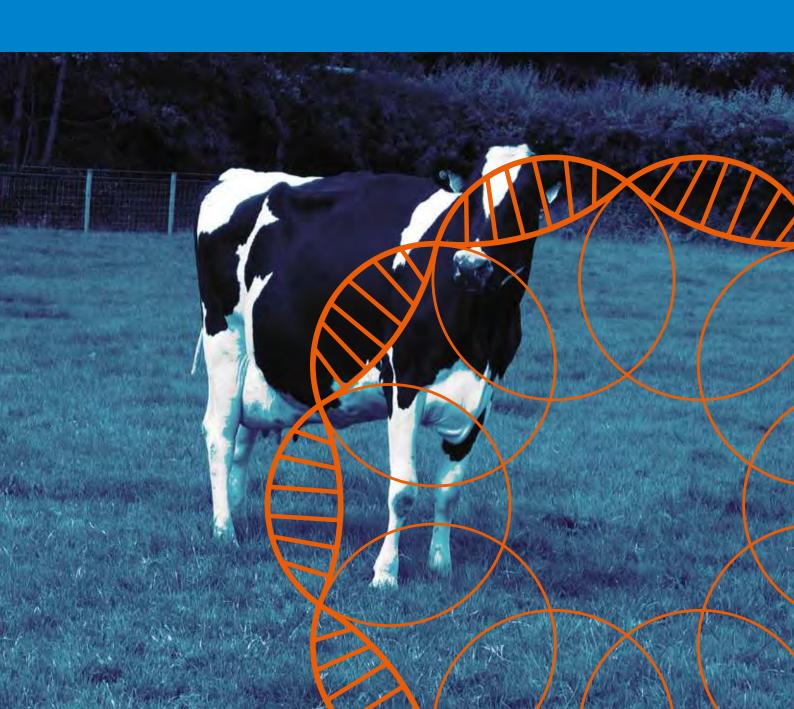


Network. Guidelines. Certification.

ICAR PROFICIENCY TEST - SEPTEMBER 2025

Raw cow milk
"Reference" Methods





FRAME OF ACTIVITY:

ICAR MILK ANALYSES SUB-COMMITTEE (MA SC)

ORGANISER: ICAR, ARTHUR VAN SCHENDELSTRAAT 550, 3511 MH UTRECHT, THE NETHERLANDS

Email: pt@icar.org



Raw cow milk "Reference" Methods



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Somatic Cell (microscopic and fluoro-optoelectronic method)

Fatty Acids



1. Introduction

Dear Participant,

Thank you for participating in the ICAR Proficiency Test (PT) September 2025!

This is the twentieth round that ICAR organized since 2016 !!!

The samples preparation and statistical elaboration have been done by ICAR with Sub- contractors Actalia and Lactanet for fatty acids. Both subcontractors are accredited ISO 17025 and ISO 17043.

The synthetic report and control charts over the time are prepared by ICAR.

The advantage to participate in the PT round is to obtain a worldwide updated picture of the analytical situation for milk analyses.

For somatic cell parameter, since March 2020 we have the possibility to build the international traceability to the EC JRC Certified reference material for somatic cell counting in milk. Following the ISO/IDF Bullettin 508/2021 Guidance and application of EC JRC Certified reference material for somatic cell counting in milk we have characterized the ICAR PT samples These values and the ISO 13366-2 standard deviation of reproducibility, will be used to calculate, for **SCC ZScore FIX.** In this way the ZS FIX will better anchored to the international metrological traceability and you can follow your instrument performance over the time.

NOTE: The ZS FIX for SCC was not calculated because we have introduce a new batch of Sample A and Sample B that will be offered again in the next ICAR PT rounds. From March 2026 we will start again to calculate the ZS FIX for SCC

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

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

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

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

In tables A,B,C,D,E,F,G if 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, for each parameter, 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. ICAR Milk Analyses Sub Committee is monitoring these limits and eventually will 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-Scorept (standard deviation calculated on this proficiency test) and the Z-Scorefix (standard deviation of the standard method) are summarized.

Z-Score_{FIX} is calculated considering the standard deviation of reproducibility of the standardized method

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.

Control Charts and tables

On the control charts are reported the last 3 proficiency tests where your lab participated

In the associated table are reported all the ZS-PT and ZS Fix where your laboratory participated

For this reason from this round the ZS values are reported according the sample order from 1 to 10 and not according the sample concentration as organized in the previous PT

<u>In the second part of the report</u> 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.

For the new parameter fatty acids, the statistical elaboration is done for each fatty acid. In case we did not receive enough results the data are reported in the complete report without any statistic.

