



THE GLOBAL STANDARD  
FOR LIVESTOCK DATA

Network. Guidelines. Certification.

## ICAR PROFICIENCY TEST - SEPTEMBER 2017

Raw cow milk

Chemical “Reference” Methods





THE GLOBAL STANDARD  
FOR LIVESTOCK DATA

Network. Guidelines. Certification.

FRAME OF ACTIVITY :

ICAR MILK ANALYSES SUB-COMMITTEE (MA SC)

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## Introduction

Dear Participant,

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

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

The proficiency test is a tool to help evaluate the performance of the laboratory process and to support your laboratory quality assurance system. 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,H if all the information is reported correctly from the participant, then the cells are filled in green, otherwise they are highlighted in red for your attention, so you can review and verify any causal reasons internally. The control charts H will help you to follow your performance over the time.

- A) In table A you find your participation codes and the information if all the results from the samples received, have been sent to the PT provider.
- B) In table B is indicated if the results have been sent on time.
- C) In table C is indicated if the results have been reported in the correct unit of measurements.
- D) It is the ranking of your laboratory. The values of table 1 for each parameter are reported. In table D 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 Grubbs or Cochran 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<sub>PT</sub> (standard deviation calculated on this proficiency test) and the Z-Score<sub>FIX</sub> (standard deviation of the reference method) are summarized. If you have obtained all the -2<Z-Score results<+2 the cell will be filled in green. If you have obtained one or more results in the moderate or poor performance range the cells will be filled in yellow or red respectively.
- H) Your ZS Control Charts have been updated with data of the previous ICAR PT if you have participated. On the control charts the samples are ordered by concentration.

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 chaired by Dr. Gavin Scott (NZ). You find the statistical elaboration for all the ICAR interested parameters, fat, protein, lactose, urea and somatic cell.

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

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

ICAR 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 in September 2017 with at least one parameter is reported below and also available in ICAR website at:

<http://www.icar.org/index.php/certifications/milk-analysis-laboratories-certifications/results-of-the-milk-laboratories-proficiency-test-september-2017/>



Name of the laboratory	Country
Comité du Lait ASBL	Belgium
Department Valorisation des productions Agricoles	Belgium
ILVO	Belgium
Can West	Canada
Central Milk Testing Lab	Canada
Horizon Lab Ltd	Canada
Pacific Milk Analysis	Canada
Valacta - Centre d'Expertise en Production Laitière du Québec	Canada
COOPRINSEM	Chile
Dairy Cattle Research Center of Shandong Academy of Agricultural Sciences	China
Shanghai Dairy Breeding Center Co. Ltd	China
Croatian Agricultural Agency, Central Laboratory for Milk Quality Control	Croatia
University of Zagreb Faculty of Agriculture Dept. of Dairy Science	Croatia
Laborator pro rozbor mléka Brno, Ceskomoravská společnost chovatelů a.s.	Czech Republic
Laborator pro rozbor mléka Bustehrad, Ceskomoravská společnost chovatelů a.s.	Czech Republic
Milkom a.s Dairy research institute	Czech Republic
The National laboratory for raw milk Agrovyzkum Rapotin s.r.o.	Czech Republic
ChemoMetec A/S	Denmark
Eurofins Steins Laboratory A/S	Denmark



Name of the laboratory	Country
Cattle Information Service (CIS)	England
Osuuskunta Satamaito, laboratorio	Finland
Valio Oy/Seinajoen aluelaboratorio	Finland
Actalia	France
Milchprüfring Baden-Württemberg e.V.	Germany
Teagasc Food research Center	Ireland
Central Milk Lab ICBA	Israel
Laboratorio Standard Latte	Italy
Japan Dairy Technical Association	Japan
Pieno Tyrimai	Lithuania
Laboratorium Oceny Mleka KCHZ Laboratorium Referencyjne z/s w Parzniewie	Poland
Alip	Portugal
LRV-Laboratorio Regional de Veterinaria	Portugal
Holstein-Ro Breeders Association Laboratory	Romania
Laboratorija za ispitivanje kvaliteta mleka, Poljoprivredni fakultet Novi Sad	Serbia
Deltamune	South Africa
Merieux Nutriscience South Africa	South Africa
University of Pretoria, Faculty Vet Sciences, Dept. Production Animal Studies Milk Lab. Onderstepoort	South Africa
Lab Agroalimentario de Santander	Spain
LIGAL Edificio de Laboratorio Agrarios	Spain
Eurofins Steins Laboratory A/B	Sweden
Agroscope Institute for food Sciences IFS	Switzerland
SuisseLab AG	Switzerland
Taiwan Livestock research Institute	Taiwan
Qlip B.V.,	The Netherlands
Direction de l' Amelioration Genetique	Tunisia
Eastern Lab services	USA
Vetlab Agricultural Showgroups	Zambia

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  
RF0917

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

A	<b>Your participation Codes</b>					
	Subscription	<b>Fat<sub>ref</sub></b>	<b>Protein<sub>ref</sub></b>	<b>Lactose<sub>ref</sub></b>	<b>Urea<sub>ref</sub></b>	<b>SCC<sub>ref/alt</sub></b>
		Yes	Yes	Yes	Yes	Yes
Participation Codes	5	5	4	3	4	
Are all the sample results received?	Yes	Yes	Yes	Yes	Yes	Yes

### B Data results received on time

No	18-09-2017
----	------------

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

C		<b>Fat<sub>ref</sub></b>	<b>Protein<sup>*</sup><sub>ref</sub></b>	<b>Lactose<sub>ref</sub></b>	<b>Urea<sub>ref</sub></b>	<b>SCC<sub>ref/alt</sub></b>
		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

D		<b>Fat<sub>ref</sub></b>	<b>Protein<sup>*</sup><sub>ref</sub></b>	<b>Lactose<sub>ref</sub></b>	<b>Urea<sub>ref</sub></b>	<b>SCC<sub>ref/alt</sub></b>
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
Code	5	5	4	3	4	
%	69	69	80	60	45%	
d	-0.001	0.012	0.059	1.231	-14%	
Sd	0.019	0.020	0.098	0.392	11%	
D	0.019	0.023	0.115	1.292	18%	
Method	ISO 1211 IDF 1	ISO8968 IDF20	ISO26642 IDF214	ISO14637 IDF195	ISO13366-2 IDF148-2	

### Limits

d	0,020	0,025	0,10	2,5	10%
Sd	0,030	0,020	0,10	1,5	10%

### E Outliers

E		<b>Fat<sub>ref</sub></b>	<b>Protein<sup>*</sup><sub>ref</sub></b>	<b>Lactose<sub>ref</sub></b>	<b>Urea<sub>ref</sub></b>	<b>SCC<sub>ref/alt</sub></b>
		g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
Sample 1			Cochran	Cochran		
Sample 2				Cochran		
Sample 3			Cochran			
Sample 4			Cochran			
Sample 5			Cochran			
Sample 6						
Sample 7						
Sample 8						
Sample 9						
Sample 10						

## Repeatability

Your "r" performance

	Fat	Protein	Lactose	Urea	SCC
	g/100g	nitrogen g/100g	g/100g	mg/dl	SCC*1000/ml
Sample 1	0.010	0.070	0.077	0.660	2
Sample 2	0.010	0.007	0.056	0.430	19
Sample 3	0.010	0.049	0.003	0.030	20
Sample 4	0.010	0.085	0.016	0.160	2
Sample 5	0.010	0.073	0.021	0.690	11
Sample 6	0.000	0.021	0.012	0.070	42
Sample 7	0.010	0.002	0.023	0.730	0.000
Sample 8	0.010	0.031	0.025	1.320	1
Sample 9	0.010	0.021	0.038	0.520	3
Sample 10	0.020	0.004	0.013	0.330	4

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

## Limits

	Fat	Protein	Lactose	Urea	SCC
	g/100g	g/100g	g/100g	mg/dl	SCC*1000/ml
	ISO 1211 IDF 1D	ISO 8968 IDF 20	ISO 22662 IDF 198	ISO 14637 IDF 195	ISO 13366-2 IDF 148-2
	0,043	0,038	0,06	1,52	Level   r
					150   25
					300   42
					450   51
					750   64
					1500   126

## Your Z-Score PT

	Fat	Protein	Lactose	Urea	SCC
Sample 1	-0.294	0.223	-1.127	1.367	-0.90
Sample 2	1.745	-1.234	-1.139	1.380	-0.56
Sample 3	-0.517	2.406	0.456	1.290	-0.73
Sample 4	-0.180	1.474	1.162	0.970	-0.62
Sample 5	-0.348	1.937	0.668	0.559	-0.62
Sample 6	1.505	0.477	1.595	1.052	-0.69
Sample 7	-2.230	0.223	1.824	0.971	-0.55
Sample 8	-0.478	0.022	0.873	1.242	-0.55
Sample 9	-0.786	-0.043	1.264	1.270	-0.79
Sample 10	-0.213	0.997	2.400	1.900	-0.68

## Your Z-Score Fix

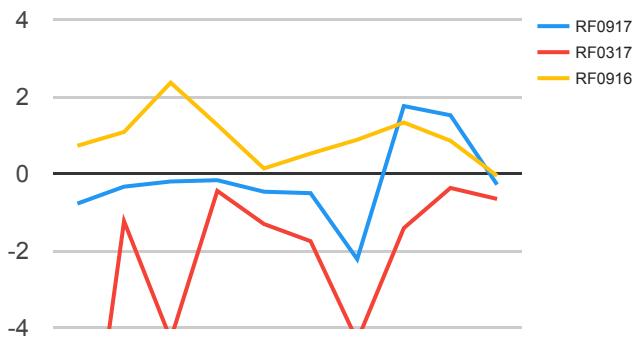
	Fat	Protein	Lactose	Urea	SCC
Sample 1	-0.117	0.267	-1.250	0.912	-2.26
Sample 2	2.022	-1.333	-3.545	0.786	-1.95
Sample 3	-0.182	2.588	0.992	0.673	-1.96
Sample 4	-0.100	1.440	1.493	0.907	-1.65
Sample 5	-0.175	1.796	1.695	0.324	-1.87
Sample 6	0.729	0.437	2.620	0.814	-2.34
Sample 7	-1.711	0.190	2.686	0.404	-1.79
Sample 8	-0.268	0.034	2.600	0.880	-2.15
Sample 9	-0.411	-0.074	2.444	0.474	-2.08
Sample 10	-0.180	1.122	2.825	0.628	-2.35

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

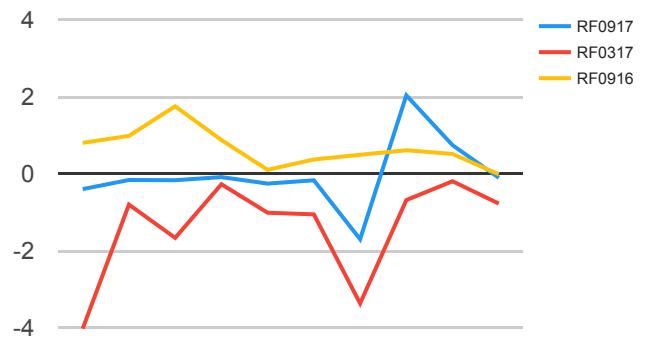
## Interpretation Z-Score

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

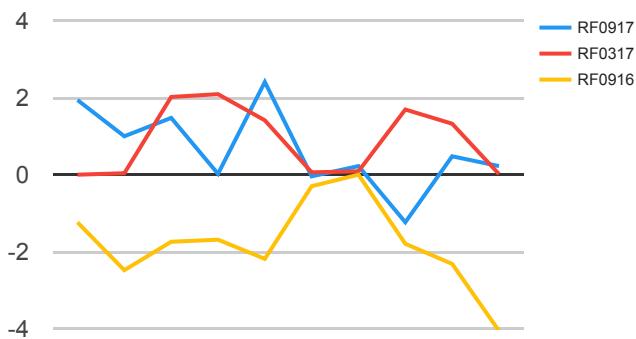
ZSCORE-PT - fat\_ref



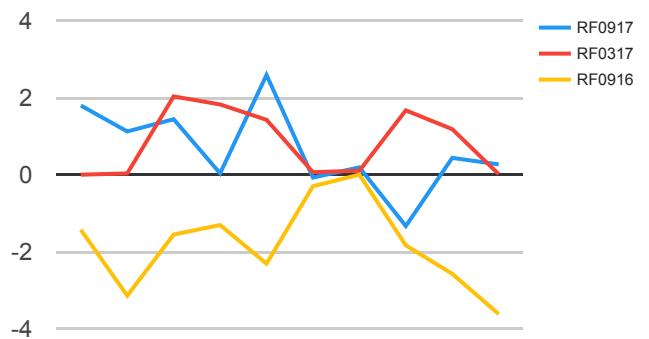
ZSCORE-FIX - fat\_ref



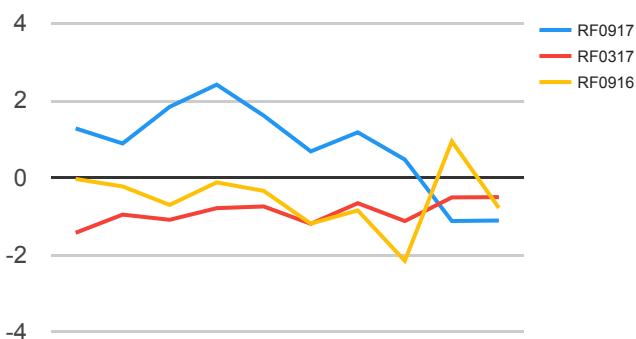
ZSCORE-PT - protein\_ref



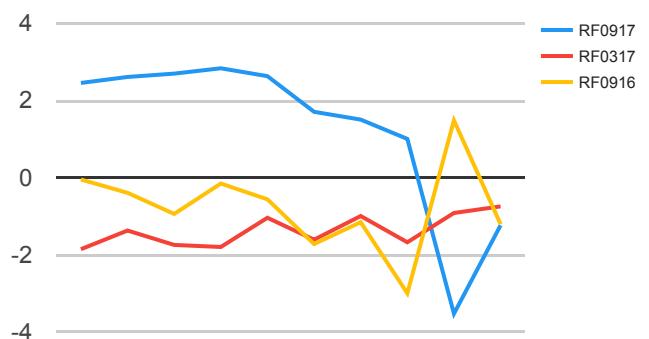
ZSCORE-FIX - protein\_ref

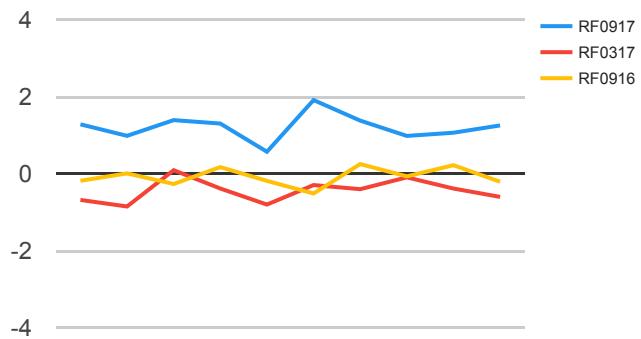
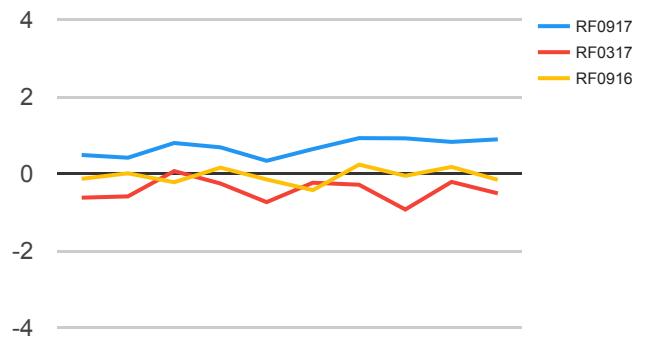
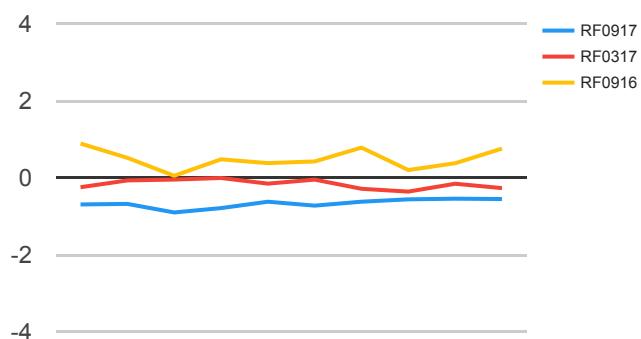
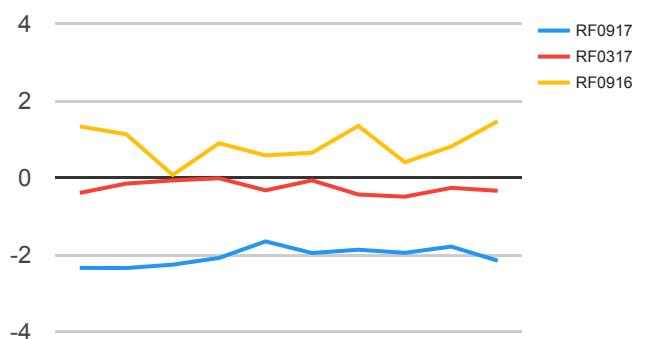


