	40	17	24	19	7	10	31	14	20
37	2	1	52	7	8	51	23	21	4	4	18	20
38	7	9	8	35	31	32	2	5	1	4	13	20
39	7	1	0	20	20	4	5	5	2	3	7	20
40	6	4	1	35	16	22	9	8	9	10	11	20
41	4	4	11	12	3	12	24	4	1	3	7	20
42	126	*	11	52	40	14	76	111	25	16	20	45
43	23	1	21	16	15	28	29	2	13	21	14	20
44	10	5	7	26	7	26	2	2	15	27	11	20
Sr	10	4	18	17	18	21	26	6	7	9		600
r	42	25	63	63	50	126	126	25	25	50		
NE	60	60	60	60	60	60	60	60	60	60		
L	32	16	81	88	64	98	114	29	25	41		

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

NL : number of measurements per laboratory

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

SE : repeatability standard deviation per sample

NE : number of measurements per sample

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

** : missing data

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

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

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

Sample Lab code	31	32	33	34	35	36	37	38	39	40
1	170	35	428	544	345	768	795	71	123	261
2	213	45	566	733	447	878	1101	88	163	322
3	187	34	447	570	337	708	792	71	123	279
4	240	46	595	758	429	965	991	101	165	369
5	209	32	568	748	400	857	847	65	123	341
6	203	39	485	646	375	751	873	87	136	311
7	206	40	471	557	334	764	841	70	134	291
8	199	34	495	646	380	800	908	85	139	316
9	269	56	664	907 *	507	1081	1284	98	181	376
10	306	52	760 *	996 *	588 *	1176 *	1287	108	204	462
11	291	65	463	583	377	674	734	118	201	422
12	208	41	515	654	399	819	895	84	157	324
13	208	37	494	622	385	808	887	77	135	297
14	183	35	447	596	361	721	805	77	128	278
15	252	47	632	812	487	1030	1068	98	175	400
16	185	35	455	550	365	700	775	70	135	275
17	350	59	638	770	503	955	1051	136 *	196	443
18	245	45	584	738	454	907	1015	86	156	360
19	210	35	452	597	374	847	838	79	151	318
20	263	40	509	678	450	829	880	82	176	372
21	338	44	558	736	473	883	981	105	185	389
22	208	43	475	659	383	772	916	87	148	306
23	197	41	508	639	379	810	932	78	145	308
24	216	41	531	688	405	858	958	84	149	325
25	212	36	483	659	390	810	934	83	139	313
26	196	38	507	666	401	815	912	84	143	317
27	195	37	458	620	374	765	876	79	136	308
28	205	40	425	599	375	681	788	83	149	281
29	177	38	418	553	316	690	758	71	125	272
30	211	58	438	500	357	761	797	97	160	335

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 42 laboratories using the reference method ISO 13366 | IDF 148-1 and alternative method ISO 13366-2 | IDF 148-2, after outlier discarding using Grubbs test at 5% risk level

Table IV : Outlier identification

Sample	31	32	33	34	35	36	37	38	39	40
Outliers										
Cochran	20;30				5				11	
Outlier			10	9;10	10	10		17		
Grubbs										
sr	7	4	19	20	15	23	26	6	6	9
SR	44	8	62	70	49	91	139	14	22	51
sr %	3%	9%	4%	3%	4%	3%	3%	7%	4%	3%
SR %	21%	21%	12%	11%	12%	11%	16%	17%	15%	16%

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

Sample Lab code	31	32	33	34	35	36	37	38	39	40
31	216	73 *	489	589	384	761	808	110	161	308
32	198	35	441	598	370	752	840	79	138	304
33	259	50	536	715	485	928	1107	112	175	385
34	198	40	473	618	387	789	886	87	135	313
35	197	42	489	632	381	777	877	83	144	310
36	202	44	465	607	364	766	836	98	135	306
37	179	41	487	632	375	738	826	82	143	299
38	196	42	502	644	411	786	823	85	153	299
39	212	39	516	642	401	800	907	80	141	310
40	205	39	481	611	372	769	864	80	137	295
41	214	39	526	665	406	823	941	84	145	318
42	261	35	482	639	384	813	892	73	131	296
43	173	37	411	573	312	695	762	79	123	289
44	114	20	277 *	350 *	224 *	416 *	487	49	98	188
M	217	41	500	641	395	807	895	85	148	322
REF.	214	40	494	638	391	798	883	84	147	319
SD	44	8	60	69	47	90	138	13	23	50