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!



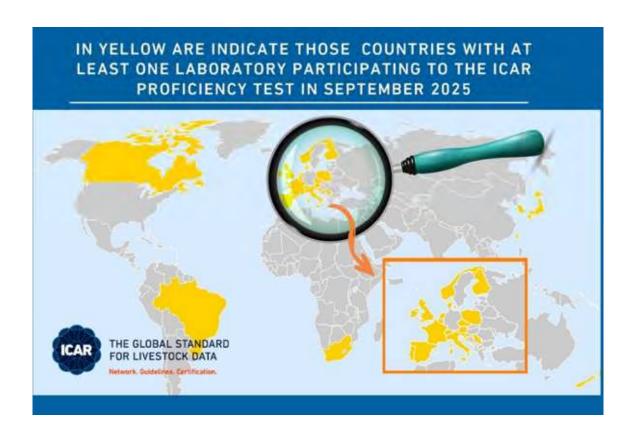
The list of laboratories that participated in ICAR PT September 2025 with at least one parameter is reported below.

Table 1. Participating milk laboratories to the ICAR Proficiency Test (September 2025)

Country	Laboratory	City
Austria	Pruefstelle Labor Rotholz	Rotholz 50a
Belgium	Comite du Lait ASBL	Battice
Belgium	Department of Agricultural products of Walloon Agricultural Research Centre	Gembloux
Brazil	Clínica do Leite Ltda	Piracibaca – SP Brazil
Canada	Horizon Lab Ltd	Winnipeg, MB
Canada	Lactanet	Sainte-Anne-de-Bellevue
Canada	Lactanet Guelph	Ontario
Croatia	Hrvatska Agencija za poljoprivredu i hranu	Krizevci
Czech Rep.	Laborator pro rozbor mleka Brno, Ceskomoravská spolecnost chovatelu a.s.	Brno
Czech Rep.	MILCOM a.s Dairy Research Institute	Praha 6
Denmark	Eurofins Milk Testing Denmark	Vejen
Denmark	Foss Analytical A/S	Hillerod
Finland	Osuuskunta Satamaito,	Ulvila
Finland	Valio Oy, Regional laboratory	Seinajoki
France	LSI - Thermo Fisher Scientific	Lissieu
France	Oxygen Laboratory	Maroeuil
Ireland	Dale Farm Dairy Co Operative	Ballymena Co Antrim
Ireland	Independent Milk Laboratories Ltd	Bailieborough
Israel	Central Milk Laboratory – ICBA	Caesarea
Italy	Associazione Italiana Allevatori, Laboratorio Standard Latte (LSL-AIA)	Maccarese
Italy	Associazione Regionale Allevatori della Lombardia	Crema
Italy	Federazione Latterie Alto Adige Soc. Agr. Coop.	Bolzano
Japan	Japan Dairy Technical Association	Chiyoda-Ku, Tokyo
Korea	Korea Animal Improvement Association	Anseong-si, gyeonggi-do
Netherlands	Qlip B.V.	Zutphen
New Zealand	MilkTestNZ	Hamilton
Norway	Tine Ramelklaboratoriet Heimdal	Heimdal
Poland	Krajowe Centrum Hodowli Zwierząt, Laboratorium Referencyjne Parzniew	Pruszków
Poland	MlekoLab Sp.z o.o.	Czerwin
Poland	Okręgowa Spółdzielnia Mleczarska w Piątnicy	Piątnica
Poland	PFHBiPM Laboratorium w Bialymstoku zs.w Jezewie Starym	Tykocin
Poland	PFHBiPM Laboratorium w Kobiernie	Kobierno
Poland	PFHBiPM Laboratorium w Parzniewie	Pruszkow
Poland	PFHBiPM Region Oceny Bydgoszcz z/s w Minikowie	Minikowo