ZSCORE-PT - lactose\_ref



ZSCORE-FIX - lactose\_ref



**ZSCORE-PT - urea\_ref****ZSCORE-FIX - urea\_ref****ZSCORE-PT - scc\_ref****ZSCORE-FIX - scc\_ref**



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

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**September 2017**

**Raw Milk**

**Determination of FAT CONTENT**  
**Röse Gottlieb method**

Sending date of statistical treatment :      9<sup>th</sup> October 2017

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

Proficiency test accredited ISO 17043



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

Nb	%	N°	d	Sd	D	Method
1	6	12	- 0,000	0,004	0,004	A
2	13	11	- 0,001	0,005	0,005	A
3	19	15	+ 0,003	0,004	0,005	A
4	25	2	+ 0,002	0,005	0,005	A
5	31	9	- 0,003	0,005	0,006	A
6	38	14	- 0,002	0,006	0,006	A
7	44	13	- 0,003	0,008	0,009	A
8	50	3	+ 0,009	0,004	0,010	A
9	56	10	- 0,002	0,011	0,011	A
10	63	8	+ 0,007	0,010	0,012	A
11	69	5	- 0,001	0,019	0,019	A
12	75	6	- 0,006	0,021	0,022	B
13	81	4	- 0,026	0,031	0,041	A
14	88	16	- 0,037	0,023	0,044	A
15	94	7	+ 0,048	0,089	0,101	A
16	100	1	+ 0,059	0,159	0,170	A

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

+/- 0,020 g / 100 g for 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 16 laboratories using the reference method ISO 1211|IDF 1, after outliers discarding using Grubbs test at 5% risk level,

A ISO 1211 IDF 1D Röse Gottlieb Method

B ISO 2446 / IDF 226 Gerber method

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

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

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

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

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

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

S<sub>r<sub>PT</sub></sub> 0,007

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

S<sub>R<sub>PT</sub></sub> 0,014

**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	0,006	0,005	0,002	0,004	1,000 *	0,014	0,006	0,003	0,003	0,002	0,224	20
2	0,012	0,017	0,002	0,002	0,019	0,000	0,001	0,000	0,013	0,020	0,008	20
3	0,002	0,002	0,001	0,003	0,001	0,001	0,000	0,001	0,003	0,001	0,001	20
4	0,003	0,015	0,019	0,002	0,021	0,005	0,006	0,003	0,022	0,004	0,009	20
5	0,010	0,010	0,010	0,010	0,010	0,000	0,010	0,010	0,010	0,020	0,008	20
6	0,030	0,020	0,000	0,020	0,000	0,020	0,010	0,010	0,010	0,000	0,011	20
7	0,025	0,008	0,027	0,007	0,002	0,008	0,014	0,002	0,006	0,014	0,010	20
8	0,003	0,013	0,011	0,003	0,001	0,011	0,007	0,006	0,000	0,003	0,005	20
9	0,000	0,006	0,006	0,004	0,004	0,000	0,011	0,001	0,000	0,001	0,003	20
10	**	**	**	0,013	0,011	0,009	0,002	0,015	0,012	0,004	0,007	14
11	0,013	0,012	0,018	0,002	0,007	0,010	0,006	0,003	0,013	0,000	0,007	20
12	0,000	0,003	0,001	0,001	0,010	0,016	0,013	0,013	0,006	0,007	0,006	20
13	0,014	0,013	0,006	0,010	0,006	0,014	0,007	0,007	0,000	0,005	0,007	20
14	0,005	0,005	0,006	0,006	0,002	0,003	0,006	0,002	0,002	0,005	0,003	20
15	0,008	**	0,000	0,008	0,003	0,000	0,011	0,010	0,002	0,013	0,005	18
16	0,046 *	0,021	0,010	0,017	0,007	0,019	0,004	0,003	0,008	0,012	0,013	20
Sr	0,012	0,009	0,008	0,006	0,177	0,008	0,006	0,005	0,006	0,007		312
NE	30	28	30	32	32	32	32	32	32	32		
L	0,033	0,032	0,030	0,024	0,025	0,029	0,022	0,019	0,025	0,026		

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	4,742	4,123	3,480	2,750	2,640 *	4,459	3,752	3,091	1,501	2,456
2	4,757	4,113	3,466	2,737	2,137	4,465	3,737	3,080	1,473	2,455
3	4,760	4,120	3,477	2,746	2,138	4,471	3,746	3,091	1,486	2,456
4	4,739	4,080	3,467	2,724	2,120	4,356 *	3,720	3,053	1,471	2,407
5	4,755	4,145	3,465	2,735	2,125	4,480	3,705	3,075	1,465	2,440
6	4,725 *	4,090	3,480	2,710	2,120	4,460	3,775	3,095	1,455	2,430
7	5,038 *	4,218 *	3,500 *	2,745	2,151	4,463	3,738	3,083	1,473	2,469
8	4,761	4,099	3,458	2,751	2,142	4,487	3,745	3,092	1,484	2,450
9	4,762	4,106	3,470	2,740	2,124	4,463	3,730	3,080	1,464	2,435
10				2,733	2,121	4,451	3,752	3,069	1,471	2,457
11	4,761	4,115	3,466	2,739	2,129	4,458	3,736	3,079	1,469	2,439
12	4,762	4,105	3,463	2,737	2,128	4,460	3,740	3,078	1,479	2,446
13	4,758	4,104	3,471	2,721	2,119	4,477	3,745	3,068	1,469	2,438
14	4,753	4,098	3,460	2,734	2,119	4,468	3,739	3,089	1,476	2,448
15	4,763		3,471	2,744	2,137	4,462	3,744	3,085	1,479	2,441
16	4,682 *	4,048	3,426 *	2,681 *	2,091 *	4,424 *	3,727	3,070	1,468	2,407
M	4,756	4,103	3,469	2,736	2,129	4,466	3,739	3,080	1,474	2,442
REF.	4,757	4,105	3,469	2,737	2,128	4,465	3,739	3,080	1,473	2,444
SD	0,008	0,023	0,007	0,011	0,010	0,010	0,015	0,011	0,010	0,017

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 16 laboratories using the reference method ISO 12111 IDF 1, after outliers discarding using Grubbs test at 5 % risk level.

**Table IV :** Outlier identification

Sample	1	2	3	4	5	6	7	8	9	10
<b>Outliers Cochran</b>	16				1					
<b>Outlier Grubbs</b>	6; 7; 16	7	7; 16	7; 16	16	4; 16				
<b>sr</b>	0,006	0,009	0,006	0,006	0,007	0,007	0,006	0,005	0,006	0,007
<b>SR</b>	0,009	0,024	0,008	0,012	0,011	0,011	0,016	0,012	0,011	0,018

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

Sample Lab code	1	2	3	4	5	6	7	8	9	10	d	Sd <sub>lab</sub>	t
1	- 0,015	+ 0,018	+ 0,011	+ 0,013	+ 0,512	- 0,006	+ 0,013	+ 0,010	+ 0,027	+ 0,012	+ 0,059	0,159	1,18
2	- 0,000	+ 0,008	- 0,003	+ 0,000	+ 0,008	- 0,000	- 0,003	- 0,000	- 0,001	+ 0,011	+ 0,002	0,005	1,25
3	+ 0,003	+ 0,015	+ 0,008	+ 0,009	+ 0,009	+ 0,005	+ 0,007	+ 0,010	+ 0,012	+ 0,012	+ 0,009	0,004	7,64
4	- 0,019	- 0,025	- 0,002	- 0,013	- 0,009	- 0,110	- 0,019	- 0,028	- 0,002	- 0,037	- 0,026	0,031	2,66
5	- 0,002	+ 0,040	- 0,004	- 0,002	- 0,003	+ 0,015	- 0,034	- 0,005	- 0,008	- 0,004	- 0,001	0,019	0,13
6	- 0,032	- 0,015	+ 0,011	- 0,027	- 0,008	- 0,005	+ 0,036	+ 0,015	- 0,018	- 0,014	- 0,006	0,021	0,88
7	+ 0,280	+ 0,113	+ 0,031	+ 0,008	+ 0,023	- 0,002	- 0,001	+ 0,003	- 0,000	+ 0,025	+ 0,048	0,089	1,71
8	+ 0,003	- 0,006	- 0,011	+ 0,014	+ 0,013	+ 0,021	+ 0,005	+ 0,012	+ 0,011	+ 0,006	+ 0,007	0,010	2,21
9	+ 0,005	+ 0,001	+ 0,001	+ 0,003	- 0,004	- 0,002	- 0,010	- 0,001	- 0,009	- 0,009	- 0,003	0,005	1,49
10				- 0,004	- 0,008	- 0,015	+ 0,013	- 0,012	- 0,002	+ 0,013	- 0,002	0,011	0,51
11	+ 0,003	+ 0,010	- 0,003	+ 0,002	+ 0,000	- 0,007	- 0,003	- 0,002	- 0,005	- 0,005	- 0,001	0,005	0,55
12	+ 0,004	+ 0,001	- 0,006	+ 0,000	- 0,001	- 0,005	+ 0,000	- 0,002	+ 0,005	+ 0,003	- 0,000	0,004	0,07
13	+ 0,001	- 0,001	+ 0,002	- 0,016	- 0,009	+ 0,012	+ 0,005	- 0,013	- 0,004	- 0,006	- 0,003	0,008	1,11
14	- 0,005	- 0,007	- 0,009	- 0,003	- 0,009	+ 0,002	- 0,000	+ 0,009	+ 0,003	+ 0,004	- 0,002	0,006	0,84
15	+ 0,006		+ 0,002	+ 0,007	+ 0,008	- 0,003	+ 0,004	+ 0,005	+ 0,006	- 0,003	+ 0,003	0,004	2,51
16	- 0,076	- 0,056	- 0,043	- 0,056	- 0,038	- 0,041	- 0,012	- 0,011	- 0,005	- 0,036	- 0,037	0,023	5,24
d	- 0,001	- 0,001	- 0,000	- 0,001	+ 0,001	+ 0,000	+ 0,000	- 0,001	+ 0,001	- 0,002	+ 0,003	0,051	
Sd	0,008	0,023	0,007	0,011	0,010	0,010	0,015	0,011	0,010	0,017	0,013		

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

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

**ISO 1211 | IDF 1 : Precision of the method :** Sr = 0,016 g / 100 g  
SR = 0,020 g / 100 g

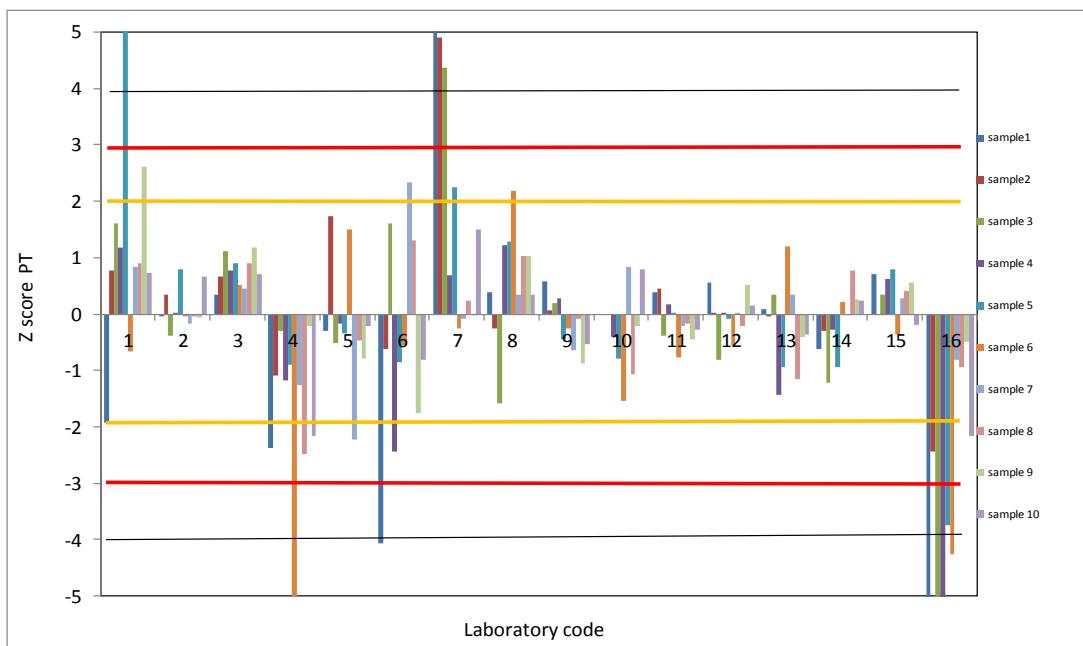
**Table VI :** Zscore of the different laboratories for each sample.  
ZS calculated on the PT standard deviation

Sample Lab code \ Lab code	1	2	3	4	5	6	7	8	9	10
1	-1,93	+0,77	+1,61	+1,17	+50,93	-0,66	+0,83	+0,90	+2,61	+0,74
2	-0,04	+0,34	-0,37	+0,00	+0,80	-0,04	-0,18	-0,03	-0,07	+0,68
3	+0,33	+0,67	+1,11	+0,77	+0,90	+0,52	+0,44	+0,90	+1,18	+0,71
4	-2,37	-1,08	-0,30	-1,17	-0,90	-11,35	-1,25	-2,49	-0,21	-2,17
5	-0,29	+1,74	-0,52	-0,18	-0,35	+1,51	-2,23	-0,48	-0,79	-0,21
6	-4,07	-0,63	+1,61	-2,43	-0,85	-0,56	+2,33	+1,31	-1,74	-0,81
7	+35,25	+4,89	+4,38	+0,68	+2,24	-0,25	-0,08	+0,24	-0,02	+1,51
8	+0,40	-0,26	-1,58	+1,22	+1,30	+2,18	+0,34	+1,04	+1,03	+0,35
9	+0,59	+0,06	+0,19	+0,27	-0,45	-0,25	-0,63	-0,08	-0,88	-0,54
10				-0,41	-0,80	-1,54	+0,83	-1,06	-0,21	+0,79
11	+0,40	+0,45	-0,37	+0,18	+0,00	-0,77	-0,21	-0,17	-0,45	-0,27
12	+0,56	+0,03	-0,82	+0,01	-0,08	-0,55	+0,02	-0,21	+0,51	+0,16
13	+0,08	-0,05	+0,33	-1,44	-0,94	+1,20	+0,34	-1,15	-0,40	-0,36
14	-0,61	-0,30	-1,23	-0,27	-0,94	+0,21	-0,01	+0,77	+0,27	+0,23
15	+0,71		+0,33	+0,63	+0,80	-0,35	+0,28	+0,41	+0,55	-0,18
16	-9,51	-2,43	-6,04	-5,03	-3,74	-4,26	-0,80	-0,95	-0,50	-2,15