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 42 laboratories using the reference method ISO 13366 | IDF 148-1 and alternative method ISO 13366-2 | IDF 148-2,
after outlier discarding using Grubbs test at 5% risk level

Table IV : Outlier identification

Sample	31	32	33	34	35	36	37	38	39	40
Outliers Cochran	42									
Outlier Grubbs		31	44	44	44	44				
sr	7	4	19	20	15	23	26	6	6	9
SR	44	8	62	70	49	91	139	14	22	51
sr %	3%	9%	4%	3%	4%	3%	3%	7%	4%	3%
SR %	21%	21%	12%	11%	12%	11%	16%	17%	15%	16%

Table V : ACCURACY - differences (laboratory - reference) in %

Sample Lab code	31	32	33	34	35	36	37	38	39	40	d	Sd _{lab}	t
1	- 21%	- 14%	- 13%	- 15%	- 12%	- 4%	- 10%	- 16%	- 17%	- 18%	- 12%	7%	4,94
2	- 0%	+ 12%	+ 15%	+ 15%	+ 14%	+ 10%	+ 25%	+ 4%	+ 10%	+ 1%	+ 14%	17%	2,54
3	- 12%	- 17%	- 9%	- 11%	- 14%	- 11%	- 10%	- 16%	- 17%	- 13%	- 12%	7%	4,90
4	+ 12%	+ 15%	+ 20%	+ 19%	+ 10%	+ 21%	+ 12%	+ 20%	+ 12%	+ 15%	+ 16%	14%	3,74
5	- 2%	- 20%	+ 15%	+ 17%	+ 2%	+ 7%	- 4%	- 23%	- 17%	+ 7%	+ 5%	12%	1,19
6	- 5%	- 3%	- 2%	+ 1%	- 4%	- 6%	- 1%	+ 3%	- 8%	- 3%	- 3%	4%	2,24
7	- 4%	- 2%	- 5%	- 13%	- 15%	- 4%	- 5%	- 17%	- 9%	- 9%	- 8%	6%	3,91
8	- 7%	- 15%	+ 0%	+ 1%	- 3%	+ 0%	+ 3%	+ 1%	- 6%	- 1%	- 0%	3%	0,21
9	+ 26%	+ 38%	+ 34%	+ 42%	+ 30%	+ 35%	+ 45%	+ 16%	+ 22%	+ 18%	+ 35%	34%	3,31
10	+ 43%	+ 29%	+ 54%	+ 56%	+ 50%	+ 47%	+ 46%	+ 28%	+ 38%	+ 45%	+ 48%	38%	4,05
11	+ 36%	+ 62%	- 6%	- 9%	- 4%	- 16%	- 17%	+ 40%	+ 36%	+ 32%	- 2%	21%	0,31
12	- 3%	+ 1%	+ 4%	+ 2%	+ 2%	+ 3%	+ 1%	- 0%	+ 6%	+ 1%	+ 2%	2%	2,99
13	- 3%	- 9%	- 0%	- 2%	- 1%	+ 1%	+ 0%	- 8%	- 8%	- 7%	- 1%	2%	2,03
14	- 15%	- 14%	- 9%	- 7%	- 8%	- 10%	- 9%	- 9%	- 13%	- 13%	- 9%	6%	4,76
15	+ 18%	+ 16%	+ 28%	+ 27%	+ 25%	+ 29%	+ 21%	+ 16%	+ 19%	+ 25%	+ 25%	20%	3,93
16	- 13%	- 13%	- 8%	- 14%	- 7%	- 12%	- 12%	- 17%	- 8%	- 14%	- 12%	9%	3,87
17	+ 64%	+ 47%	+ 29%	+ 21%	+ 29%	+ 20%	+ 19%	+ 62%	+ 33%	+ 39%	+ 27%	13%	6,76
18	+ 15%	+ 11%	+ 18%	+ 16%	+ 16%	+ 14%	+ 15%	+ 2%	+ 5%	+ 13%	+ 14%	12%	3,85
19	- 2%	- 14%	- 9%	- 6%	- 4%	+ 6%	- 5%	- 7%	+ 2%	- 1%	- 3%	7%	1,24
20	+ 23%	- 2%	+ 3%	+ 6%	+ 15%	+ 4%	- 0%	- 3%	+ 19%	+ 17%	+ 7%	6%	3,59
21	+ 58%	+ 8%	+ 13%	+ 15%	+ 21%	+ 11%	+ 11%	+ 25%	+ 25%	+ 22%	+ 17%	9%	5,72
22	- 3%	+ 6%	- 4%	+ 3%	- 2%	- 3%	+ 4%	+ 4%	+ 0%	- 4%	- 0%	4%	0,20
23	- 8%	+ 1%	+ 3%	+ 0%	- 3%	+ 2%	+ 6%	- 8%	- 2%	- 4%	+ 1%	5%	0,44
24	+ 1%	+ 1%	+ 8%	+ 8%	+ 4%	+ 8%	+ 8%	- 0%	+ 1%	+ 2%	+ 6%	7%	2,73
25	- 1%	- 12%	- 2%	+ 3%	- 0%	+ 2%	+ 6%	- 1%	- 6%	- 2%	+ 1%	5%	0,82
26	- 8%	- 5%	+ 3%	+ 4%	+ 3%	+ 2%	+ 3%	- 0%	- 3%	- 1%	+ 2%	4%	1,46
27	- 9%	- 9%	- 7%	- 3%	- 4%	- 4%	- 1%	- 6%	- 8%	- 4%	- 4%	3%	4,67
28	- 4%	- 2%	- 14%	- 6%	- 4%	- 15%	- 11%	- 2%	+ 1%	- 12%	- 10%	11%	2,89
29	- 17%	- 7%	- 15%	- 13%	- 19%	- 13%	- 14%	- 16%	- 15%	- 15%	- 15%	10%	4,56
30	- 1%	+ 44%	- 11%	- 22%	- 9%	- 5%	- 10%	+ 15%	+ 9%	+ 5%	- 7%	13%	1,80