Country	Laboratory	City
Poland	ZHW w Warszawie Oddział Terenowy w Siedlcach	Siedlce
Portugal	Laboratório de Microbiologia dos Alimentos	Vairão - Vila do Conde
Portugal	Sercla Terceira	Angra do Heroismo
Serbia	Laboratorija za ispitivanje kvaliteta mleka, Poljoprivredni fakultet Novi Sad	Novi Sad
Slovenia	KGZS Zavod Ptuj	Ptuj
Slovenia	University of Ljubljana, Biotechnical Faculty, Department of Animal Science, Institute of Dairy Science and Probiotics	Domzale
South Africa	Merieux NutriScience J Bay	Claremont, Cape Town
South Africa	Milk Lab., Univ. of Pretoria, Fac, vet. Sc Dept. Animal Studie, Pathology. Building room N.1-32	Pretoria
Spain	CICAP	Pozoblanco
Spain	Laboratorio Agroalimentario de Santander	Santander
Sweden	Eurofins Milk Testing Sweden AB	Jonkoping
Switzerland	Agroscope	Bern-Liebefeld
Switzerland	Suisselab AG	Zollikofen
Taiwan	Council of Agriculture, Executive Yuan, Taiwan Animal Germplasm Center of TLRI	Pitoumian, Xihu Township, Miaoli County 368,
UK	National Milk Records Limited_ Glasgow	Glasgow, Scotland
UK	National Milk Records Limited_ Wolverhampton	Wolverhampton, England
UK	The Cattle Information Service	Tellford, Shrpshire





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 RF0925

Laboratory Name

		Your participation Codes										
	Subscription	Fat ref	Protein ref	Lactose ref	Urea ref	SCC ref/alt						
A		Yes	Yes	Yes	Yes	Yes						
	Participation Codes	1	1	1	1	1						
	Are all the sample results received?	No	No	Yes	Yes	Yes						

	Data results received on time									
B		Fat ref	Protein ref	Lactose ref	Urea ref	SCC ref/alt				
	Results reception date	12/09/2025	12/09/2025	12/09/2025	12/09/2025	12/09/2025				

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 repor	t the value in total nitrogen							

	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	1	1 1		1	1					
D	%	31	15	20	71	70					
	d	0.007	-0.006	-0.012	-3.270	4%					
	Sd	0.009	0.004	0.016	0.586	4%					
	D	0.012	0.008	0.019	3.322	6%					
		Limits									
	d	<= 0.020	<= 0.025	<= 0.10	-2.5 <= d <= 2.5	-10% <= d <= 10%					
	Sd	<= 0.030	<= 0.020	<= 0.10	<= 1.5	<= 10%					

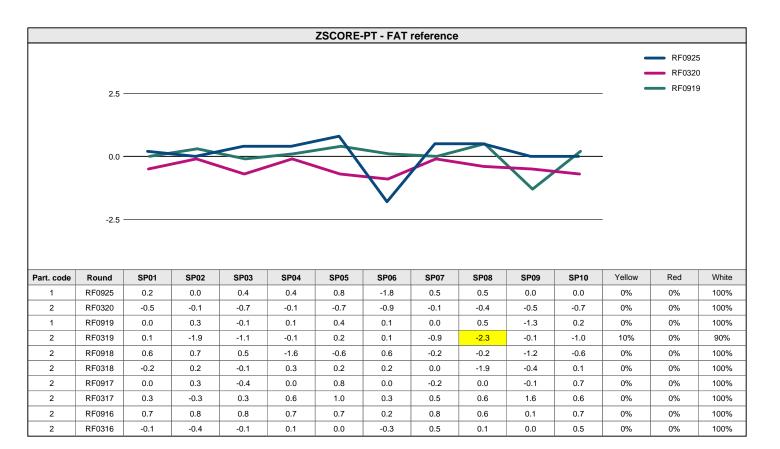
	Outliers										
		Fat ref	Protein ref	Lactose ref	Urea ref	SCC ref/alt					
		g/100g	nitrogen g/100g *	g/100g	mg/dl	SCC*1000/ml					
	Sample 1										
	Sample 2										
E	Sample 3										
_	Sample 4										
	Sample 5										
	Sample 6										
	Sample 7										
	Sample 8										
	Sample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

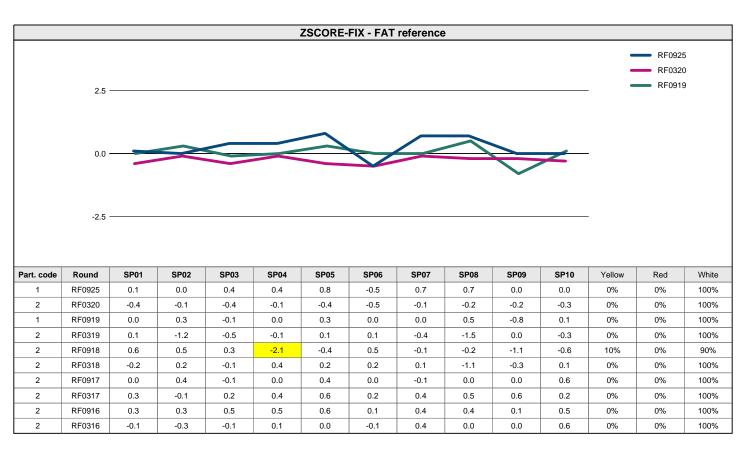
Repeatability										
Your "r" performance										
	Fat ref	Protein ref	Lactose ref	Urea ref	SCC ref/alt					
	g/100g	nitrogen g/100g *	g/100g	mg/dl	SCC*1000/ml					
Sample 1	0.003	0.001	0.01	0.16	5					
Sample 2	0.004	0.004	0.00	0.39	11					
Sample 3	0.004	0.005	0.01	0.23	16					
Sample 4 0.002		0.014	0.00	0.03	27					
Sample 5	0.003	0.001	0.02	0.75	3					
Sample 6	0.005	0.009	0.00	0.50	10					
Sample 7	0.009	0.010	0.00	0.11	7					
Sample 8	0.009	0.010	0.00	0.11	7					
Sample 9	0.000	0.003	0.00	0.23	1					
Sample 10	0.000	0.009	0.02	0.05	1					