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

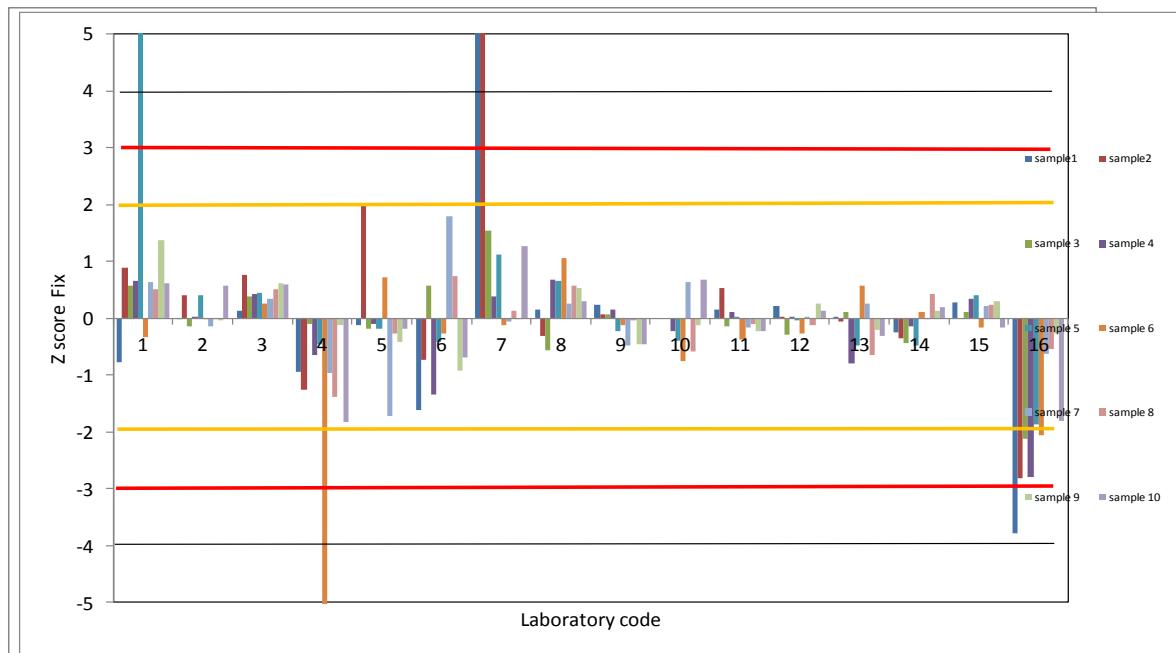
Sample Lab code	1	2	3	4	5	6	7	8	9	10
1	-0,77	+0,90	+0,57	+0,65	+25,58	-0,32	+0,64	+0,51	+1,36	+0,62
2	-0,02	+0,40	-0,13	+0,00	+0,40	-0,02	-0,14	-0,02	-0,04	+0,57
3	+0,13	+0,77	+0,39	+0,43	+0,45	+0,25	+0,34	+0,51	+0,61	+0,60
4	-0,94	-1,25	-0,11	-0,65	-0,45	-5,50	-0,96	-1,39	-0,11	-1,83
5	-0,12	+2,02	-0,18	-0,10	-0,17	+0,73	-1,71	-0,27	-0,41	-0,18
6	-1,62	-0,73	+0,57	-1,35	-0,42	-0,27	+1,79	+0,73	-0,91	-0,68
7	+14,01	+5,67	+1,54	+0,38	+1,13	-0,12	-0,06	+0,13	-0,01	+1,27
8	+0,16	-0,30	-0,56	+0,68	+0,65	+1,05	+0,26	+0,58	+0,54	+0,30
9	+0,23	+0,07	+0,07	+0,15	-0,22	-0,12	-0,49	-0,04	-0,46	-0,45
10				-0,22	-0,40	-0,75	+0,64	-0,59	-0,11	+0,67
11	+0,16	+0,52	-0,13	+0,10	+0,00	-0,37	-0,16	-0,09	-0,24	-0,23
12	+0,22	+0,03	-0,29	+0,00	-0,04	-0,27	+0,02	-0,12	+0,26	+0,14
13	+0,03	-0,05	+0,12	-0,80	-0,47	+0,58	+0,26	-0,64	-0,21	-0,30
14	-0,24	-0,35	-0,43	-0,15	-0,47	+0,10	-0,01	+0,43	+0,14	+0,20
15	+0,28		+0,12	+0,35	+0,40	-0,17	+0,21	+0,23	+0,29	-0,15
16	-3,78	-2,81	-2,13	-2,79	-1,88	-2,06	-0,62	-0,53	-0,26	-1,81

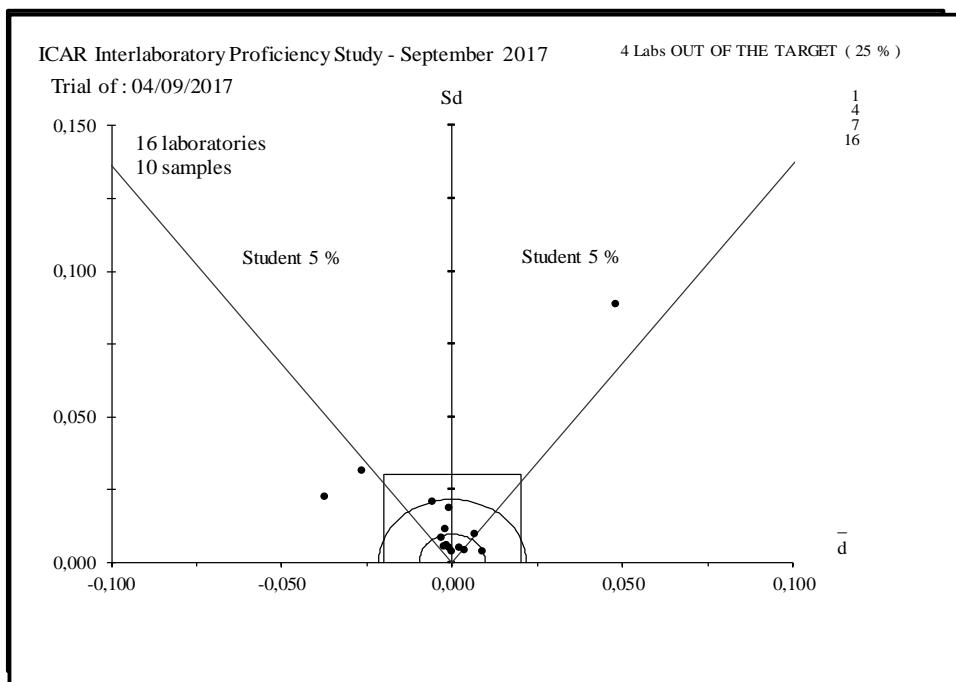
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=0,02

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  
September 2017

Name	City	Country
Actalia	Poligny	France
Agroscope	3003 Bern-Liebefeld	Switzerland
Central Milk Lab ICBA	Caesarea	IL
Department Valorisation des productions Agricoles	Gembloix	Belgium
Eastern Lab services	Medina	USA
ILVO	Melle	Belgium
Japan Dairy Technical Association	Tokyo	Japan
Lab Agroalimentario de Santander	Santander Cantabria	Spain
Laboratorio Standard Latte	Maccarese	Italy
Laboratorium Oceny Mleka KCHZ Parzniewie	Pruszkow	Poland
Milchprüfung Baden-Württemberg e.V.	Kirchheim unter Teck	Germany
Pieno Tyrimai	Kaunas	Lithuania
Qlip B.V.,	Zutphen	NL
Teagasc Food research Center	Cork	IR
Nat. Lab. for raw milk Agrovyzkum Rapotin s.r.o.	Vikyrovice	Czech Republic
Valacta -	Quebec	Canada



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

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**September 2017**

**Raw Milk**

**Determination of CRUDE PROTEIN CONTENT**  
**KJELDAHL Method**

Sending date of statistical treatment : 9<sup>th</sup> October 2017

Frame of activity :	ICAR Milk Analyses Sub Committee (MA SC)
Contact :	Gavin Scott Silvia Orlandini
ICAR Staff	gavin@milktest.co.nz 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	6	2	+ 0,002	0,005	0,006
2	13	7	+ 0,004	0,008	0,008
3	19	9	- 0,007	0,006	0,009
4	25	10	- 0,008	0,007	0,011
5	31	8	- 0,011	0,008	0,014
6	38	11	+ 0,010	0,013	0,016
7	44	4	- 0,017	0,008	0,019
8	50	14	+ 0,019	0,008	0,021
9	56	1	+ 0,002	0,022	0,022
10	63	12	- 0,017	0,014	0,022
11	69	5	+ 0,012	0,020	0,023
12	75	13	- 0,008	0,022	0,024
13	81	15	+ 0,023	0,012	0,026
14	88	6	+ 0,011	0,026	0,028
15	94	16	- 0,050	0,031	0,059
16	100	3	- 0,054	0,201	0,208

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 15 laboratories using the reference method (ISO 8968-1|IDF 20-1), after outlier discarding using Grubbs test at 5% risk level

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

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

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

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

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

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

S<sub>r<sub>PT</sub></sub> 0,007

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

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

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

Sample Lab code	1	2	3	4	5	6	7	8	9	10	Sr	NL
1	0,001	0,001	0,001	0,000	0,001	0,008	0,008	0,002	0,003	0,002	0,003	20
2	0,010	0,006	0,001	0,001	0,000	0,001	0,001	0,000	0,008	0,003	0,003	20
3	0,006	0,006	0,006	0,006	0,006	0,000	0,006	0,000	0,006	0,000	0,004	20
4	0,012	0,009	0,011	0,002	0,008	0,009	0,000	0,006	0,015	0,002	0,006	20
5	0,070 *	0,007	0,049 *	0,085 *	0,073 *	0,021	0,002	0,031	0,021	0,004	0,033	20
6	0,002	0,048 *	0,018	0,057 *	0,005	0,093 *	0,004	0,009	0,004	0,053 *	0,030	20
7	0,006	0,007	0,004	0,003	0,013	0,009	0,027	0,001	0,010	0,020	0,009	20
8	0,003	0,002	0,021	0,017	0,008	0,006	0,016	0,007	0,000	0,001	0,008	20
9	0,004	0,006	0,003	0,002	0,008	0,004	0,005	0,006	0,005	0,003	0,004	20
10	0,015	0,006	0,002	0,003	0,009	0,012	0,000	0,003	0,007	0,005	0,005	20
11	0,008	0,006	0,001	0,006	0,006	0,006	0,015	0,001	0,003	0,000	0,005	20
12	0,003	0,013	0,002	0,010	0,004	0,001	0,011	0,008	0,026	0,008	0,008	20
13	0,033 *	0,008	0,017	0,014	0,000	0,013	0,006	0,011	0,002	0,005	0,010	20
14	0,002	0,001	0,005	0,008	0,004	0,002	0,004	0,006	0,008	0,003	0,003	20
15	0,006	0,006	0,024	0,010	0,012	0,011	0,001	0,034	0,003	0,017	0,011	20
16	0,010	0,014	0,065 *	0,005	0,006	0,102 *	0,027	0,003	0,002	0,030 *	0,029	20
Sr	0,014	0,010	0,016	0,019	0,014	0,025	0,008	0,009	0,007	0,012		320
NE	32	32	32	32	32	32	32	32	32	32		
L	0,020	0,020	0,030	0,021	0,019	0,024	0,032	0,034	0,028	0,021		

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 / IDF 20

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

(\*) : Data have been modified in right units.

Sample Lab code	1	2	3	4	5	6	7	8	9	10	
(*)	1	3,817	3,593	3,316	2,973	2,792	3,717	3,518	3,161	3,294	2,890
	2	3,798	3,611	3,322	2,951	2,766	3,720	3,536	3,150	3,347	2,871
	3	3,825	3,627	3,327	2,964	2,772	3,720	3,531	3,152	2,715 *	2,877
	4	3,774	3,589	3,319	2,926	2,745	3,700	3,508	3,131	3,336	2,853
	5	3,800	3,583	3,372	2,979	2,801	3,719	3,531	3,150	3,338	2,897
	6	3,806	3,629	3,343	2,924	2,784	3,689	3,530	3,214	3,356	2,890
	7	3,800	3,625	3,325	2,954	2,764	3,711	3,526	3,149	3,355	2,881
	8	3,795	3,602	3,306	2,943	2,745	3,700	3,505	3,141	3,333	2,867
	9	3,792	3,612	3,320	2,946	2,758	3,699	3,514	3,136	3,337	2,870
	10	3,778	3,606	3,323	2,949	2,761	3,690	3,514	3,138	3,340	2,872
	11	3,803	3,620	3,326	2,955	2,766	3,719	3,542	3,151	3,355	2,918
	12	3,783	3,596	3,318	2,935	2,755	3,703	3,515	3,135	3,283	2,857
	13	3,758	3,578	3,287	2,947	2,762	3,695	3,543	3,159	3,366	2,882
	14	3,823	3,639	3,348	2,966	2,778	3,733	3,548	3,165	3,364	2,882
	15	3,798	3,620	3,344	2,982	2,788	3,750	3,556	3,188	3,373	2,885
	16	3,747	3,576	3,311	2,879 *	2,700 *	3,723	3,438 *	3,081	3,268	2,828
M		3,793	3,607	3,325	2,953	2,769	3,712	3,528	3,150	3,336	2,876
REF.		3,795	3,607	3,325	2,953	2,769	3,711	3,528	3,150	3,340	2,877
SD		0,022	0,019	0,019	0,018	0,017	0,016	0,015	0,028	0,031	0,020

M = mean per sample

REF. = reference values

SD = standard deviation per sample

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

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528,  
of 15 laboratories using the reference method ISO 8968-1 | 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
<b>Outliers Cochran</b>	5; 13	6	5; 16	5; 6	5	6; 16				6; 16
<b>Outlier Grubbs</b>				16	16		16		3	
<b>sr</b>	0,005	0,005	0,008	0,006	0,005	0,007	0,007	0,009	0,007	0,006
<b>SR</b>	0,021	0,020	0,017	0,016	0,015	0,017	0,016	0,029	0,032	0,017

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

Sample Lab code	1	2	3	4	5	6	7	8	9	10	d	Sd <sub>lab</sub>	t
1	+ 0,023	- 0,013	- 0,008	+ 0,020	+ 0,023	+ 0,006	- 0,010	+ 0,011	- 0,046	+ 0,013	+ 0,002	0,022	0,27
2	+ 0,003	+ 0,004	- 0,003	- 0,002	- 0,003	+ 0,009	+ 0,009	+ 0,000	+ 0,007	- 0,006	+ 0,002	0,005	1,21
3	+ 0,030	+ 0,020	+ 0,002	+ 0,011	+ 0,003	+ 0,009	+ 0,004	+ 0,002	- 0,625	+ 0,001	- 0,054	0,201	0,86
4	- 0,021	- 0,018	- 0,006	- 0,027	- 0,024	- 0,010	- 0,020	- 0,019	- 0,003	- 0,024	- 0,017	0,008	6,87
5	+ 0,005	- 0,024	+ 0,047	+ 0,026	+ 0,032	+ 0,008	+ 0,003	+ 0,001	- 0,001	+ 0,020	+ 0,012	0,020	1,84
6	+ 0,011	+ 0,022	+ 0,018	- 0,029	+ 0,015	- 0,022	+ 0,003	+ 0,065	+ 0,017	+ 0,013	+ 0,011	0,026	1,39
7	+ 0,005	+ 0,019	- 0,000	+ 0,001	- 0,005	- 0,000	- 0,002	- 0,000	+ 0,016	+ 0,004	+ 0,004	0,008	1,55
8	+ 0,001	- 0,005	- 0,018	- 0,009	- 0,024	- 0,011	- 0,023	- 0,008	- 0,007	- 0,010	- 0,011	0,008	4,60
9	- 0,003	+ 0,006	- 0,004	- 0,007	- 0,011	- 0,012	- 0,013	- 0,013	- 0,002	- 0,007	- 0,007	0,006	3,49
10	- 0,017	- 0,001	- 0,002	- 0,004	- 0,008	- 0,021	- 0,013	- 0,012	- 0,000	- 0,005	- 0,008	0,007	3,66
11	+ 0,008	+ 0,014	+ 0,001	+ 0,002	- 0,003	+ 0,009	+ 0,014	+ 0,001	+ 0,015	+ 0,041	+ 0,010	0,013	2,57
12	- 0,011	- 0,010	- 0,007	- 0,018	- 0,014	- 0,008	- 0,012	- 0,014	- 0,056	- 0,020	- 0,017	0,014	3,79
13	- 0,037	- 0,029	- 0,038	- 0,006	- 0,007	- 0,016	+ 0,015	+ 0,010	+ 0,026	+ 0,006	- 0,008	0,022	1,08
14	+ 0,028	+ 0,032	+ 0,023	+ 0,013	+ 0,010	+ 0,023	+ 0,020	+ 0,015	+ 0,024	+ 0,006	+ 0,019	0,008	7,40
15	+ 0,003	+ 0,014	+ 0,019	+ 0,029	+ 0,020	+ 0,039	+ 0,028	+ 0,039	+ 0,033	+ 0,009	+ 0,023	0,012	5,91
16	- 0,048	- 0,030	- 0,014	- 0,074	- 0,069	+ 0,012	- 0,089	- 0,069	- 0,072	- 0,049	- 0,050	0,031	5,06
d	- 0,001	- 0,000	+ 0,001	- 0,000	+ 0,000	+ 0,001	+ 0,000	+ 0,001	- 0,003	- 0,001	- 0,006	0,054	
Sd	0,022	0,019	0,019	0,018	0,017	0,016	0,015	0,028	0,031	0,020	0,021		

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

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

**ISO 8968-1 | IDF 20-1 : Precision of the method :** Sr = 0,014 g / 100 g  
SR = 0,018 g / 100 g

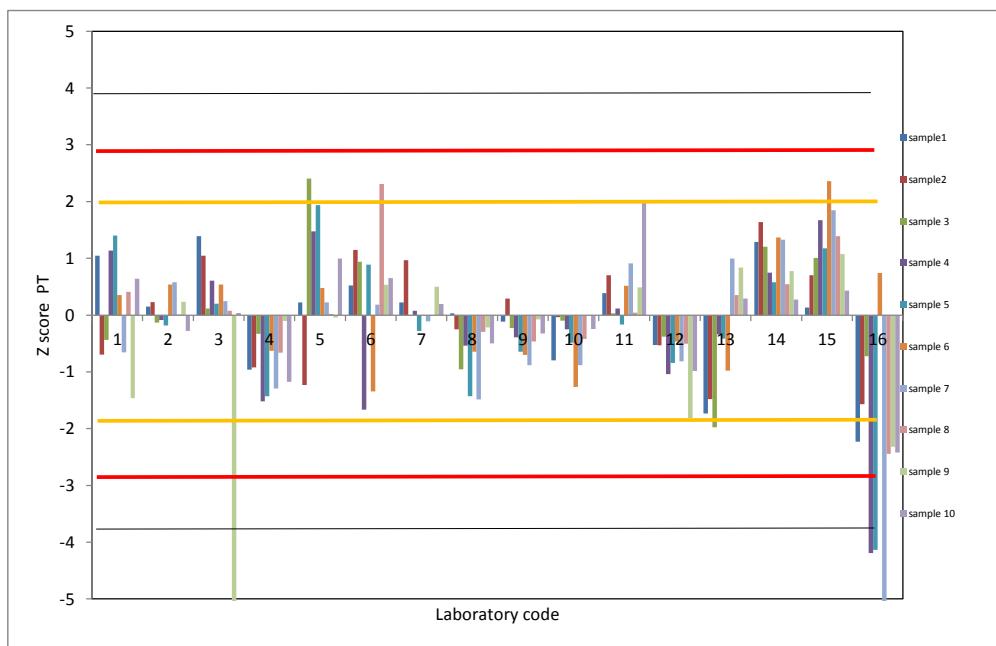
**Table VI :** Zscore of the different laboratories for each sample.  
ZS calculated on the PT standard deviation