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

Upper limits : $\bar{d} = \pm 10\%$ Sd = 10%

ISO 13366-2 | IDF 148-2 : Precision of the method :

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

Table V : ACCURACY - differences (laboratory - reference) in %

Sample Lab code	31	32	33	34	35	36	37	38	39	40	d	Sd _{lab}	t
31	+ 1%	+ 82%	- 1%	- 8%	- 2%	- 5%	- 9%	+ 31%	+ 9%	- 4%	- 3%	8%	1,01
32	- 7%	- 14%	- 11%	- 6%	- 5%	- 6%	- 5%	- 7%	- 6%	- 5%	- 6%	5%	4,43
33	+ 21%	+ 23%	+ 9%	+ 12%	+ 24%	+ 16%	+ 25%	+ 33%	+ 19%	+ 21%	+ 19%	16%	3,70
34	- 8%	- 2%	- 4%	- 3%	- 1%	- 1%	+ 0%	+ 3%	- 9%	- 2%	- 2%	2%	3,05
35	- 8%	+ 3%	- 1%	- 1%	- 2%	- 3%	- 1%	- 2%	- 3%	- 3%	- 2%	2%	3,64
36	- 5%	+ 8%	- 6%	- 5%	- 7%	- 4%	- 5%	+ 16%	- 8%	- 4%	- 5%	5%	3,26
37	- 16%	+ 1%	- 1%	- 1%	- 4%	- 8%	- 7%	- 3%	- 3%	- 6%	- 5%	6%	2,91
38	- 8%	+ 3%	+ 2%	+ 1%	+ 5%	- 1%	- 7%	+ 1%	+ 3%	- 6%	- 2%	6%	0,97
39	- 1%	- 4%	+ 5%	+ 1%	+ 3%	+ 0%	+ 3%	- 5%	- 4%	- 3%	+ 1%	3%	1,05
40	- 4%	- 3%	- 3%	- 4%	- 5%	- 4%	- 2%	- 5%	- 7%	- 8%	- 4%	2%	5,17
41	+ 0%	- 3%	+ 6%	+ 4%	+ 4%	+ 3%	+ 7%	- 0%	- 2%	- 1%	+ 4%	5%	2,37
42	+ 22%	- 14%	- 2%	+ 0%	- 2%	+ 2%	+ 1%	- 14%	- 11%	- 7%	- 0%	5%	0,04
43	- 19%	- 9%	- 17%	- 10%	- 20%	- 13%	- 14%	- 6%	- 17%	- 10%	- 14%	10%	4,31
44	- 47%	- 51%	- 44%	- 45%	- 43%	- 48%	- 45%	- 42%	- 34%	- 41%	- 45%	35%	4,08
d	+ 2%	+ 2%	+ 1%	+ 1%	+ 1%	+ 1%	+ 1%	+ 1%	+ 1%	+ 1%	- 2%	17%	
Sd	20%	20%	12%	11%	12%	11%	16%	16%	15%	16%			

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

Upper limits : d = +/- 10% Sd = 10%

ISO 13366-2|IDF 148-2 : Precision of the method :