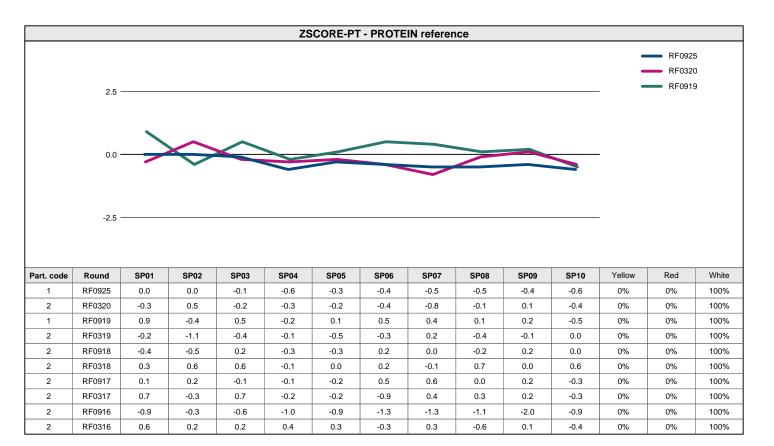
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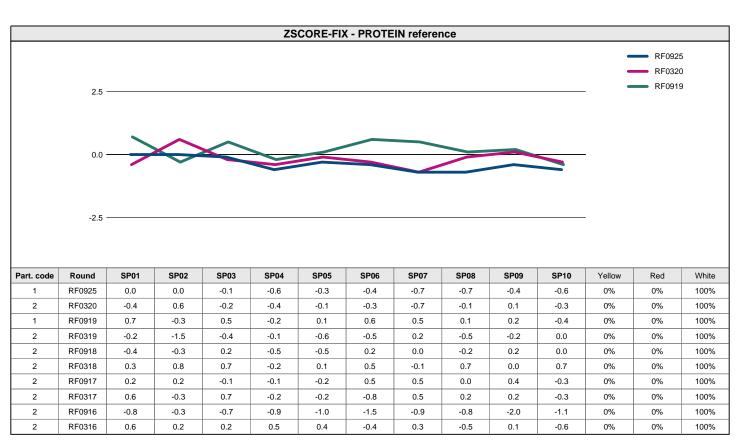
	Limits										
	Fat ref	Protein ref	Lactose ref	Urea ref	SCC r	ef/alt					
-	g/100g ISO 1211 IDF 1D	nitrogen g/100g * ISO 8968 IDF 20	g/100g ISO 22662 IDF 198	mg/dl ISO 14637 IDF 195	SCC*10 ISO 13 IDF 1	3366-2					
	<= 0.043	<= 0.038	<= 0.06	<= 1.52	Level	r					
					150	25					
					300	42					
					450	51					
					750	64					
-					1500	126					