Sample lab code \ Sample lab code	1	2	3	4	5	6	7	8	9	10
1	+1,04	-0,69	-0,44	+1,14	+1,40	+0,35	-0,66	+0,41	-1,46	+0,64
2	+0,15	+0,23	-0,13	-0,09	-0,18	+0,54	+0,58	+0,01	+0,23	-0,28
3	+1,39	+1,05	+0,12	+0,60	+0,20	+0,54	+0,24	+0,08	-20,08	+0,04
4	-0,96	-0,92	-0,33	-1,52	-1,43	-0,63	-1,30	-0,66	-0,10	-1,18
5	+0,22	-1,23	+2,41	+1,47	+1,94	+0,48	+0,22	+0,02	-0,04	+1,00
6	+0,52	+1,15	+0,94	-1,66	+0,89	-1,34	+0,18	+2,31	+0,53	+0,65
7	+0,22	+0,97	-0,02	+0,08	-0,28	-0,01	-0,11	-0,01	+0,50	+0,19
8	+0,03	-0,25	-0,95	-0,54	-1,43	-0,65	-1,48	-0,30	-0,22	-0,50
9	-0,12	+0,29	-0,23	-0,39	-0,64	-0,70	-0,88	-0,47	-0,07	-0,33
10	-0,80	-0,04	-0,10	-0,25	-0,49	-1,27	-0,88	-0,42	-0,00	-0,25
11	+0,39	+0,70	+0,03	+0,11	-0,17	+0,52	+0,91	+0,04	+0,49	+2,02
12	-0,53	-0,54	-0,38	-1,04	-0,85	-0,46	-0,81	-0,51	-1,82	-0,98
13	-1,73	-1,48	-1,98	-0,34	-0,41	-0,98	+0,99	+0,35	+0,84	+0,29
14	+1,29	+1,64	+1,20	+0,75	+0,58	+1,37	+1,33	+0,55	+0,78	+0,27
15	+0,13	+0,70	+1,01	+1,67	+1,17	+2,36	+1,85	+1,39	+1,07	+0,43
16	-2,23	-1,57	-0,72	-4,19	-4,13	+0,74	-5,84	-2,44	-2,32	-2,42

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 \ Sample Lab code	1	2	3	4	5	6	7	8	9	10
1	+1,25	-0,75	-0,47	+1,11	+1,30	+0,32	-0,56	+0,64	-2,53	+0,72
2	+0,18	+0,24	-0,14	-0,08	-0,17	+0,49	+0,49	+0,02	+0,40	-0,31
3	+1,67	+1,13	+0,12	+0,59	+0,18	+0,49	+0,21	+0,12	-34,72	+0,04
4	-1,15	-1,00	-0,35	-1,48	-1,32	-0,57	-1,10	-1,03	-0,18	-1,32
5	+0,27	-1,33	+2,59	+1,44	+1,80	+0,44	+0,19	+0,03	-0,07	+1,12
6	+0,62	+1,24	+1,01	-1,63	+0,82	-1,23	+0,15	+3,60	+0,92	+0,73
7	+0,27	+1,04	-0,02	+0,08	-0,26	-0,01	-0,09	-0,02	+0,87	+0,22
8	+0,04	-0,27	-1,03	-0,53	-1,32	-0,59	-1,26	-0,46	-0,38	-0,56
9	-0,14	+0,32	-0,25	-0,39	-0,60	-0,64	-0,75	-0,73	-0,13	-0,37
10	-0,96	-0,04	-0,11	-0,24	-0,45	-1,16	-0,75	-0,66	-0,00	-0,28
11	+0,46	+0,76	+0,04	+0,11	-0,15	+0,47	+0,78	+0,07	+0,85	+2,27
12	-0,63	-0,58	-0,41	-1,01	-0,78	-0,42	-0,69	-0,79	-3,14	-1,11
13	-2,07	-1,60	-2,13	-0,33	-0,38	-0,89	+0,85	+0,55	+1,45	+0,32
14	+1,54	+1,77	+1,29	+0,73	+0,54	+1,25	+1,13	+0,85	+1,34	+0,31
15	+0,16	+0,76	+1,08	+1,64	+1,09	+2,16	+1,57	+2,16	+1,86	+0,48
16	-2,67	-1,69	-0,78	-4,10	-3,83	+0,68	-4,97	-3,81	-4,01	-2,73

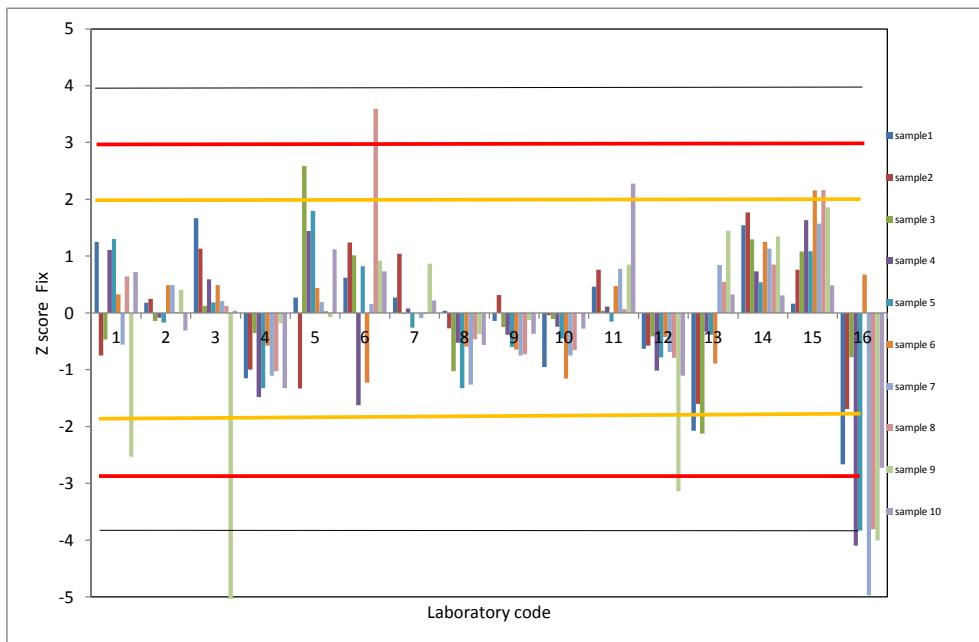
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=0,018

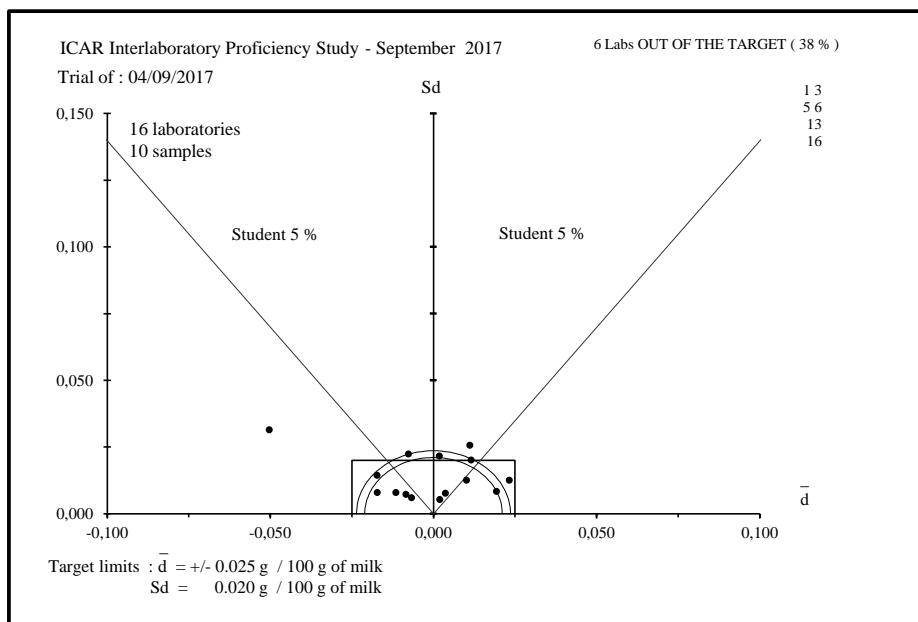
In yellow the values bigger or smaller than 2/-2

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

**Figure 3 :**

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



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

## LIST OF THE PARTICIPANTS ICAR

ICAR PROFICIENCY TEST  
RAW MILK  
NITROGEN CONTENT  
September 2017

Name	City	Country
Actalia	Poligny	France
Agroscope	Bern-Liebefeld	Switzerland
Central Milk Lab ICBA	Caesarea	IL
Department Valorisation des productions Agricoles	Gembloix	Belgium
Eastern Lab services	Medina	USA
ILVO	Melle	Belgium
Japan Dairy Technical Association	Tokyo	Japan
Lab Agroalimentario de Santander	Santander Cantabria	Spain
Laboratorio Standard Latte	Maccarese	Italy
Laboratorium Oceny Mleka KCHZ Laboratorium Referencyjne z/s w Parzniewie	Pruszkow	Poland
Milchprüfung Baden-Württemberg e.V.	Kirchheim unter Teck	Germany
Pieno Tyrimai	Kaunas	Lithuania
Qlip B.V.,	Zutphen	NL
Teagasc Food research Center	Cork	IR
The National laboratory for raw milk Agrovyzkum Rapotin s.r.o.	Vikyrovice	Czech Republic
Valacta - Centre d'Expertise en Production Laitière du Québec	Quebec	Canada



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

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**September 2017**

**Raw Milk**

**Determination of LACTOSE CONTENT**

Sending date of statistical treatment : 9<sup>th</sup> October 2017

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

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

Nb	%	N°	d	Sd	D	Method
1	7	13	- 0,010	0,020	0,023	IDF 214
2	13	5	+ 0,011	0,021	0,024	N.C.
3	20	9	+ 0,026	0,018	0,032	IDF 214
4	27	1	- 0,038	0,018	0,042	Own method
5	33	3	- 0,016	0,043	0,046	Enzymatic method
6	40	7	- 0,049	0,018	0,052	IDF 198
7	47	14	- 0,025	0,048	0,054	IDF 79B:1991
8	53	2	+ 0,056	0,023	0,061	Polarimeter
9	60	6	- 0,059	0,019	0,062	IDF 198
10	67	12	- 0,066	0,028	0,072	IDF 198
11	73	8	- 0,025	0,080	0,084	IDF 214
12	80	4	+ 0,059	0,098	0,115	IDF 214
13	87	10	+ 0,091	0,079	0,121	Lane-Eynon
14	93	15	+ 0,021	0,205	0,206	Polarimeter
15	100	11	+ 0,315	0,078	0,324	analyse à flux continu

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

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

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

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

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

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

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

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

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

S<sub>r<sub>PT</sub></sub> 0,014

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

S<sub>R<sub>PT</sub></sub> 0,091

**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	0,006	0,014	0,006	0,004	0,005	0,004	0,001	0,008	0,001	0,002	0,004	20
2	0,000	0,000	0,010	0,000	0,010	0,010	0,010	0,010	0,010	0,000	0,005	20
3	0,018	0,019	0,018	0,037	0,000	0,019	0,038	0,037	0,019	0,001	0,017	20
4	0,077 *	0,056 *	0,003	0,016	0,021	0,012	0,023	0,025	0,038	0,013	0,025	20
5	0,020	0,010	0,000	0,000	0,010	0,010	0,010	0,000	0,010	0,000	0,007	20
6	0,001	0,005	0,001	0,023	0,026	0,034	0,014	0,007	0,013	0,005	0,012	20
7	0,003	0,014	0,004	0,010	0,008	0,000	0,006	0,006	0,001	0,006	0,005	20
8	0,033	0,080 *	0,035	0,009	0,063 *	0,018	0,005	0,068	0,006	0,015	0,030	20
9	0,000	0,000	0,005	0,008	0,004	0,003	0,001	0,021	0,027	0,012	0,008	20
10	0,003	0,012	0,010	0,012	0,004	0,027	0,006	0,001	0,020	0,013	0,009	20
11	0,019	0,165 *	0,010	0,000	0,039	0,039	0,117 *	0,010	0,117 *	0,087 *	0,057	20
12	0,005	0,008	0,002	0,007	0,000	0,011	0,039	0,036	0,033	0,001	0,014	20
13	0,004	0,008	0,004	0,021	0,006	0,063	0,004	0,064	0,010	0,002	0,021	20
14	0,003	0,028	0,030	0,018	0,008	0,020	0,031	0,024	0,020	0,013	0,015	20
15	0,015	0,015	0,029	0,059 *	0,029	0,044	0,015	0,017	0,060	0,017	0,024	20
Sr	0,017	0,036	0,011	0,015	0,016	0,019	0,025	0,021	0,027	0,017		300
NE	30	30	30	30	30	30	30	30	30	30		
L	0,035	0,034	0,042	0,041	0,044	0,072	0,051	0,080	0,065	0,024		

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

(\*) : Data have been modified in the right units.

Sample lab code	1	2	3	4	5	6	7	8	9	10	
(*)	1	5,109	5,043	4,960	4,861	4,742	4,676	4,610	4,553	4,486	4,643
	2	5,230	5,170	5,075	4,970	4,835	4,765	4,695	4,625	4,555	4,700
	3	5,163	4,995	5,010	4,904	4,757	4,749	4,608	4,519	4,530	4,670
	4	5,072	4,914	5,075	4,982	4,882	4,830	4,764	4,714	4,635	4,783
	5	5,180	5,115	5,010	4,910	4,795	4,725	4,655	4,590	4,515	4,670
	6	5,082	5,026	5,005	4,834	4,719	4,628	4,583	4,521	4,468	4,609
	7	5,102	5,031	4,945	4,852	4,730	4,668	4,599	4,545	4,474	4,623
	8	5,192	5,147	5,125	4,939	4,696	4,627	4,577	4,481	4,430	4,590
	9	5,159	5,116	5,024	4,941	4,811	4,729	4,672	4,618	4,538	4,713
	10	5,141	5,097	5,007	5,012	4,979	4,836	4,798	4,770	4,672	4,659
	11	5,534 *	5,451	5,248	5,252 *	5,117	5,117 *	5,010 *	4,879	4,718	4,879 *
	12	5,116	5,008	4,917	4,826	4,705	4,641	4,596	4,530	4,464	4,594
	13	5,117	5,063	5,000	4,886	4,759	4,731	4,631	4,591	4,512	4,667
	14	5,048	5,011	4,960	4,868	4,739	4,715	4,666	4,647	4,505	4,655
	15	5,327 *	5,403	5,265	4,982	4,940	4,566	4,572	4,259	4,375	4,576
M	5,131	5,106	5,042	4,912	4,814	4,706	4,645	4,589	4,525	4,654	
REF.	5,130	5,081	5,028	4,912	4,802	4,707	4,637	4,591	4,520	4,650	
SD	0,052	0,146	0,102	0,060	0,119	0,077	0,069	0,140	0,091	0,055	

M = mean per sample

REF. = reference values

SD = standard deviation per sample

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

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

**Table IV :** Outlier identification

Sample	1	2	3	4	5	6	7	8	9	10
<b>Outliers Cochran</b>	4	4; 8; 11		15	8		11	8	11	11
<b>Outlier Grubbs</b>	11; 15			11		11	11			11
<b>sr</b>	0,009	0,009	0,011	0,011	0,012	0,018	0,014	0,021	0,017	0,007
<b>SR</b>	0,051	0,112	0,103	0,060	0,119	0,078	0,070	0,141	0,077	0,056