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

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

Sample Lab code	31	32	33	34	35	36	37	38	39	40
1	-1,01	-0,70	-1,10	-1,37	-0,98	-0,33	-0,64	-0,97	-1,08	-1,17
2	-0,02	+0,60	+1,21	+1,38	+1,20	+0,89	+1,58	+0,26	+0,67	+0,05
3	-0,61	-0,83	-0,78	-0,99	-1,16	-1,00	-0,66	-0,97	-1,11	-0,82
4	+0,60	+0,72	+1,69	+1,75	+0,82	+1,86	+0,78	+1,23	+0,78	+0,99
5	-0,11	-1,01	+1,25	+1,61	+0,19	+0,66	-0,27	-1,42	-1,08	+0,44
6	-0,24	-0,15	-0,14	+0,11	-0,33	-0,52	-0,08	+0,18	-0,53	-0,18
7	-0,19	-0,08	-0,39	-1,18	-1,22	-0,38	-0,31	-1,09	-0,59	-0,57
8	-0,35	-0,77	+0,02	+0,12	-0,24	+0,02	+0,18	+0,07	-0,40	-0,08
9	+1,27	+1,90	+2,85	+3,93	+2,49	+3,16	+2,91	+1,00	+1,47	+1,13
10	+2,11	+1,47	+4,45	+5,22	+4,21	+4,22	+2,93	+1,75	+2,49	+2,85
11	+1,77	+3,08	-0,52	-0,80	-0,30	-1,38	-1,08	+2,53	+2,38	+2,05
12	-0,13	+0,04	+0,35	+0,23	+0,17	+0,24	+0,08	-0,00	+0,40	+0,09
13	-0,14	-0,46	-0,00	-0,23	-0,12	+0,11	+0,02	-0,53	-0,55	-0,45
14	-0,71	-0,70	-0,78	-0,61	-0,63	-0,86	-0,57	-0,56	-0,86	-0,84
15	+0,87	+0,79	+2,31	+2,53	+2,05	+2,59	+1,34	+1,00	+1,22	+1,62
16	-0,66	-0,64	-0,64	-1,28	-0,55	-1,09	-0,79	-1,05	-0,55	-0,89
17	+3,12	+2,34	+2,42	+1,93	+2,40	+1,75	+1,22	+3,87	+2,16	+2,47
18	+0,72	+0,54	+1,51	+1,46	+1,35	+1,22	+0,95	+0,11	+0,36	+0,81
19	-0,08	-0,70	-0,70	-0,60	-0,36	+0,55	-0,33	-0,41	+0,14	-0,04
20	+1,12	-0,08	+0,26	+0,59	+1,26	+0,34	-0,02	-0,19	+1,25	+1,06
21	+2,83	+0,41	+1,07	+1,43	+1,76	+0,95	+0,71	+1,56	+1,65	+1,39
22	-0,13	+0,29	-0,31	+0,31	-0,16	-0,29	+0,23	+0,22	+0,03	-0,27
23	-0,39	+0,04	+0,24	+0,01	-0,25	+0,14	+0,35	-0,49	-0,13	-0,24
24	+0,04	+0,04	+0,63	+0,73	+0,30	+0,67	+0,54	-0,00	+0,07	+0,11
25	-0,05	-0,58	-0,18	+0,31	-0,02	+0,14	+0,37	-0,08	-0,40	-0,13
26	-0,40	-0,27	+0,22	+0,41	+0,22	+0,19	+0,21	-0,00	-0,22	-0,06
27	-0,43	-0,46	-0,60	-0,27	-0,36	-0,37	-0,05	-0,38	-0,53	-0,24
28	-0,21	-0,08	-1,16	-0,56	-0,35	-1,30	-0,70	-0,12	+0,05	-0,78
29	-0,85	-0,33	-1,26	-1,23	-1,60	-1,20	-0,91	-1,01	-0,99	-0,95
30	-0,07	+2,21	-0,93	-2,01	-0,72	-0,41	-0,63	+0,97	+0,56	+0,30