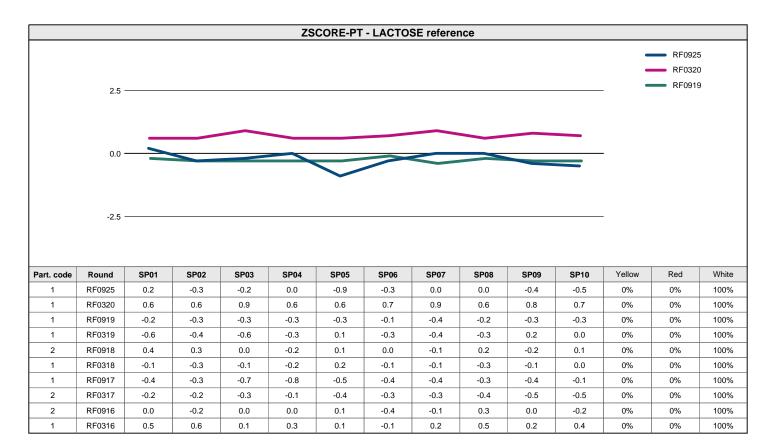
				Your Z-	Score PT					
		Fat ref	Pro	tein ref	Lactose re	ef Urea ref	SCC ref/alt			
	Sample 1	0.16		0.01	0.21	-1.39	0.57			
	Sample 2	-0.03	-0.05		-0.25	-1.48	1.13			
	Sample 3	0.39		-0.07	-0.23	-1.35	0.73			
	Sample 4	0.43		-0.58	0.02	-1.20	0.98			
	Sample 5	0.79		-0.28	-0.88	-1.18	1.18			
	Sample 6	-1.76		-0.37	-0.29	-1.09	0.49			
	Sample 7	0.52		-0.51	0.00	-1.10	0.35			
	Sample 8	0.52		-0.51	0.00	-1.10	0.35			
	Sample 9	0.00		-0.39	-0.41	-1.36	-0.85			
	Sample 10	0.00		-0.63	-0.49	-1.35	-1.54			
	Your Z-Score Fix									
3 🗆		Fat ref	Pro	tein ref	Lactose re	ef Urea ref	SCC ref/alt			
	Sample 1	0.11		0.00	0.23	-1.48	0.45			
	Sample 2	-0.01		-0.05	-0.28	-1.68	0.91			
	Sample 3	0.40		-0.09	-0.19	-1.82	0.59			
	Sample 4	0.44		-0.57	0.02	-2.17	0.78			
	Sample 5	0.79		-0.32	-0.93	-2.38	0.78			
	Sample 6	-0.52		-0.43	-0.23	-2.10	0.41			
	Sample 7	0.72		-0.69	0.00	-1.89	0.22			
	Sample 8	0.72		-0.69	0.00	-1.89	0.22			
	Sample 9	0.00		-0.35	-0.36	-1.44	-0.60			
	Sample 10	0.00		-0.55	-0.60	-1.46	-1.61			
		If there is a sample with a "z-sc	ore" in the ye	llow or red area p	lease check table VI	and VII in correspondence of your lab	code.			
				Interpreta	tion Z-Score					
	Z-Score < -3	-3 <= Z-Score <	: -2	-2 -2 <= Z-Score <= 2		2 < Z-Score <= 3	Z-Score > 3			
	Poor	Moderate		G	iood	Moderate	Poor			