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

Sample Lab code	1	2	3	4	5	6	7	8	9	10	d	Sd <sub>lab</sub>	t
1	- 0,021	- 0,038	- 0,068	- 0,051	- 0,060	- 0,031	- 0,028	- 0,038	- 0,035	- 0,007	- 0,038	0,018	6,54
2	+ 0,100	+ 0,089	+ 0,047	+ 0,058	+ 0,033	+ 0,058	+ 0,058	+ 0,034	+ 0,035	+ 0,050	+ 0,056	0,023	7,85
3	+ 0,033	- 0,086	- 0,018	- 0,008	- 0,045	+ 0,042	- 0,029	- 0,073	+ 0,009	+ 0,020	- 0,016	0,043	1,14
4	- 0,059	- 0,167	+ 0,047	+ 0,070	+ 0,080	+ 0,123	+ 0,126	+ 0,122	+ 0,115	+ 0,133	+ 0,059	0,098	1,90
5	+ 0,050	+ 0,034	- 0,018	- 0,002	- 0,007	+ 0,018	+ 0,018	- 0,001	- 0,005	+ 0,020	+ 0,011	0,021	1,62
6	- 0,049	- 0,055	- 0,023	- 0,078	- 0,083	- 0,079	- 0,054	- 0,071	- 0,053	- 0,041	- 0,059	0,019	9,77
7	- 0,029	- 0,050	- 0,083	- 0,060	- 0,072	- 0,039	- 0,038	- 0,046	- 0,047	- 0,027	- 0,049	0,018	8,59
8	+ 0,062	+ 0,066	+ 0,097	+ 0,028	- 0,106	- 0,080	- 0,060	- 0,111	- 0,090	- 0,060	- 0,025	0,080	1,01
9	+ 0,029	+ 0,035	- 0,004	+ 0,029	+ 0,009	+ 0,022	+ 0,034	+ 0,026	+ 0,017	+ 0,063	+ 0,026	0,018	4,62
10	+ 0,010	+ 0,016	- 0,021	+ 0,100	+ 0,177	+ 0,129	+ 0,161	+ 0,178	+ 0,152	+ 0,009	+ 0,091	0,079	3,64
11	+ 0,404	+ 0,371	+ 0,220	+ 0,341	+ 0,315	+ 0,410	+ 0,372	+ 0,287	+ 0,198	+ 0,229	+ 0,315	0,078	12,75
12	- 0,015	- 0,073	- 0,111	- 0,086	- 0,097	- 0,066	- 0,042	- 0,061	- 0,057	- 0,056	- 0,066	0,028	7,61
13	- 0,013	- 0,018	- 0,028	- 0,026	- 0,043	+ 0,024	- 0,006	- 0,000	- 0,008	+ 0,017	- 0,010	0,020	1,58
14	- 0,083	- 0,070	- 0,068	- 0,044	- 0,063	+ 0,008	+ 0,028	+ 0,056	- 0,015	+ 0,005	- 0,025	0,048	1,63
15	+ 0,197	+ 0,323	+ 0,237	+ 0,071	+ 0,138	- 0,141	- 0,065	- 0,332	- 0,145	- 0,074	+ 0,021	0,205	0,32
d	+ 0,001	+ 0,025	+ 0,014	+ 0,000	+ 0,012	- 0,001	+ 0,007	- 0,002	+ 0,005	+ 0,004	+ 0,019	0,114	
Sd	0,052	0,146	0,102	0,060	0,119	0,077	0,069	0,140	0,091	0,055	0,097		

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

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

Sr = 0.022 g / 100 g

SR = 0.047 g / 100 g

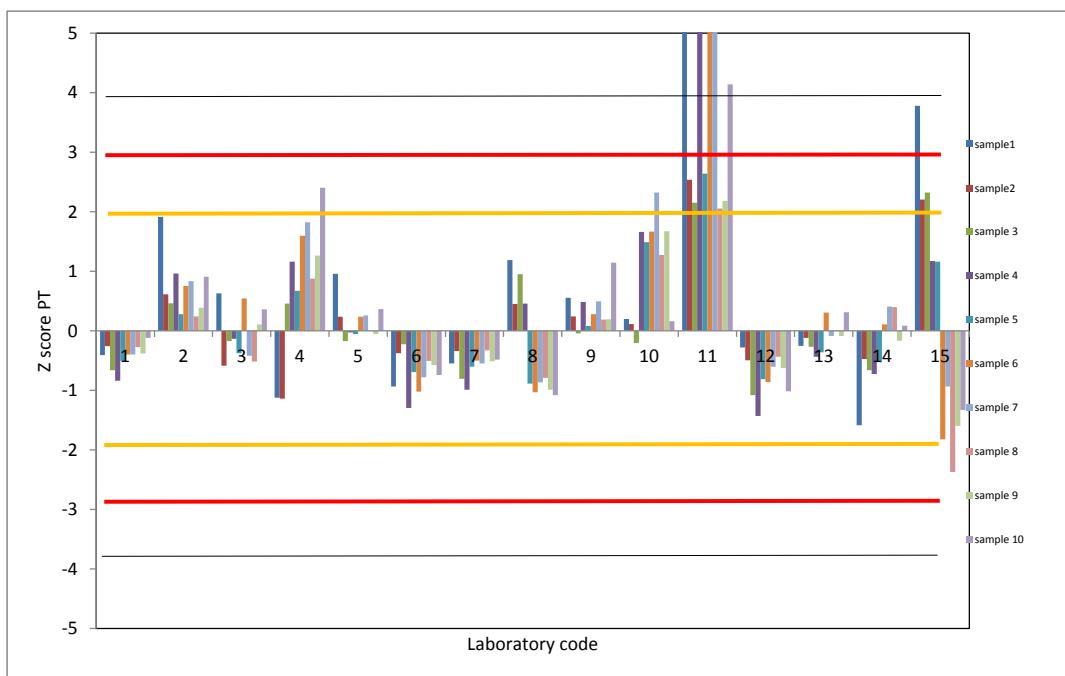
**Table VI :** Zscore of the different laboratories for each sample.  
ZS calculated on the PT standard deviation

Sample lab code	1	2	3	4	5	6	7	8	9	10
1	-0,41	-0,26	-0,66	-0,84	-0,51	-0,40	-0,40	-0,27	-0,38	-0,12
2	+1,91	+0,61	+0,46	+0,96	+0,28	+0,75	+0,83	+0,24	+0,38	+0,91
3	+0,63	-0,59	-0,17	-0,14	-0,38	+0,54	-0,42	-0,52	+0,10	+0,36
4	-1,13	-1,14	+0,46	+1,16	+0,67	+1,60	+1,82	+0,87	+1,26	+2,40
5	+0,95	+0,24	-0,17	-0,03	-0,06	+0,24	+0,26	-0,01	-0,06	+0,37
6	-0,94	-0,38	-0,23	-1,30	-0,69	-1,02	-0,78	-0,51	-0,58	-0,75
7	-0,55	-0,34	-0,81	-0,99	-0,60	-0,50	-0,55	-0,33	-0,51	-0,48
8	+1,19	+0,45	+0,95	+0,46	-0,89	-1,03	-0,87	-0,79	-0,99	-1,08
9	+0,55	+0,24	-0,04	+0,48	+0,08	+0,28	+0,49	+0,19	+0,19	+1,14
10	+0,20	+0,11	-0,20	+1,66	+1,49	+1,67	+2,32	+1,27	+1,67	+0,16
11	+7,75	+2,54	+2,15	+5,64	+2,64	+5,31	+5,38	+2,05	+2,18	+4,14
12	-0,28	-0,50	-1,08	-1,43	-0,81	-0,86	-0,60	-0,44	-0,62	-1,02
13	-0,25	-0,12	-0,27	-0,44	-0,36	+0,31	-0,09	-0,00	-0,09	+0,31
14	-1,59	-0,48	-0,66	-0,73	-0,53	+0,11	+0,41	+0,40	-0,17	+0,09
15	+3,78	+2,21	+2,32	+1,17	+1,16	-1,82	-0,94	-2,37	-1,60	-1,33

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	-0,45	-0,80	-1,44	-1,08	-1,28	-0,66	-0,59	-0,82	-0,74	-0,14
2	+2,12	+1,90	+1,00	+1,24	+0,71	+1,24	+1,23	+0,72	+0,74	+1,07
3	+0,70	-1,83	-0,38	-0,18	-0,95	+0,89	-0,62	-1,55	+0,20	+0,42
4	-1,25	-3,54	+0,99	+1,49	+1,69	+2,62	+2,69	+2,60	+2,44	+2,82
5	+1,06	+0,73	-0,38	-0,04	-0,15	+0,39	+0,38	-0,03	-0,11	+0,43
6	-1,04	-1,17	-0,50	-1,67	-1,76	-1,68	-1,15	-1,51	-1,12	-0,88
7	-0,61	-1,06	-1,76	-1,27	-1,53	-0,83	-0,81	-0,99	-0,99	-0,57
8	+1,32	+1,40	+2,07	+0,59	-2,26	-1,70	-1,28	-2,36	-1,92	-1,28
9	+0,61	+0,75	-0,09	+0,62	+0,19	+0,46	+0,73	+0,56	+0,37	+1,35
10	+0,22	+0,35	-0,44	+2,13	+3,77	+2,74	+3,42	+3,79	+3,23	+0,19
11	+8,59	+7,89	+4,67	+7,25	+6,69	+8,72	+7,92	+6,11	+4,22	+4,87
12	-0,31	-1,54	-2,36	-1,84	-2,06	-1,41	-0,89	-1,30	-1,20	-1,20
13	-0,28	-0,37	-0,59	-0,56	-0,91	+0,50	-0,13	-0,01	-0,17	+0,37
14	-1,76	-1,48	-1,44	-0,93	-1,34	+0,17	+0,60	+1,18	-0,32	+0,10
15	+4,19	+6,86	+5,05	+1,50	+2,94	-2,99	-1,38	-7,06	-3,09	-1,56

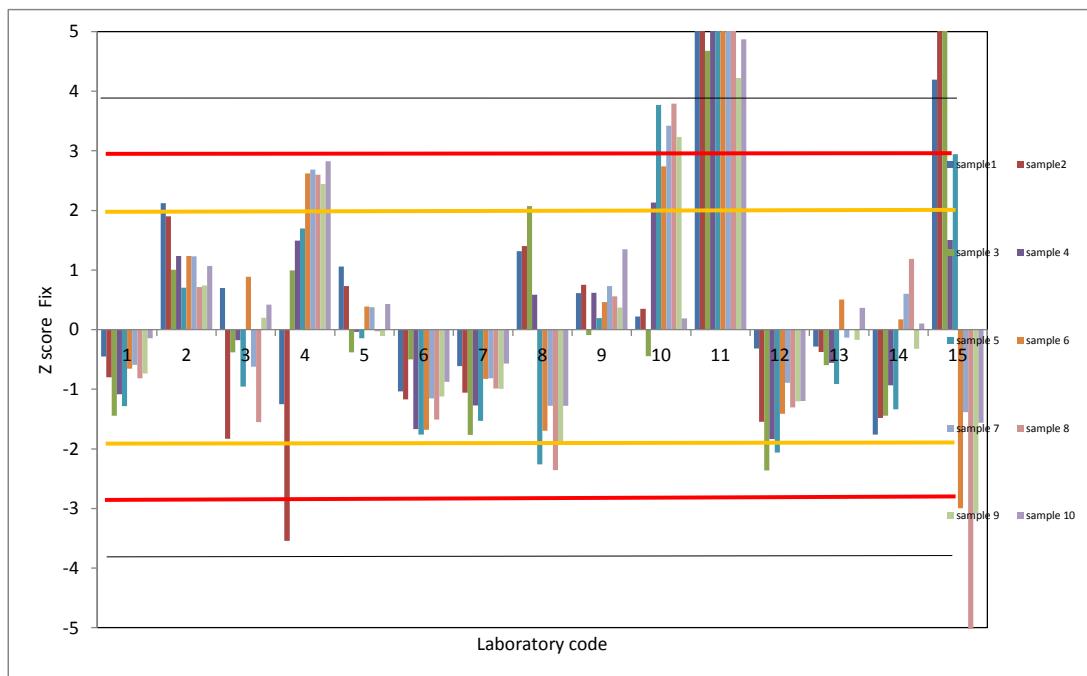
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=0,047

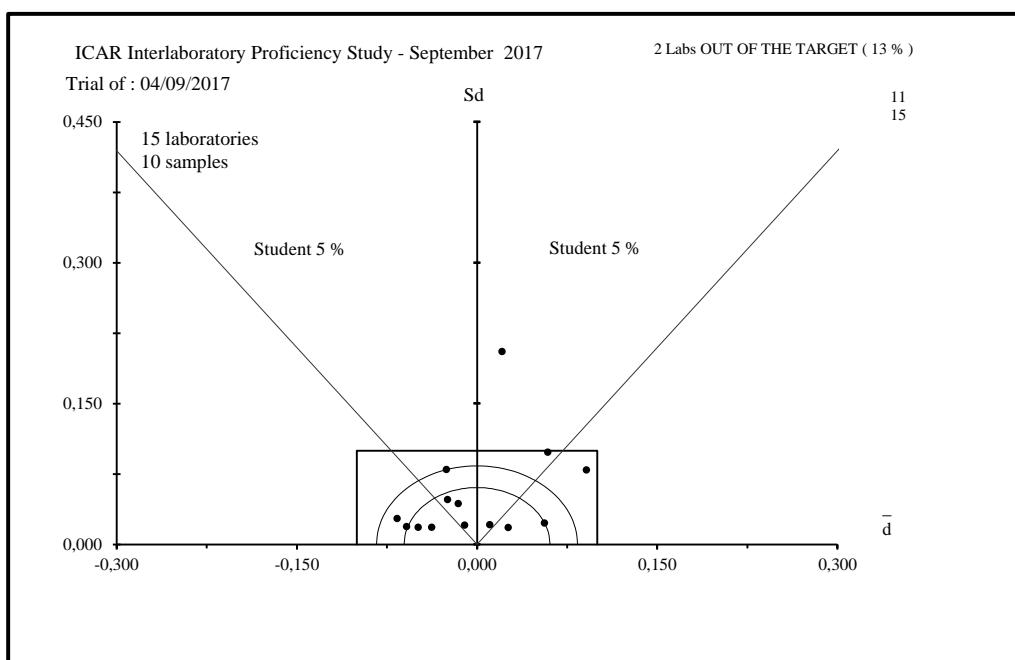
In yellow the values bigger or smaller than 2/-2

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  
September 2017

Name	City	Country
Agroscope	Bern-Liebefeld	Switzerland
Central Milk Lab ICBA	Caesarea	IL
Department Valorisation des productions Agricoles	Gembloux	Belgium
ILVO	Melle	Belgium
Japan Dairy Technical Association	Tokyo	Japan
Lab Agroalimentario de Santander	Santander Cantabria	Spain
Laboratorio Standard Latte	Maccarese	Italy
Laboratorium Oceny Mleka KCHZ Laboratorium Referencyjne z/s w Parzniewie	Pruszkow	Poland
Milchprüfung Baden-Württemberg e.V.	Kirchheim unter Teck	Germany
Milkom a.s Dairy research institute	Praha	Czech Republic
Pieno Tyrimai	Kaunas	Lithuania
Qlip B.V.,	Zutphen	NL
Teagasc Food research Center	Cork	IR
The National laboratory for raw milk Agrovyzkum Rapotin s.r.o.	Vikyrovice	Czech Republic
Valacta - Centre d'Expertise en Production Laitière du Québec	Quebec	Canada



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

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**September 2017**

**Raw Milk**

**Determination of UREA CONTENT**

Sending date of statistical treatment : 9<sup>th</sup> October 2017

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



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**Table I : Ranking of the laboratories**

Units : mg / 100 g

Nb	%	N°	d	Sd	D	Method
1	10	6	+ 0,20	0,36	0,41	IDF 195
2	20	2	+ 0,49	0,50	0,70	Photometric method
3	30	10	- 0,43	0,64	0,77	IDF 195
4	40	7	+ 0,68	0,37	0,78	IDF 195
5	50	8	+ 0,75	0,54	0,92	Continuous flow analyzer
6	60	3	+ 1,23	0,39	1,29	IDF 195
7	70	4	+ 0,49	1,29	1,38	IDF 195
8	80	5	+ 1,47	0,79	1,67	Continuous flow analyzer
9	90	1	- 1,72	0,32	1,75	IDF 195
10	100	9	- 0,93	11,19	11,23	IDF 195

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

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

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 7 laboratories using reference method (ISO 14657/IDF 195), after outlier discarding using Grubbs test at 5% risk level

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

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

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

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

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

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

S<sub>r</sub><sub>PT</sub> 0,57

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

S<sub>R</sub><sub>PT</sub> 1,19

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

Sample Lab Code \ Lab Code	1	2	3	4	5	6	7	8	9	10	Sr	NL
1	0,50	0,10	0,10	0,20	0,10	0,10	0,50	0,70	0,60	0,40	0,28	20
2	2,30	1,70 *	1,80	0,70	1,40	1,70 *	1,70	2,90	1,20	1,50	1,26	20
3	0,66	0,43	0,03	0,16	0,69	0,07	0,73	1,32	0,52	0,33	0,44	20
4	1,90	0,30	1,30	0,30	0,20	0,40	0,60	0,00	0,30	0,70	0,58	20
5	0,00	0,10	0,10	0,00	0,10	0,00	0,20	0,60	0,00	0,20	0,15	20
6	0,29	0,00	0,29	0,68	0,29	0,49	0,29	0,00	0,10	0,39	0,25	20
7	1,12	0,82	1,07	1,22	1,38	0,68	0,95	1,34	1,18	1,11	0,78	20
8	0,06	0,06	0,41	0,09	0,03	0,28	0,15	0,66	0,31	0,27	0,21	20
9	1,10	0,30	0,10	0,60	0,80	0,20	0,30	0,50	1,00	0,70	0,46	20
10	0,20	0,90	0,30	0,10	0,40	0,50	0,50	0,80	0,30	0,50	0,36	20
Sr	0,78	0,49	0,57	0,39	0,51	0,45	0,52	0,84	0,49	0,51		200
NE	20	20	20	20	20	20	20	20	20	20		
L	2,71	1,09	1,97	1,34	1,78	0,89	1,81	2,92	1,69	1,78		

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

(\*) : Data have been modified in the right units.