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

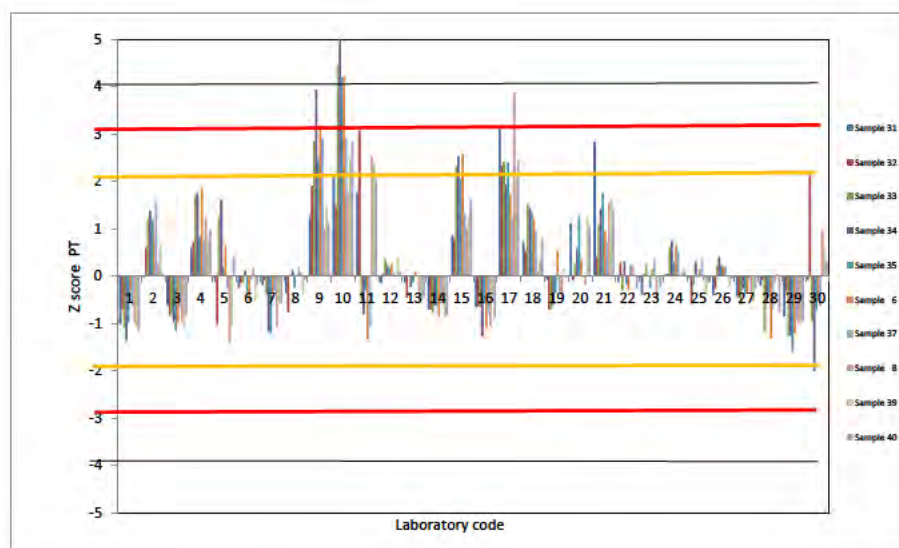


Table VI : Zscore of the different laboratories for each sample.
ZS calculated on the PT standard deviation

Sample Lab code	31	32	33	34	35	36	37	38	39	40
31	+0,05	+4,07	-0,08	-0,72	-0,14	-0,41	-0,55	+1,94	+0,60	-0,23
32	-0,36	-0,70	-0,88	-0,59	-0,44	-0,51	-0,31	-0,41	-0,42	-0,31
33	+1,04	+1,16	+0,71	+1,13	+2,00	+1,46	+1,62	+2,05	+1,22	+1,32
34	-0,37	-0,08	-0,35	-0,29	-0,08	-0,10	+0,02	+0,18	-0,57	-0,14
35	-0,39	+0,17	-0,08	-0,09	-0,21	-0,24	-0,05	-0,12	-0,17	-0,19
36	-0,27	+0,41	-0,49	-0,45	-0,58	-0,35	-0,35	+1,00	-0,55	-0,28
37	-0,79	+0,04	-0,11	-0,09	-0,33	-0,67	-0,42	-0,19	-0,20	-0,41
38	-0,42	+0,17	+0,14	+0,08	+0,42	-0,13	-0,44	+0,03	+0,23	-0,41
39	-0,05	-0,21	+0,38	+0,06	+0,22	+0,03	+0,17	-0,34	-0,28	-0,20
40	-0,20	-0,15	-0,22	-0,40	-0,40	-0,32	-0,14	-0,30	-0,48	-0,49
41	+0,01	-0,15	+0,54	+0,40	+0,32	+0,28	+0,42	-0,00	-0,13	-0,04
42	+1,08	-0,70	-0,19	+0,02	-0,14	+0,17	+0,06	-0,86	-0,73	-0,47
43	-0,94	-0,46	-1,39	-0,94	-1,69	-1,14	-0,88	-0,38	-1,11	-0,62
44	-2,28	-2,57	-3,63	-4,19	-3,57	-4,26	-2,88	-2,61	-2,21	-2,64

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

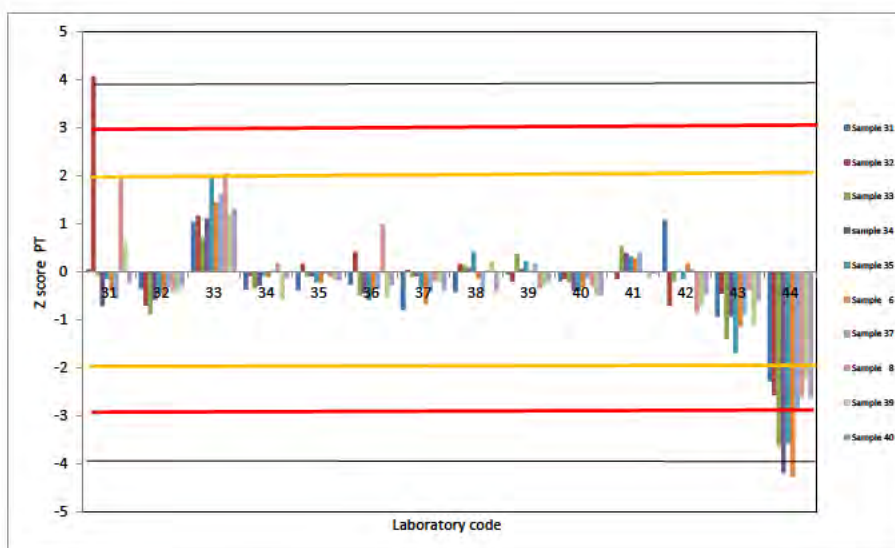


Table VII : Zscore of the different laboratories for each sample.
 ZS calculated on the standard deviation of reproducibility of the method