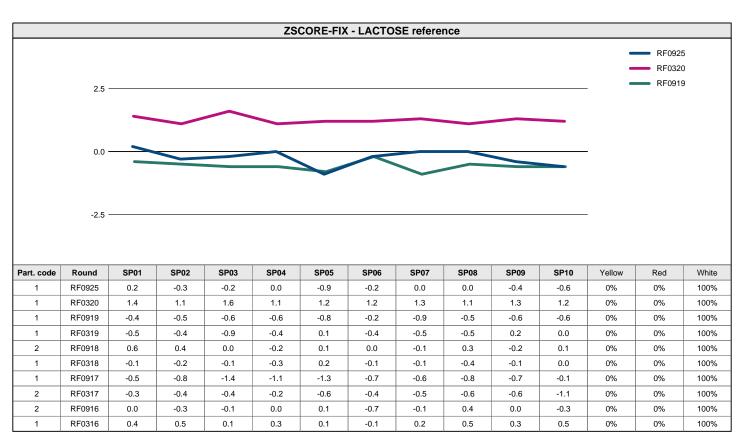


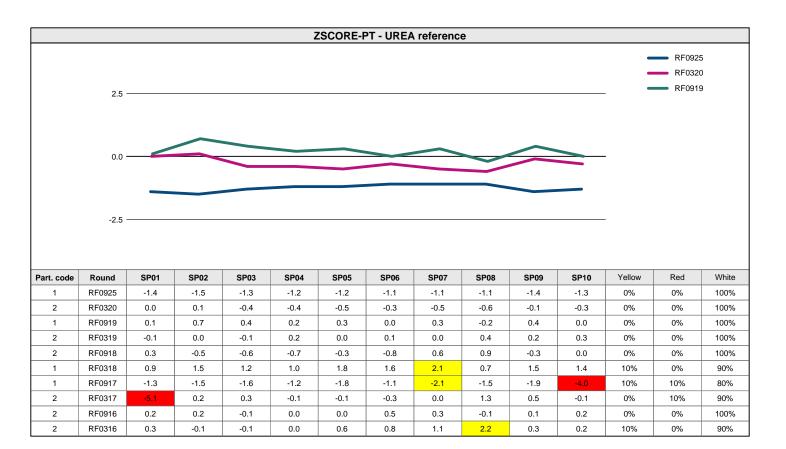


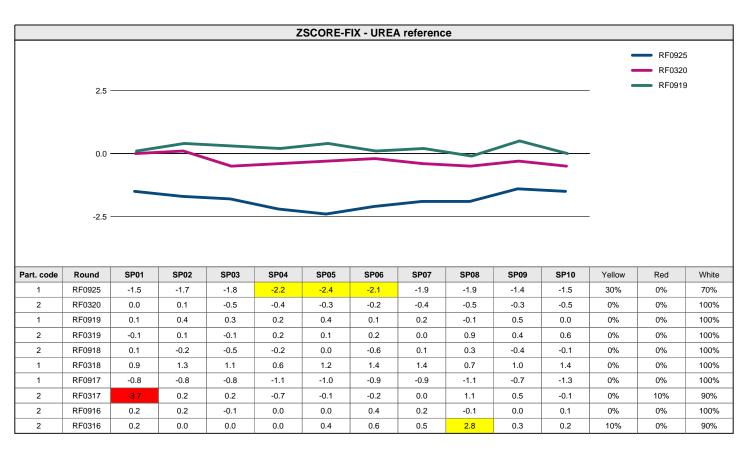


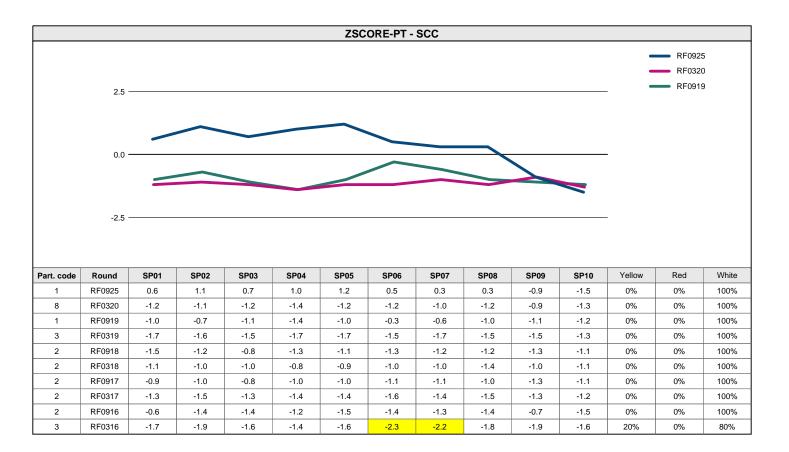


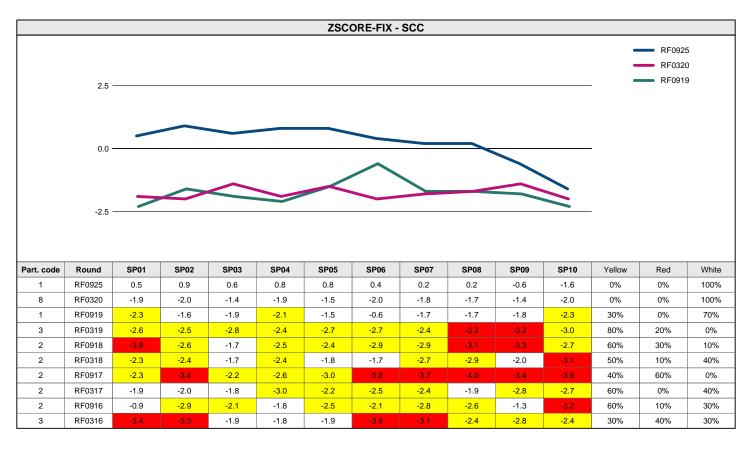


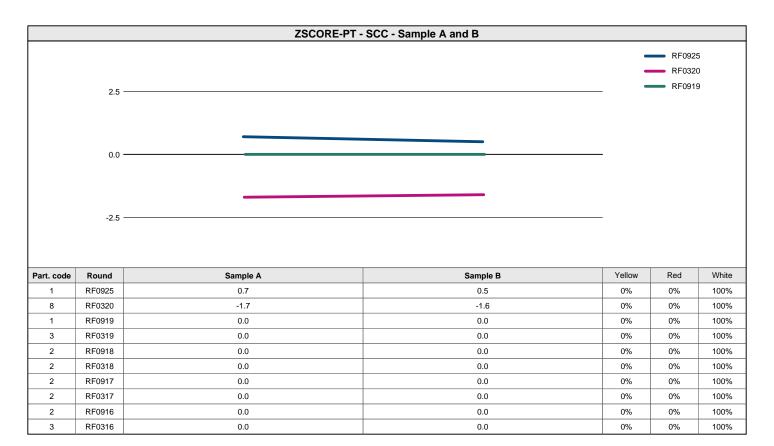


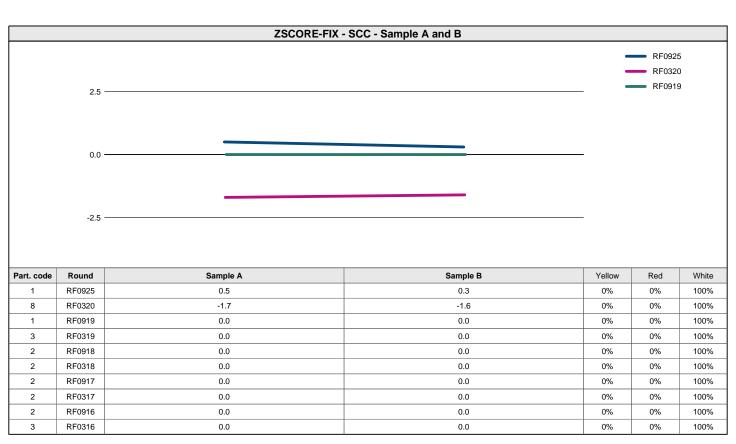














ICAR PROFICIENCY TESTING SCHEME

September 2025

Raw Milk

Determination of FAT CONTENT Röse Gottlieb method

Sending date of statistical treatment : 25^{th} September 2025

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

ICAR Staff Silvia Orlandini pt@icar.org silvia@icar.org

Proficiency test accreditated ISO 17043





Nb	%	N°	d	Sd	D	Method	The table should be studied i
1	8	8	- 0,002	0,004	0,005	В	laboratories are located acco
2	15	9	- 0,002	0,006	0,006	A	the limits of which are :
3	23	7	+ 0,005	0,006	0,008	A	
4	31	1	+ 0,007	0,009	0,012	A	
5	38	11	+ 0,011	0,005	0,012	В	
6	46	10	+ 0,010	0,008	0,013	A	+/- 0,020 g / 100 g
7	54	12	+ 0,010	0,010	0,014	В	
8	62	2	- 0,006	0,016	0,017	В	
9	69	5	- 0,017	0,010	0,019	В	REF: Assigned values are rol
10	77	6	+ 0,024	0,021	0,032	В	algorithm A of standard ISO
11	85	13	- 0,032	0,028	0,043	В	method ISO 1211 IDF 1, aft
12	92	3	- 0,857	1,854	2,042	A	at 5% risk level,
13	100	4	- 0,908	1,859	2,069	В	

The table should be studied in parallel with figure 1 where the aboratories are located according to an acceptability area (or target)

+/- 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 13 laboratories using the reference method ISO 1211|IDF 1, after outliers discarging using Grubbs test at 5% risk level,