Sample Lab Code	1	2	3	4	5	6	7	8	9	10	
(*)	1	43,85	24,85	29,55	48,50	34,35	53,15	20,25	57,85	16,20	39,10 *
	2	46,35	27,55	31,70	51,05	37,00	55,25	22,25	59,15	17,60	41,85
	3	47,03	27,78	32,30	52,13	36,79	56,21	22,58	61,35	18,35	42,65
	4	44,65	25,65	30,55	53,75	37,70	56,10	22,20	60,60	16,85	41,65
	5	47,60	27,55	32,35	52,30	37,55	57,70	22,70	61,80	17,90	42,10
	6	45,87	26,80	31,60	50,53	36,65	54,81	22,09	60,19	17,43	40,87
	7	46,30	27,60	32,14	50,66	36,91	55,41	22,58	60,45	18,07	41,53
	8	46,96	27,03	31,81	51,46	36,94	56,05	22,05	60,92	17,34	41,77
	9	45,05	25,55	30,25	49,20	35,50	53,50	21,15	58,35	40,20 *	16,75 *
	10	44,90	26,25	31,15	48,65	35,50	53,95	21,75	59,50	18,05	40,85
M	45,86	26,66	31,34	50,82	36,49	55,21	21,96	60,02	17,53	41,66	
REF.	45,38	26,35	31,08	50,49	36,20	54,73	21,84	59,76	17,49	41,51	
SD	1,21	1,03	0,94	1,69	1,05	1,40	0,75	1,28	0,68	0,60	

M = mean per sample

REF. = reference values

SD = standard deviation per sample

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

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

**Table IV :** Outlier identification

Sample	1	2	3	4	5	6	7	8	9	10
<b>Outliers Cochran</b>		2				2				
<b>Outlier Grubbs</b>									9	1; 9
<b>sr</b>	0,78	0,32	0,57	0,39	0,51	0,26	0,52	0,84	0,46	0,54
<b>SR</b>	1,33	1,07	1,03	1,71	1,11	1,50	0,84	1,41	0,75	0,71

**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 <sub>lab</sub>	t
1	- 1,53	- 1,50	- 1,53	- 1,99	- 1,85	- 1,58	- 1,59	- 1,91	- 1,29	- 2,41	- 1,72	0,32	16,82
2	+ 0,97	+ 1,20	+ 0,62	+ 0,56	+ 0,80	+ 0,52	+ 0,41	- 0,61	+ 0,11	+ 0,34	+ 0,49	0,50	3,13
3	+ 1,65	+ 1,42	+ 1,22	+ 1,64	+ 0,59	+ 1,47	+ 0,73	+ 1,59	+ 0,86	+ 1,14	+ 1,23	0,39	9,92
4	- 0,73	- 0,70	- 0,53	+ 3,26	+ 1,50	+ 1,37	+ 0,36	+ 0,84	- 0,64	+ 0,14	+ 0,49	1,29	1,19
5	+ 2,22	+ 1,20	+ 1,27	+ 1,81	+ 1,35	+ 2,97	+ 0,86	+ 2,04	+ 0,41	+ 0,59	+ 1,47	0,79	5,87
6	+ 0,49	+ 0,44	+ 0,53	+ 0,04	+ 0,45	+ 0,07	+ 0,24	+ 0,44	- 0,06	- 0,63	+ 0,20	0,36	1,76
7	+ 0,92	+ 1,25	+ 1,06	+ 0,17	+ 0,71	+ 0,68	+ 0,73	+ 0,69	+ 0,58	+ 0,02	+ 0,68	0,37	5,81
8	+ 1,58	+ 0,68	+ 0,73	+ 0,97	+ 0,74	+ 1,32	+ 0,20	+ 1,16	- 0,16	+ 0,26	+ 0,75	0,54	4,40
9	- 0,33	- 0,80	- 0,83	- 1,29	- 0,70	- 1,23	- 0,69	- 1,41	+ 22,71	- 24,76	- 0,93	11,19	0,26
10	- 0,48	- 0,10	+ 0,07	- 1,84	- 0,70	- 0,78	- 0,09	- 0,26	+ 0,56	- 0,66	- 0,43	0,64	2,11
d	+ 0,48	+ 0,31	+ 0,26	+ 0,33	+ 0,29	+ 0,48	+ 0,11	+ 0,26	+ 0,04	+ 0,15	+ 0,22	3,55	
Sd	1,21	1,03	0,94	1,69	1,05	1,40	0,75	1,28	0,68	0,60	1,11		

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

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

Sr = 0,54 mg / 100 g

SR = 1,81 mg / 100 g

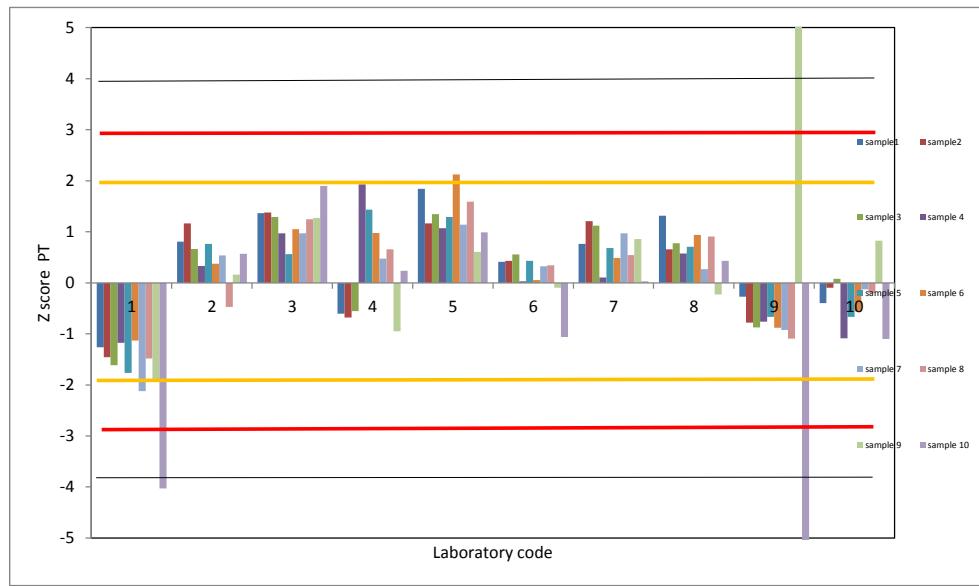
**Table VI :** Zscore of the different laboratories for each sample.  
ZS calculated on the PT standard deviation

Sample lab Code	1	2	3	4	5	6	7	8	9	10
1	-1,27	-1,46	-1,62	-1,18	-1,77	-1,13	<b>-2,12</b>	-1,49	-1,91	<b>-4,03</b>
2	+0,80	<b>+1,16</b>	+0,66	+0,33	+0,76	+0,37	+0,54	-0,47	+0,16	+0,57
3	+1,37	<b>+1,38</b>	+1,29	+0,97	+0,56	+1,05	+0,97	+1,24	+1,27	+1,90
4	-0,60	-0,68	-0,56	+1,93	+1,43	+0,98	+0,47	+0,66	-0,95	+0,24
5	+1,84	<b>+1,16</b>	+1,35	+1,07	+1,29	<b>+2,12</b>	+1,14	+1,59	+0,60	+0,99
6	+0,41	+0,43	+0,56	+0,03	+0,43	+0,05	+0,32	+0,34	-0,09	-1,06
7	+0,76	<b>+1,21</b>	+1,12	+0,10	+0,68	+0,48	+0,97	+0,54	+0,86	+0,03
8	+1,31	+0,66	+0,78	+0,57	+0,71	+0,94	+0,27	+0,90	-0,23	+0,43
9	-0,27	-0,78	-0,87	-0,76	-0,67	-0,88	-0,92	-1,10	<b>+33,59</b>	<b>-41,40</b>
10	-0,40	-0,10	+0,08	-1,09	-0,67	-0,56	-0,13	-0,20	+0,83	-1,10

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	-0,84	-0,83	-0,84	-1,10	-1,02	-0,87	-0,88	-1,05	-0,71	-1,33
2	+0,54	+0,66	+0,34	+0,31	+0,44	+0,29	+0,22	-0,33	+0,06	+0,19
3	+0,91	+0,79	+0,67	+0,91	+0,32	+0,81	+0,40	+0,88	+0,47	+0,63
4	-0,40	-0,39	-0,29	+1,80	+0,83	+0,76	+0,20	+0,47	-0,35	+0,08
5	+1,23	+0,66	+0,70	+1,00	+0,75	+1,64	+0,47	+1,13	+0,23	+0,33
6	+0,27	+0,24	+0,29	+0,02	+0,25	+0,04	+0,13	+0,24	-0,04	-0,35
7	+0,51	+0,69	+0,59	+0,09	+0,39	+0,37	+0,40	+0,38	+0,32	+0,01
8	+0,88	+0,37	+0,40	+0,54	+0,41	+0,73	+0,11	+0,64	-0,09	+0,14
9	-0,18	-0,44	-0,46	-0,71	-0,39	-0,68	-0,38	-0,78	+12,55	-13,68
10	-0,26	-0,06	+0,04	-1,02	-0,39	-0,43	-0,05	-0,14	+0,31	-0,36

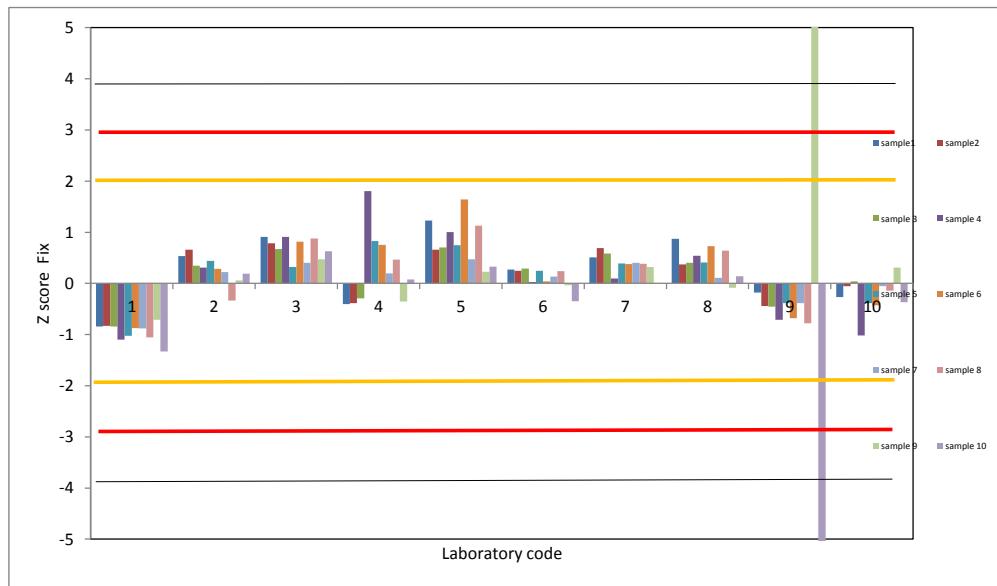
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=1,81

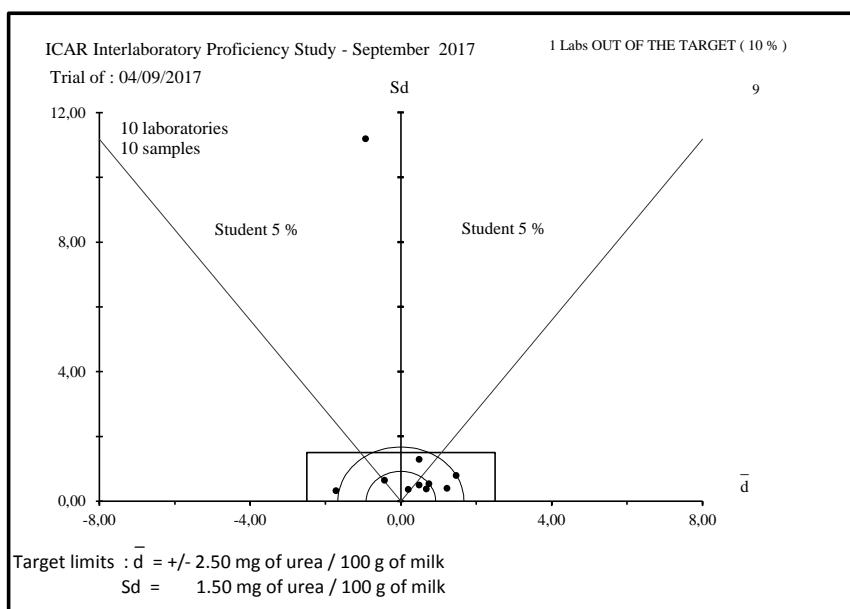
In yellow the values bigger or smaller than 2/-2

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  
September 2017

Name	City	Country
Actalia	Poligny	France
Agroscope	Bern-Liebefeld	Switzerland
Central Milk Lab ICBA	Caesarea	IL
Department Valorisation des productions Agricoles	Gembloux	Belgium
Laboratorio Standard Latte	Maccarese	Italy
Laboratorium Oceny Mleka KCHZ Parzniewie	Pruszkow	Poland
Milchprüfung Baden-Würtemberg e.V.	Kirchheim unter Teck	Germany
Pieno Tyrimai	Kaunas	Lithuania
Qlip B.V.,	Zutphen	NL
Valacta - Centre d'Expertise en Production Laitière du Québec	Quebec	Canada



**ICAR  
PROFICIENCY TESTING SCHEME**

**September 2017**

**Raw Milk**

**Enumeration of SOMATIC CELLS**

Sending date of statistical treatment : 9<sup>th</sup> October 2017

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

Proficiency test accredited ISO 17043



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**Table I : Ranking of the laboratories in %**

Nb	%	N°	d	Sd	D	Method
1	3	5	+ 1%	3%	4%	B
2	5	35	- 4%	2%	4%	B
3	8	8	- 7%	5%	9%	B
4	10	32	- 9%	5%	11%	B
5	13	34	+ 10%	6%	11%	B
6	15	15	+ 8%	9%	12%	A
7	18	39	- 13%	2%	13%	A
8	20	3	+ 11%	8%	13%	B
9	23	27	- 11%	8%	14%	B
10	25	36	- 14%	3%	14%	B
11	28	40	+ 15%	3%	15%	A
12	30	37	+ 15%	3%	16%	B
13	33	31	- 15%	5%	16%	B
14	35	30	- 16%	3%	16%	B
15	38	1	- 13%	10%	17%	A
16	40	38	- 17%	3%	17%	(1)
17	43	10	+ 14%	10%	17%	A
18	45	4	- 14%	11%	18%	B
19	48	29	- 17%	2%	18%	B
20	50	12	+ 13%	11%	18%	B
21	53	13	- 15%	12%	19%	B
22	55	25	- 15%	13%	20%	B
23	58	18	- 15%	13%	20%	B
24	60	14	- 16%	13%	21%	B
25	63	26	+ 17%	15%	23%	B
26	65	20	+ 20%	15%	25%	B
27	68	23	+ 19%	16%	25%	B
28	70	21	- 19%	16%	25%	B
29	73	16	- 20%	16%	26%	B
30	75	6	- 22%	14%	26%	A
31	78	9	+ 21%	19%	28%	B
32	80	22	+ 20%	20%	29%	B
33	83	2	- 23%	18%	29%	B
34	85	7	- 23%	18%	30%	B
35	88	33	- 29%	8%	30%	B
36	90	28	+ 36%	8%	36%	B
37	93	24	+ 33%	25%	41%	B
38	95	19	+ 38%	29%	48%	B
39	98	11	+ 47%	39%	61%	A
40	100	17	+ 59%	42%	73%	B

The table should be studied in parallel with figure 1 where the laboratories are located according to an acceptability area (or target) the limits of which are :  
 $\pm 10\%$  for d and  $10\%$  for Sd

REF : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 39 laboratories using reference method ISO 13366-1|IDF 148-1 and alternative method ISO 13366-2|IDF 148-2 after outlier discarding using Grubbs test at 5% risk level

A ISO 13366-1|IDF 148-1  
B ISO 13366-2|IDF 148-2  
(1) Image Cytometer - NucleoCounter SCC-400