Sample Lab code	31	32	33	34	35	36	37	38	39	40
1	-2,30	-1,57	-1,90	-2,11	-1,46	-0,62	-1,67	-1,73	-1,84	-2,28
2	-0,03	+1,34	+2,10	+2,12	+1,80	+1,67	+4,11	+0,46	+1,14	+0,11
3	-1,39	-1,85	-1,35	-1,52	-1,73	-1,87	-1,72	-1,73	-1,88	-1,60
4	+1,37	+1,61	+2,92	+2,70	+1,23	+3,49	+2,03	+2,17	+1,33	+1,93
5	-0,24	-2,26	+2,16	+2,47	+0,28	+1,24	-0,69	-2,52	-1,84	+0,85
6	-0,55	-0,32	-0,25	+0,18	-0,50	-0,97	-0,20	+0,32	-0,90	-0,34
7	-0,42	-0,19	-0,67	-1,81	-1,83	-0,71	-0,81	-1,92	-1,01	-1,11
8	-0,79	-1,71	+0,04	+0,19	-0,36	+0,04	+0,47	+0,13	-0,67	-0,15
9	+2,88	+4,24	+4,92	+6,03	+3,72	+5,91	+7,56	+1,78	+2,49	+2,20
10	+4,80	+3,27	+7,70	+8,03	+6,30	+7,90	+7,61	+3,10	+4,23	+5,57
11	+4,02	+6,87	-0,90	-1,22	-0,45	-2,59	-2,82	+4,49	+4,04	+4,00
12	-0,29	+0,09	+0,61	+0,35	+0,25	+0,45	+0,21	-0,01	+0,69	+0,19
13	-0,32	-1,02	-0,00	-0,35	-0,18	+0,21	+0,06	-0,93	-0,93	-0,87
14	-1,62	-1,57	-1,35	-0,94	-0,95	-1,61	-1,49	-1,00	-1,46	-1,64
15	+1,97	+1,75	+3,99	+3,89	+3,07	+4,85	+3,48	+1,78	+2,08	+3,16
16	-1,49	-1,43	-1,12	-1,96	-0,82	-2,04	-2,04	-1,86	-0,93	-1,73
17	+7,09	+5,21	+4,18	+2,96	+3,59	+3,28	+3,17	+6,87	+3,66	+4,82
18	+1,63	+1,20	+2,62	+2,24	+2,03	+2,29	+2,48	+0,19	+0,61	+1,58
19	-0,19	-1,57	-1,22	-0,92	-0,53	+1,02	-0,86	-0,73	+0,23	-0,07
20	+2,54	-0,19	+0,45	+0,90	+1,88	+0,65	-0,06	-0,34	+2,12	+2,06
21	+6,44	+0,92	+1,85	+2,19	+2,63	+1,77	+1,84	+2,77	+2,80	+2,71
22	-0,29	+0,64	-0,54	+0,48	-0,25	-0,53	+0,61	+0,39	+0,05	-0,52
23	-0,89	+0,09	+0,42	+0,02	-0,37	+0,26	+0,92	-0,87	-0,22	-0,46
24	+0,10	+0,09	+1,08	+1,13	+0,44	+1,26	+1,40	-0,01	+0,12	+0,22
25	-0,11	-1,29	-0,32	+0,48	-0,04	+0,26	+0,95	-0,14	-0,67	-0,25
26	-0,92	-0,60	+0,38	+0,62	+0,33	+0,35	+0,54	-0,01	-0,37	-0,11
27	-0,97	-1,02	-1,04	-0,41	-0,53	-0,69	-0,14	-0,67	-0,90	-0,46
28	-0,48	-0,19	-2,00	-0,87	-0,52	-2,44	-1,81	-0,20	+0,08	-1,52
29	-1,93	-0,74	-2,19	-1,90	-2,39	-2,25	-2,36	-1,79	-1,69	-1,85
30	-0,16	+4,93	-1,61	-3,08	-1,08	-0,77	-1,63	+1,71	+0,95	+0,60