A ISO 23318 | IDF 249

B ISO 1211 / IDF 1

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

(Nb : laboratory rank; % : relative rank) (N° : laboratory identification number)

 $(d\ et\ Sd\ :\ mean\ and\ standard\ deviation\ of\ the\ differences\ (laboratory\ \hbox{-reference}))$

(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} 0,006 Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) SR_{PT} 0,775

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

Sample Lab	1	2	3	4	5	6	7	8	FAT A	FAT B	Sr	NL
1	0,003	0,004	0,004	0,002	0,003	0,005	0,009	0,004	**	**	0,003	16
2	0,003	0,027	0,018	0,005	0,023 *	0,010	0,022	0,022	0,014	0,014	0,012	20
3	0,040 *	0,000	0,000	0,060 *	0,000	0,000	0,020	0,000	0,050 *	0,000	0,020	20
4	0,008	0,014	0,003	0,005	0,015	0,004	0,036	0,014	0,003	0,014	0,011	20
5	0,004	0,004	0,011	0,002	0,008	0,001	0,007	0,010	0,007	0,008	0,005	20
6	0,009	0,003	0,009	0,003	0,000	0,012	0,002	0,003	0,006	0,000	0,004	20
7	0,009	0,017	0,016	0,003	0,010	0,005	0,006	0,007	0,013	0,001	0,007	20
8	0,000	0,009	0,006	0,004	0,003	0,002	0,002	0,003	**	**	0,003	16
9	0,001	0,002	0,002	0,026 *	0,003	0,003	0,009	0,010	0,018	0,002	0,008	20
10	0,001	0,001	0,002	0,005	0,001	0,002	0,001	0,004	0,019	0,002	0,005	20
11	0,013	0,007	0,002	0,000	0,003	0,001	0,010	0,005	0,014	0,002	0,005	20
12	**	**	**	**	**	**	**	**	**	**		
13	0,001	0,003	0,003	0,007	0,003	0,001	0,003	0,002	0,002	0,002	0,002	20
Sr	0,009	0,008	0,006	0,014	0,006	0,004	0,010	0,006	0,014	0,005		232
NE	24	24	24	24	24	24	24	24	20	20		
L	0,016	0,028	0,022	0,010	0,016	0,013	0,037	0,023	0,029	0,017		