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

(Nb : laboratory rank; % : relative rank)

(N° : laboratory identification number)

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

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

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

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

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

S<sub>r<sub>PT</sub></sub> 15 3%

S<sub>R<sub>PT</sub></sub> 146 27%

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

Sample lab code	1	2	3	4	5	6	7	8	9	10	Sr	NL
1	0	5	5	10	20	10	10	0	10	20	8	20
2	4	14	8	4	3	5	30	3	4	20	9	20
3	1	2	20	6	9	71	34	18	2	26	20	20
4	2	19	20	2	11	42	0	1	3	4	12	20
5	9	3	11	8	12	43	5	28	7	54	17	20
6	3	46	15	29	53	*	46	25	62	3	3	25
7	0	29	11	15	28	18	1	22	2	26	13	20
8	6	15	24	1	2	6	9	4	2	37	11	20
9	8	3	32	26	3	25	18	33	8	98	26	20
10	1	15	3	12	5	28	16	3	8	37	12	20
11	3	7	10	5	5	**	10	25	5	**	8	16
12	4	11	23	2	3	16	2	8	3	8	7	20
13	1	7	4	3	6	5	13	34	16	29	11	20
14	1	0	1	0	0	6	2	0	1	2	2	20
15	1	3	3	3	2	17	10	20	2	62	15	20
16	10	10	0	10	10	10	20	40	0	30	13	20
17	2	2	7	8	2	9	21	59	5	28	16	20
18	1	0	4	5	7	23	20	7	0	28	10	20
19	7	27	20	17	7	12	41	41	4	6	16	20
20	0	5	6	5	8	38	4	24	1	1	10	20
21	0	10	10	2	10	32	11	60	8	2	16	20
22	5	4	3	3	5	44	7	71	3	3	19	20
23	2	24	16	39	18	56	4	54	6	26	22	20
24	1	23	2	23	19	91	22	111	*	13	48	35
25	2	34	7	10	20	2	21	19	8	16	12	20
26	17	*	9	39	13	7	9	27	7	12	86	23
27	1	38	5	10	3	15	12	15	4	20	11	20
28	3	11	5	20	7	31	15	35	11	18	13	20
29	2	7	4	1	1	7	4	8	2	6	3	20
30	5	6	1	3	8	20	44	2	4	15	12	20
31	3	5	2	3	4	10	5	17	5	7	5	20
32	4	2	17	22	11	30	0	27	10	21	12	20
33	13	21	3	1	17	1	8	32	2	9	10	20
34	0	55	8	23	45	*	8	36	26	7	66	25
35	8	34	4	29	24	3	63	23	0	38	21	20
36	2	26	7	11	6	35	40	17	29	*	10	16
37	0	17	21	13	6	46	16	3	0	26	14	20
38	3	9	2	10	12	20	40	9	3	8	11	20
39	1	3	3	5	9	3	5	2	4	7	3	20
40	1	9	2	4	6	20	15	35	2	21	11	20
Sr	4	14	9	10	11	22	16	24	5	24		796
r	25	63	42	42	50	126	63	126	25	126		
NE	80	80	80	80	80	78	80	80	80	78		
L	13	60	41	43	34	94	69	91	19	103		

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

(\*) : Data were to modify in right units.

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

Sample lab code	1	2	3	4	5	6	7	8	9	10	
1	40	448	233	145	340	925	605	790	75	1070	
2	34	402	208	129	293	819	507	698	60	959	
3	48	578	304	191	433	1178	817	1017	110	1255	
4	34	455	214	143	328	911	600	800	71	1054	
5	42	551	264	173	390	1093	690	888	90	1260	
6	42	404	160	100	304	835	560	740	54	999	
7	30	417	193	133	305	855	524	678	65	927	
8	39	478	231	157	346	997	630	854	81	1149	
9	51	634	297	192	469	1292	793	1141	102	1513	
10	52	600	302	186	449	1209	791	1030	104	1385	
11	60	893	432	266	662	1141	1503	119			
12	44	601	292	197	426	1204	783	1026	94	1411	
13	34	438	224	152	335	904	578	758	78	1077	
14	41	454	221	146	314	905	580	746	70	1022	
15	40	569	264	175	400	1176	723	981	83	1359	
16	25	425	210	135	315	845	540	700	70	1005	
17	67	878	423	285	658	1633	1104	1452	139	1884	
18	36	434	216	155	335	904	572	781	78	1026	
19	83 *	752	376	239	494	1342	952	1290	134	1746	
20	47	665	313	200	466	1239	845	1107	104	1448	
21	36	426	218	135	309	843	565	732	79	975	
22	55	581	299	194	462	1313	783	1139	98	1526	
23	55	619	312	208	460	1248	819	1077	107	1498	
24	55	735	350	230	510	1428	882	1232	113	1586	
25	38	444	226	151	325	895	595	762	72	1040	
(*)	26	50	605	308	194	450	1274	820	1033	95	1467
27	39	459	218	142	334	928	606	810	77	1145	
28	67	706	352	222	523	1438	900	1249	130	1685	
29	36	448	219	136	309	892	536	757	74	1023	
30	35	453	221	143	321	890	567	766	79	1025	
31	30	443	219	143	332	894	578	779	73	1053	
32	43	470	240	144	340	977	623	825	70	1127	
33	41	362	177	115	278	754	480	679	63	863	
34	49	589	257	172	415	1192	705	1052	95	1369	
35	42	499	250	170	368	1039	655	867	81	1183	
36	34	447	221	139	321	901	617	796	71	1055	
37	48	610	309	202	441	1236	766	1019	104	1441	
38	39	437	220	148	320	874	572	734	77	1034	
39	40	447	234	148	335	924	608	791	76	1072	
40	51	576	306	185	443	1260	785	1048	101	1397	
M	43	536	263	170	391	1063	695	928	88	1234	
REF.	43	526	260	168	385	1060	686	918	87	1227	
SD	10	128	63	40	92	214	158	214	21	254	

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

**Table IV :** Outlier identification

Sample	1	2	3	4	5	6	7	8	9	10
Outliers Cochran	26				6; 34			24	36	
Outlier Grubbs	19									
sr	3	14	9	10	8	22	16	21	4	24
SR	10	128	63	41	94	215	158	212	21	254
sr %	7%	3%	4%	6%	2%	2%	2%	2%	5%	2%
SR %	23%	24%	24%	24%	24%	20%	23%	23%	24%	21%

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

Sample Lab Code	1	2	3	4	5	6	7	8	9	10	d	Sd <sub>lab</sub>	t
1	- 6%	- 15%	- 10%	- 14%	- 12%	- 13%	- 12%	- 14%	- 14%	- 13%	- 13%	10%	3,92
2	- 20%	- 24%	- 20%	- 23%	- 24%	- 23%	- 26%	- 24%	- 31%	- 22%	- 23%	18%	4,12
3	+ 11%	+ 10%	+ 17%	+ 14%	+ 12%	+ 11%	+ 19%	+ 11%	+ 27%	+ 2%	+ 11%	8%	4,15
4	- 20%	- 14%	- 18%	- 15%	- 15%	- 14%	- 13%	- 13%	- 19%	- 14%	- 14%	11%	4,22
5	- 3%	+ 5%	+ 1%	+ 3%	+ 1%	+ 3%	+ 1%	- 3%	+ 3%	+ 3%	+ 1%	3%	1,34
6	- 3%	- 23%	- 39%	- 41%	- 21%	- 21%	- 18%	- 19%	- 38%	- 19%	- 22%	14%	4,84
7	- 30%	- 21%	- 26%	- 21%	- 19%	- 24%	- 26%	- 25%	- 24%	- 23%	- 23%	18%	3,94
8	- 9%	- 9%	- 11%	- 7%	- 10%	- 6%	- 8%	- 7%	- 7%	- 6%	- 7%	5%	4,76
9	+ 20%	+ 20%	+ 14%	+ 14%	+ 22%	+ 22%	+ 16%	+ 24%	+ 18%	+ 23%	+ 21%	19%	3,53
10	+ 21%	+ 14%	+ 16%	+ 11%	+ 16%	+ 14%	+ 15%	+ 12%	+ 20%	+ 13%	+ 14%	10%	4,32
11	+ 39%	+ 70%	+ 66%	+ 58%	+ 72%	+ 66%	+ 64%	+ 37%	+ 47%	+ 39%	+ 39%	3,42	
12	+ 3%	+ 14%	+ 12%	+ 17%	+ 10%	+ 14%	+ 12%	+ 8%	+ 15%	+ 13%	+ 11%	3,72	
13	- 21%	- 17%	- 14%	- 10%	- 13%	- 15%	- 16%	- 17%	- 10%	- 12%	- 15%	12%	3,98
14	- 5%	- 14%	- 15%	- 13%	- 18%	- 15%	- 15%	- 19%	- 20%	- 17%	- 16%	13%	3,85
15	- 7%	+ 8%	+ 1%	+ 4%	+ 4%	+ 11%	+ 5%	+ 7%	- 4%	+ 11%	+ 8%	9%	2,63
16	- 41%	- 19%	- 19%	- 20%	- 18%	- 20%	- 21%	- 24%	- 19%	- 18%	- 20%	16%	4,06
17	+ 57%	+ 67%	+ 63%	+ 70%	+ 71%	+ 54%	+ 61%	+ 58%	+ 60%	+ 54%	+ 59%	42%	4,42
18	- 17%	- 18%	- 17%	- 8%	- 13%	- 15%	- 17%	- 15%	- 10%	- 16%	- 15%	13%	3,82
19	+ 93%	+ 43%	+ 45%	+ 42%	+ 28%	+ 27%	+ 39%	+ 40%	+ 54%	+ 42%	+ 38%	29%	4,10
20	+ 10%	+ 26%	+ 21%	+ 19%	+ 21%	+ 17%	+ 23%	+ 21%	+ 19%	+ 18%	+ 20%	15%	4,28
21	- 16%	- 19%	- 16%	- 20%	- 20%	- 18%	- 20%	- 18%	- 20%	- 21%	- 19%	16%	3,73
22	+ 28%	+ 10%	+ 15%	+ 15%	+ 20%	+ 24%	+ 14%	+ 24%	+ 12%	+ 24%	+ 20%	20%	3,19
23	+ 29%	+ 18%	+ 20%	+ 24%	+ 19%	+ 18%	+ 19%	+ 17%	+ 23%	+ 22%	+ 19%	16%	3,97
24	+ 28%	+ 40%	+ 35%	+ 37%	+ 32%	+ 35%	+ 29%	+ 34%	+ 30%	+ 29%	+ 33%	25%	4,13
25	- 11%	- 16%	- 13%	- 10%	- 16%	- 16%	- 13%	- 17%	- 17%	- 15%	- 15%	13%	3,81
26	+ 16%	+ 15%	+ 18%	+ 15%	+ 17%	+ 20%	+ 19%	+ 12%	+ 9%	+ 20%	+ 17%	15%	3,59
27	- 10%	- 13%	- 16%	- 15%	- 13%	- 12%	- 12%	- 12%	- 11%	- 7%	- 11%	8%	4,57
28	+ 56%	+ 34%	+ 35%	+ 32%	+ 36%	+ 36%	+ 31%	+ 36%	+ 49%	+ 37%	+ 36%	8%	14,22
29	- 16%	- 15%	- 16%	- 19%	- 20%	- 16%	- 22%	- 18%	- 15%	- 17%	- 17%	2%	22,94
30	- 19%	- 14%	- 15%	- 15%	- 17%	- 16%	- 17%	- 17%	- 9%	- 16%	- 16%	3%	18,77
31	- 31%	- 16%	- 16%	- 15%	- 14%	- 16%	- 16%	- 15%	- 16%	- 14%	- 15%	5%	9,70
32	+ 1%	- 11%	- 8%	- 14%	- 12%	- 8%	- 8%	- 9%	- 10%	- 19%	- 8%	5%	5,75
33	- 5%	- 31%	- 32%	- 32%	- 28%	- 29%	- 30%	- 26%	- 27%	- 30%	- 29%	8%	11,50
34	+ 15%	+ 12%	- 1%	+ 2%	+ 8%	+ 12%	+ 3%	+ 15%	+ 9%	+ 12%	+ 10%	6%	5,66
35	- 2%	- 5%	- 4%	+ 1%	- 4%	- 2%	- 5%	- 6%	- 7%	- 4%	- 4%	2%	5,50
36	- 20%	- 15%	- 15%	- 18%	- 17%	- 15%	- 10%	- 13%	- 19%	- 14%	- 14%	3%	15,38
37	+ 13%	+ 16%	+ 19%	+ 20%	+ 15%	+ 17%	+ 12%	+ 11%	+ 20%	+ 17%	+ 15%	3%	14,47
38	- 10%	- 17%	- 15%	- 12%	- 17%	- 18%	- 17%	- 20%	- 12%	- 16%	- 17%	3%	16,91
39	- 7%	- 15%	- 10%	- 12%	- 13%	- 13%	- 11%	- 14%	- 12%	- 13%	- 13%	2%	19,03
40	+ 18%	+ 9%	+ 18%	+ 10%	+ 15%	+ 19%	+ 14%	+ 14%	+ 16%	+ 14%	+ 15%	3%	14,39
d	+ 1%	+ 2%	+ 1%	+ 1%	+ 2%	+ 0%	+ 1%	+ 1%	+ 1%	+ 1%	+ 1%	- 1%	23%
Sd	23%	24%	24%	24%	24%	20%	23%	23%	24%	21%			

d = mean of differences

Sd = standard deviation of differences

t = Student test - comparison to 0

Upper limits :  $\bar{d} = +/- 10\%$    Sd = 10%**ISO 13366-2 | IDF 148-2 : Precision of the method :**