This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR : Cf page 7 and 8 /13

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

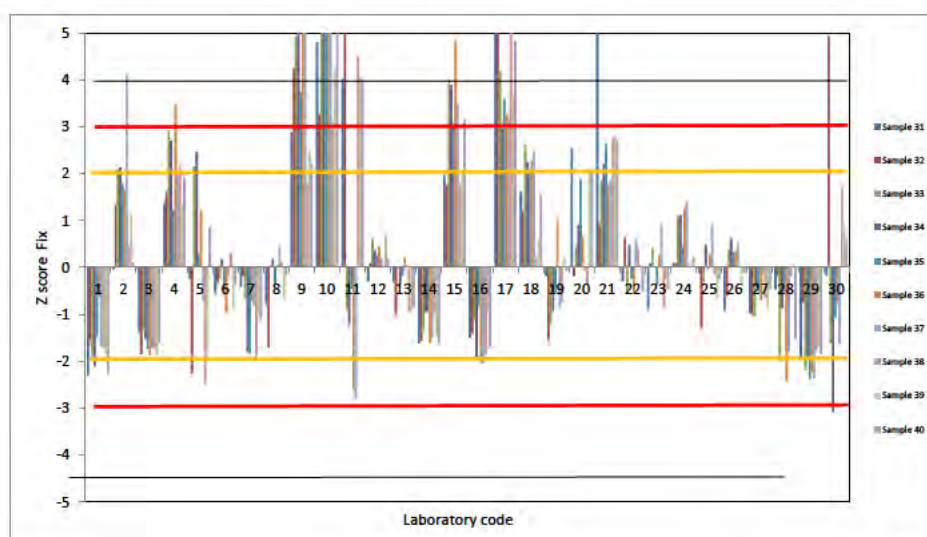


Table VII : Zscore of the different laboratories for each sample.
 ZS calculated on the standard deviation of reproducibility of the method

Sample Lab code	31	32	33	34	35	36	37	38	39	40
31	+0,12	+9,08	-0,13	-1,10	-0,21	-0,76	-1,43	+3,43	+1,02	-0,44
32	-0,81	-1,57	-1,52	-0,90	-0,66	-0,96	-0,82	-0,73	-0,71	-0,60
33	+2,36	+2,58	+1,23	+1,73	+3,00	+2,73	+4,21	+3,63	+2,08	+2,57
34	-0,84	-0,19	-0,61	-0,44	-0,12	-0,19	+0,05	+0,32	-0,97	-0,27
35	-0,89	+0,37	-0,15	-0,14	-0,31	-0,44	-0,12	-0,20	-0,29	-0,36
36	-0,61	+0,92	-0,84	-0,69	-0,87	-0,66	-0,90	+1,78	-0,93	-0,54
37	-1,80	+0,09	-0,19	-0,14	-0,50	-1,26	-1,09	-0,34	-0,33	-0,79
38	-0,94	+0,37	+0,25	+0,13	+0,63	-0,24	-1,14	+0,06	+0,38	-0,79
39	-0,11	-0,46	+0,65	+0,10	+0,33	+0,05	+0,44	-0,60	-0,48	-0,38
40	-0,45	-0,32	-0,38	-0,61	-0,60	-0,60	-0,37	-0,54	-0,82	-0,95
41	+0,02	-0,32	+0,93	+0,61	+0,47	+0,53	+1,09	-0,01	-0,22	-0,07
42	+2,46	-1,57	-0,33	+0,03	-0,21	+0,32	+0,16	-1,53	-1,24	-0,91
43	-2,14	-1,02	-2,40	-1,45	-2,53	-2,14	-2,30	-0,67	-1,88	-1,20
44	-5,18	-5,72	-6,28	-6,44	-5,35	-7,97	-7,48	-6,63	-3,76	-5,16

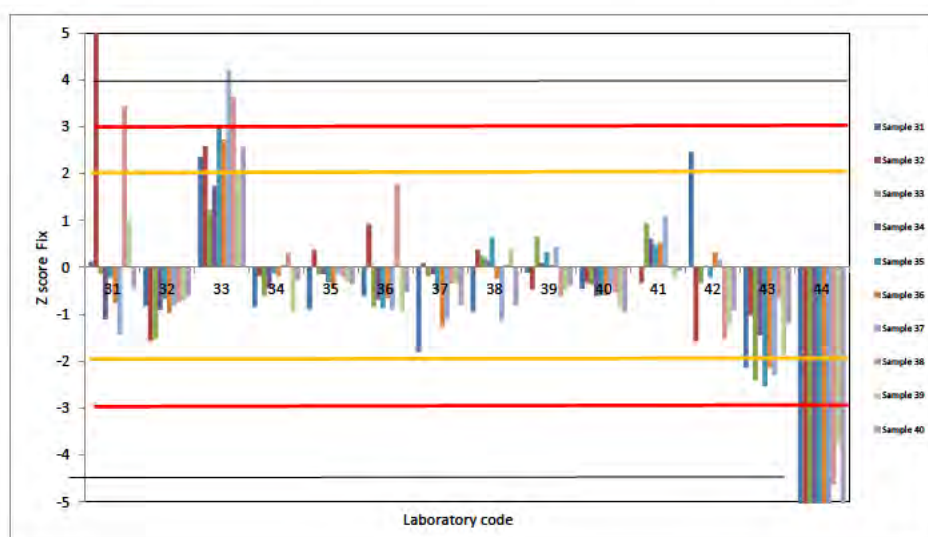
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR : Cf page 7 and 8 /13

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



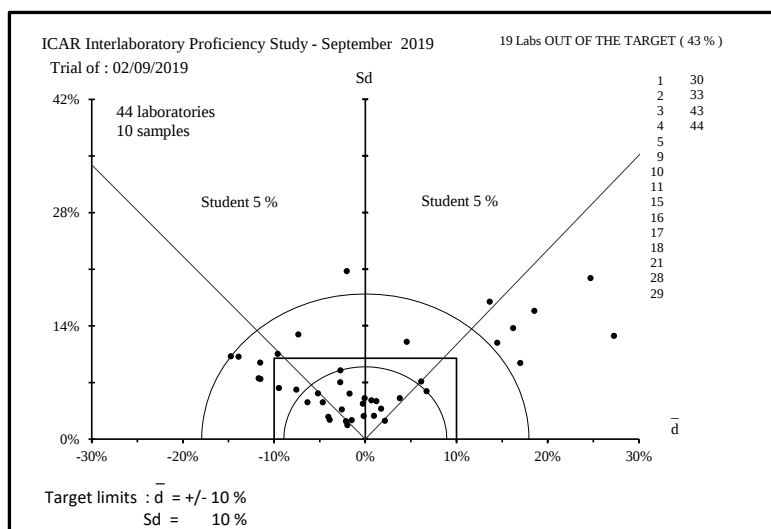


Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).

LIST OF THE PARTICIPANTS ICAR
ICAR PROFICIENCY TEST
RAW MILK
SOMATIC CELL CONTENT
September 2019

Name	City	Country
Actalia	Poligny	France
Agroscope Institute for food Sciences IFS	Bern-Liebefeld	Switzerland
Ao Agroplen	Moscow	RU
Cattle Information Service (CIS)	Teiford	England
Center for Green dairy Technology	HanKyong	Korea
Central Milk Lab ICBA	Caesarea	Israel
Chemometec	Allerød	Denmark
COOPRINSEM	Osorno	Chile
Croatian Agricultural Agency	Krizevci	Croatia
Deltamune	Pretoria	South Africa
Direction de l' Amelioration Genetique	Sidi Thabet	Tunisie
EPTA Laboratories LTD	Nicosia	Cyprus
Eurofins Steins Laboratory A/B	Jönköping	Sweden
Eurofins Steins Laboratory A/S	Vejen	Denmark
Int. National de tecnologia Industrial (INTI)	Rafaela Santa Fe	Argentina
Lab Agroalimentario de Santander	Santander Cantabria	Spain
Laboratorija Poljoprivredni fakultet Novi Sad,	Novi Sad	Serbia
Laboratorio Standard Latte	Maccarese (Roma)	Italy
Laboratorium Oceny Mleka KCHZ Parzniewie	Pruszkow	Poland
Merieux Nutriscience South Africa	Cape Town	South Africa
Merieux Nutriscience South Africa (Midrand)	Midrand	South Africa
Milchprüfing Baden-Württemberg e.V.	Kirchheim unter Teck	Germany
Osuuskunta Satamaito, laboratorio	Pori	Finland
Pieno Tyrimai	Kaunas	Lithuania
Plemenárske služby SR s.p.	Zilina	Slovakia
Qlip B.V.,	Zutphen	Netherlands
Suisselab AG	Zullikofen	Switzerland
Taiwan Livestock research Insitute	Taiwan	Taiwan
Tine Ramelklaboratoriet Heimdal	Heimdal	Norway
Univ. of Ljubljana dept. of Animal Sc. Inst. of Dairy Sc. and Probiotics	Domzale	Slovenia
University of Pretoria	Pretoria	South Africa
Lactanet-Québec	Quebec	Canada
Valio Ltd, Regional laboratory	Seinäjoki	Finland