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

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

ZS calculated on the PT standard deviation

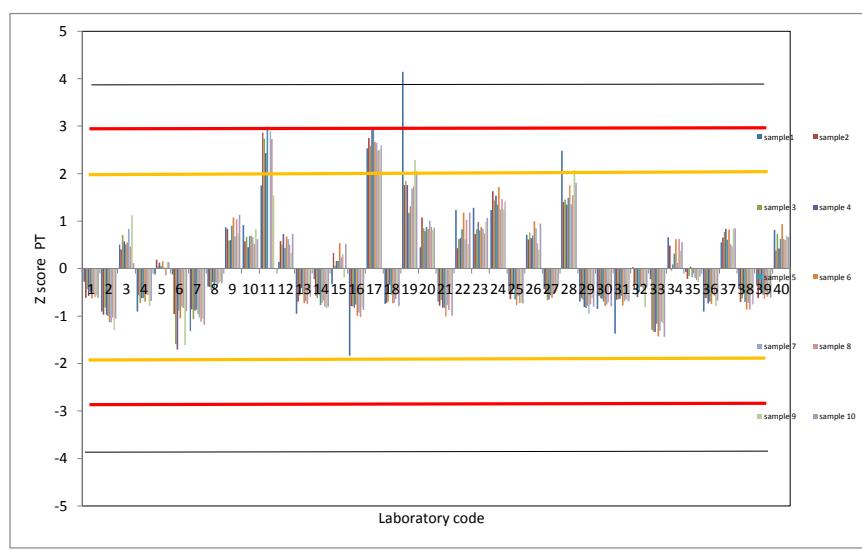
Sample lab code	1	2	3	4	5	6	7	8	9	10
1	-0,28	-0,62	-0,43	-0,57	-0,49	-0,63	-0,51	-0,60	-0,57	-0,62
2	-0,90	-0,97	-0,82	-0,97	-1,00	-1,13	-1,14	-1,03	-1,30	-1,06
3	+0,50	+0,40	+0,70	+0,57	+0,51	+0,55	+0,83	+0,46	+1,13	+0,11
4	-0,90	-0,56	-0,73	-0,62	-0,62	-0,69	-0,55	-0,55	-0,79	-0,68
5	-0,12	+0,19	+0,06	+0,12	+0,05	+0,15	+0,02	-0,14	+0,13	+0,13
6	-0,12	-0,96	-1,59	-1,71	-0,88	-1,05	-0,80	-0,83	-1,61	-0,90
7	-1,32	-0,86	-1,07	-0,89	-0,87	-0,96	-1,03	-1,12	-1,06	-1,18
8	-0,38	-0,38	-0,46	-0,29	-0,42	-0,29	-0,36	-0,30	-0,28	-0,31
9	+0,87	+0,84	+0,59	+0,60	+0,90	+1,08	+0,68	+1,04	+0,74	+1,13
10	+0,92	+0,57	+0,66	+0,45	+0,69	+0,70	+0,67	+0,52	+0,84	+0,62
11	+1,75	+2,86	+2,74	+2,43	+2,99		+2,89	+2,73	+1,54	
12	+0,14	+0,58	+0,51	+0,72	+0,44	+0,67	+0,62	+0,50	+0,33	+0,73
13	-0,95	-0,69	-0,57	-0,41	-0,54	-0,73	-0,69	-0,75	-0,43	-0,59
14	-0,23	-0,57	-0,62	-0,55	-0,77	-0,72	-0,67	-0,80	-0,84	-0,81
15	-0,33	+0,33	+0,06	+0,16	+0,16	+0,54	+0,24	+0,29	-0,18	+0,52
16	-1,84	-0,79	-0,79	-0,82	-0,76	-1,00	-0,93	-1,02	-0,81	-0,87
17	+2,53	+2,75	+2,59	+2,92	+2,95	+2,67	+2,65	+2,49	+2,51	+2,59
18	-0,75	-0,72	-0,69	-0,34	-0,55	-0,73	-0,72	-0,64	-0,43	-0,79
19	+4,14	+1,76	+1,85	+1,76	+1,17	+1,32	+1,69	+1,73	+2,29	+2,05
20	+0,45	+1,08	+0,85	+0,79	+0,87	+0,84	+1,01	+0,88	+0,81	+0,87
21	-0,69	-0,78	-0,66	-0,82	-0,82	-1,01	-0,77	-0,87	-0,38	-0,99
22	+1,23	+0,43	+0,62	+0,64	+0,83	+1,18	+0,61	+1,03	+0,52	+1,18
23	+1,28	+0,72	+0,83	+0,99	+0,81	+0,88	+0,85	+0,74	+0,98	+1,07
24	+1,23	+1,63	+1,43	+1,54	+1,35	+1,72	+1,24	+1,46	+1,25	+1,42
25	-0,49	-0,64	-0,54	-0,42	-0,65	-0,77	-0,58	-0,73	-0,72	-0,74
26	+0,71	+0,61	+0,76	+0,64	+0,70	+1,00	+0,85	+0,53	+0,40	+0,95
27	-0,43	-0,53	-0,67	-0,65	-0,56	-0,62	-0,51	-0,51	-0,47	-0,32
28	+2,48	+1,40	+1,46	+1,35	+1,49	+1,76	+1,36	+1,54	+2,07	+1,81
29	-0,69	-0,62	-0,65	-0,81	-0,83	-0,79	-0,95	-0,75	-0,62	-0,80
30	-0,85	-0,57	-0,62	-0,64	-0,69	-0,79	-0,75	-0,71	-0,38	-0,80
31	-1,37	-0,66	-0,65	-0,64	-0,57	-0,77	-0,69	-0,65	-0,69	-0,69
32	+0,03	-0,44	-0,32	-0,60	-0,49	-0,39	-0,40	-0,44	-0,81	-0,39
33	-0,23	-1,29	-1,32	-1,34	-1,16	-1,43	-1,31	-1,12	-1,15	-1,44
34	+0,66	+0,49	-0,04	+0,09	+0,32	+0,62	+0,12	+0,62	+0,38	+0,56
35	-0,07	-0,21	-0,15	+0,04	-0,19	-0,10	-0,20	-0,24	-0,28	-0,17
36	-0,90	-0,62	-0,62	-0,74	-0,69	-0,74	-0,44	-0,57	-0,79	-0,68
37	+0,55	+0,65	+0,78	+0,84	+0,60	+0,82	+0,51	+0,47	+0,84	+0,85
38	-0,43	-0,70	-0,63	-0,50	-0,70	-0,87	-0,72	-0,86	-0,50	-0,76
39	-0,33	-0,62	-0,42	-0,51	-0,55	-0,64	-0,50	-0,59	-0,52	-0,61
40	+0,82	+0,38	+0,74	+0,42	+0,63	+0,93	+0,63	+0,60	+0,69	+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	-0,69	<b>+2,14</b>	-1,16	-1,52	-1,46	<b>-2,12</b>	-1,68	<b>+2,33</b>	-1,51	<b>-2,13</b>
2	<b>-2,26</b>	<b>-3,38</b>	<b>-2,21</b>	<b>-2,58</b>	<b>-3,01</b>	<b>-3,80</b>	<b>-3,73</b>	<b>-4,01</b>	<b>-3,43</b>	<b>-3,64</b>
3	<b>+1,26</b>	+1,40	+1,90	+1,52	+1,54	+1,85	<b>+2,73</b>	+1,79	<b>+2,98</b>	+0,39
4	<b>-2,26</b>	-1,95	-1,95	-1,65	-1,87	-2,34	-1,79	-2,15	-2,08	-2,35
5	-0,30	+0,65	+0,16	+0,33	+0,16	+0,51	+0,08	-0,55	+0,35	+0,45
6	-0,30	<b>-3,32</b>	<b>-4,29</b>	<b>-4,53</b>	<b>-2,65</b>	<b>-3,54</b>	<b>-2,63</b>	<b>-3,23</b>	<b>-4,26</b>	<b>-3,10</b>
7	<b>-3,30</b>	<b>-2,98</b>	<b>-2,87</b>	<b>-2,35</b>	<b>-2,60</b>	<b>-3,22</b>	<b>-3,38</b>	<b>-4,36</b>	<b>-2,79</b>	<b>-4,07</b>
8	-0,95	-1,33	-1,23	-0,76	-1,27	-0,99	-1,17	-1,16	-0,74	-1,06
9	<b>+2,17</b>	<b>+2,91</b>	+1,60	+1,59	+2,71	<b>+3,64</b>	+2,23	<b>+4,04</b>	+1,95	<b>+3,89</b>
10	<b>+2,30</b>	+1,98	+1,79	+1,19	+2,06	+2,35	+2,19	+2,02	+2,21	<b>+2,15</b>
11	<b>+4,38</b>	<b>+9,94</b>	<b>+7,37</b>	<b>+6,45</b>	<b>+8,97</b>		<b>+9,48</b>	<b>+10,61</b>	<b>+4,06</b>	
12	+0,35	+2,01	+1,36	+1,92	+1,31	+2,27	<b>+2,02</b>	+1,96	+0,86	+2,51
13	<b>-2,39</b>	<b>-2,41</b>	-1,53	-1,09	-1,63	-2,46	-2,26	<b>-2,91</b>	-1,12	-2,04
14	-0,56	-1,96	-1,68	-1,45	<b>-2,31</b>	-2,43	-2,21	<b>-3,12</b>	-2,21	-2,78
15	-0,82	+1,14	+0,16	+0,43	+0,48	+1,82	+0,77	+1,14	-0,48	+1,80
16	<b>-4,60</b>	<b>-2,75</b>	<b>-2,13</b>	<b>-2,18</b>	<b>-2,28</b>	<b>-3,38</b>	<b>-3,04</b>	<b>-3,96</b>	<b>-2,15</b>	<b>-3,01</b>
17	<b>+6,34</b>	<b>+9,54</b>	<b>+6,97</b>	<b>+7,74</b>	<b>+8,86</b>	+9,01	+8,70	+9,68	+6,63	+8,93
18	-1,87	<b>-2,51</b>	-1,87	-0,89	-1,64	-2,46	-2,37	<b>-2,50</b>	-1,12	-2,73
19	<b>+10,37</b>	<b>+6,11</b>	<b>+4,98</b>	<b>+4,66</b>	<b>+3,52</b>	<b>+4,44</b>	<b>+5,53</b>	<b>+6,74</b>	<b>+6,05</b>	<b>+7,06</b>
20	+1,13	<b>+3,75</b>	<b>+2,28</b>	<b>+2,08</b>	<b>+2,63</b>	<b>+2,82</b>	<b>+3,31</b>	<b>+3,43</b>	<b>+2,14</b>	<b>+3,00</b>
21	-1,74	<b>-2,72</b>	-1,78	<b>-2,18</b>	-2,47	<b>-3,41</b>	-2,53	-3,38	-0,99	-3,42
22	<b>+3,08</b>	+1,48	+1,66	+1,69	+2,48	+3,98	+2,01	<b>+4,00</b>	+1,37	+4,06
23	<b>+3,21</b>	<b>+2,51</b>	<b>+2,24</b>	<b>+2,61</b>	<b>+2,43</b>	+2,96	+2,77	+2,88	+2,59	+3,69
24	<b>+3,08</b>	<b>+5,65</b>	<b>+3,86</b>	<b>+4,07</b>	<b>+4,04</b>	<b>+5,78</b>	<b>+4,09</b>	<b>+5,69</b>	<b>+3,30</b>	<b>+4,88</b>
25	-1,22	<b>-2,24</b>	-1,46	-1,12	-1,95	-2,59	-1,90	<b>-2,84</b>	-1,89	<b>-2,54</b>
26	<b>+1,78</b>	<b>+2,12</b>	<b>+2,05</b>	<b>+1,69</b>	<b>+2,09</b>	<b>+3,36</b>	<b>+2,78</b>	<b>+2,08</b>	+1,05	<b>+3,27</b>
27	-1,09	-1,83	-1,80	-1,72	-1,68	-2,08	-1,66	-1,97	-1,25	-1,11
28	<b>+6,21</b>	<b>+4,86</b>	<b>+3,93</b>	<b>+3,57</b>	<b>+4,46</b>	<b>+5,94</b>	<b>+4,45</b>	<b>+6,00</b>	<b>+5,47</b>	<b>+6,23</b>
29	-1,74	<b>-2,14</b>	-1,74	<b>-2,15</b>	<b>-2,49</b>	<b>-2,65</b>	<b>-3,12</b>	<b>-2,93</b>	-1,63	<b>-2,77</b>
30	<b>-2,13</b>	-1,99	-1,68	-1,69	<b>-2,08</b>	-2,67	-2,48	<b>-2,76</b>	-0,99	<b>-2,75</b>
31	<b>-3,43</b>	<b>-2,28</b>	-1,74	-1,69	<b>-1,72</b>	-2,61	-2,26	<b>-2,53</b>	-1,83	<b>-2,37</b>
32	+0,09	-1,53	-0,86	-1,59	-1,48	-1,30	-1,31	<b>-1,70</b>	-2,15	-1,36
33	-0,56	<b>-4,47</b>	<b>-3,56</b>	<b>-3,54</b>	<b>-3,49</b>	<b>-4,82</b>	<b>-4,29</b>	<b>-4,34</b>	<b>-3,04</b>	<b>-4,95</b>
34	+1,65	+1,69	-0,11	+0,23	+0,95	+2,08	+0,40	<b>+2,43</b>	+0,99	+1,93
35	-0,17	-0,74	-0,41	+0,10	-0,56	-0,34	-0,65	-0,94	-0,74	-0,59
36	<b>-2,26</b>	<b>-2,15</b>	-1,68	-1,95	<b>-2,08</b>	<b>-2,51</b>	-1,43	<b>-2,23</b>	<b>-2,08</b>	<b>-2,33</b>
37	+1,39	+2,26	+2,09	+2,22	+1,81	+2,77	+1,67	+1,82	+2,21	+2,91
38	-1,09	<b>-2,44</b>	-1,70	-1,32	<b>-2,11</b>	<b>-2,92</b>	<b>-2,37</b>	<b>-3,35</b>	-1,31	-2,62
39	-0,82	<b>-2,17</b>	-1,12	-1,36	-1,64	-2,14	-1,63	<b>-2,31</b>	-1,38	-2,11
40	<b>+2,04</b>	+1,33	+1,98	+1,12	+1,88	<b>+3,15</b>	+2,05	+2,35	+1,82	+2,31

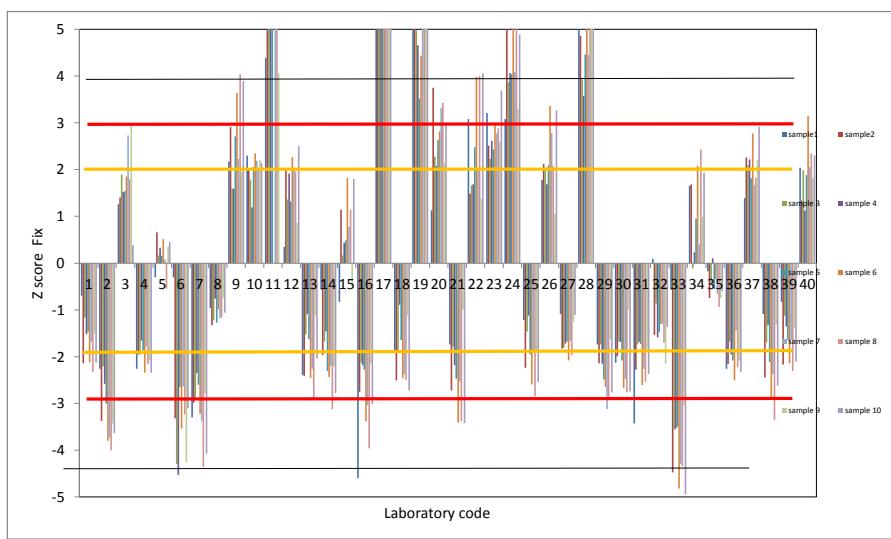
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR : Cf page 5/8

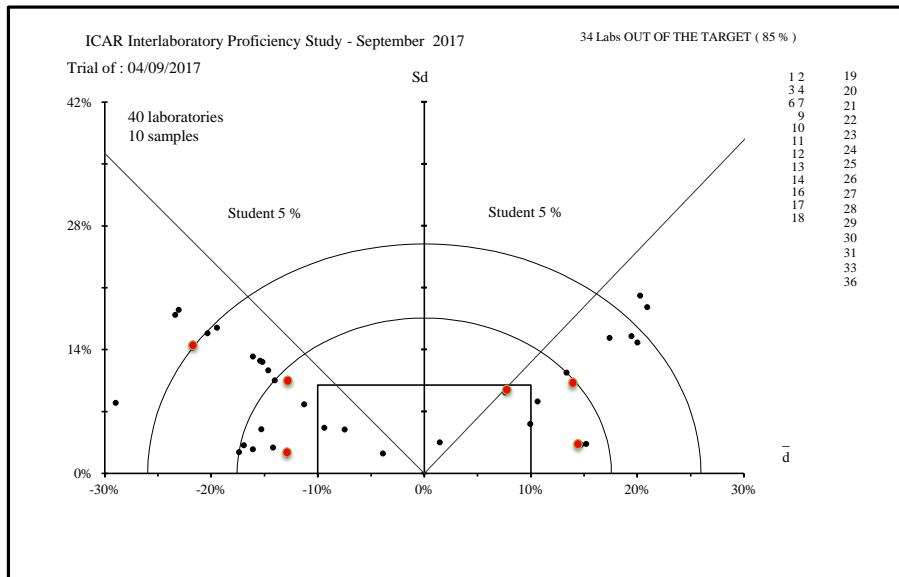
In yellow the values bigger or smaller than 2/-2

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

**Figure 3 :**

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





**Figure 1 : ACCURACY - Evaluation of the individual performances (to see table I).**  
 Reference Method ISO 13366-1 IDF 148-1

LIST OF THE PARTICIPANTS ICAR			
ICAR PROFICIENCY TEST			
RAW MILK			
Somatic Cell Counting			
Name	City	Country	
Actalia	Poligny	France	
Agroscope	3003 Bern-Liebefeld	Switzerland	
Alip	Sousada	Portugal	
Cattle Information Service (CIS)	Teiford Shropshire TF3BD	England	
Central Milk Lab ICBA	Caesarea	IL	
ChemoMetec A/S	Allerød	Denmark	
Comité du Lait ASBL	Battice	Belgium	
Dairy Cattle Res. Center Shandong Academy of Agr. Sc.	Shandong	China	
Deltamune	Centurion - Pretoria	South Africa	
Direction de l' Amelioration Genetique	Sidi Thabet	Tunisie	
Eastern Lab services	Medina	USA	
Eurofins Steins Laboratory A/B	Jönköping	Sweden	
Eurofins Steins Laboratory A/S	Vejen	Denmark	
Holstein-Ro Breeders Association Laboratory	Bucharest	Romania	
Lab Agroalimentario de Santander	Santander Cantabria	Spain	
Laborator pro rozbor mléka Brno,	Brno	Czech Republic	
Laborator pro rozbor mléka Bustehrad,	Bustehrad	Czech Republic	
Laboratorija mleka, Poljoprivredni fakultet Novi Sad,	Novi Sad	Serbia	
Laboratorio Standard Latte	Maccarese	Italy	
Laboratorium Oceny Mleka KCHZ Parzniewie	Pruszkow	Poland	
Merieux NutriScience J Bay	Cape Town	South Africa	
Merieux Nutriscience South Africa	Cape Town	South Africa	
Merieux Nutriscience South Africa (Midrand)	Midrand	South Africa	
Milchprüfung Baden-Württemberg e.V.	Kirchheim unter Teck	Germany	
Osuuskuanta Satamaito, laboratorio	Pori	Finland	
Pieno Tyrimai	Kaunas	Lithuania	
Qlip B.V.,	Zutphen	NL	
Shanghai dairy breeding center Co.Ltd	Shanghai	China	
Suisselab AG	Zullikofen	Switzerland	
Taiwan Livestock Research Institute	Taiwan	Taiwan	
Univ. of Pretoria Dept Production Animal Studies Milk Lab	Pretoria	South Africa	
Valacta - Centre d'Expertise en Production Laitière du Québec	Quebec	Canada	
Valio Oy/Seinajoen aluelaboratorio	Seinajoki	Finland	