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 betwen two replicates=0,043 according ISO 1211 IDF 1D 2010

<u>Table III:</u> Means of the replicates in g / 100 g

Sample Lab	1	2	3	4	5	6	7	8	FAT A	FAT B
1	2,131	2,801	4,159	3,476	4,670	1,454	4,516	3,814		
2	2,141	2,797	4,157	3,486	4,635	1,462	4,466	3,778	6,051	8,092
3	2,160	2,820	4,170	3,430	4,660	1,460	4,550	3,790	2,405	3,110
4	2,115	2,750 *	4,106	3,422	4,626	1,415 *	4,446	3,761	2,371	3,030
5	2,116	2,786	4,125	3,456	4,633	1,455	4,498	3,788	6,023	8,076
6	2,142	2,812	4,164	3,486	4,682	1,467	4,516	3,816	6,113	8,172
7	2,125	2,799	4,160	3,478	4,666	1,472	4,511	3,798	6,062	8,107
8	2,120	2,794	4,150	3,466	4,654	1,462	4,502	3,802		
9	2,120	2,803	4,145	3,476	4,654	1,461	4,498	3,795	6,045	8,106
10	2,127	2,805	4,167	3,477	4,671	1,467	4,515	3,813	6,056	8,128
11	2,142	2,814	4,165	3,472	4,670	1,468	4,519	3,814	6,059	8,111
12	2,130	2,800	4,160	3,480	4,660	1,470	4,510	3,810	6,090	8,110
13	2,116	2,791	4,122	3,446	4,617	1,469	4,464	3,780	5,984	8,016
М	2,129	2,802	4,150	3,465	4,653	1,464	4,501	3,797	5,387	7,187
REF.	2,128	2,801	4,151	3,467	4,654	1,464	4,501	3,797	6,055	8,104
SD	0,013	0,010	0,020	0,021	0,020	0,006	0,028	0,017	1,483	2,036

M = mean per sample REF. = reference values

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

 \underline{REF} : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 13 laboratories using the reference method ISO 1211 | IDF 1, after outliers discarging using Grubbs test at 5 % risk level.

Table IV: Outlier identification

Sample	1	2	3	4	5	6	7	8	FAT A	FAT B
Outliers	2			3.0					2	
Cochran	3			3; 9					3	
Outlier		1				4				
Grubbs		4				4				
sr	0,004	0,007	0,006	0,003	0,004	0,004	0,010	0,006	0,009	0,005
SR	0,011	0,012	0,021	0,020	0,021	0,006	0,030	0,018	1,227	2,122

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

Sample Lab code	1	2	3	4	5	6	7	8	FAT A	FAT B	d	Sd _{lab}	t
1	+ 0,002	- 0,000	+ 0,008	+ 0,009	+ 0,016	- 0,010	+ 0,014	+ 0,017			+ 0,007	0,009	2,10
2	+ 0,012	- 0,005	+ 0,006	+ 0,018	- 0,019	- 0,002	- 0,035	- 0,019	- 0,004	- 0,012	- 0,006	0,016	1,18
3	+ 0,032	+ 0,019	+ 0,019	- 0,037	+ 0,006	- 0,004	+ 0,049	- 0,007	- 3,650	- 4,994	- 0,857	1,854	1,46
4	- 0,013	- 0,051	- 0,046	- 0,046	- 0,028	- 0,049	- 0,055	- 0,036	- 3,684	- 5,074	- 0,908	1,859	1,55
5	- 0,012	- 0,015	- 0,027	- 0,011	- 0,021	- 0,009	- 0,004	- 0,009	- 0,032	- 0,028	- 0,017	0,010	5,59
6	+ 0,013	+ 0,010	+ 0,012	+ 0,018	+ 0,028	+ 0,003	+ 0,015	+ 0,018	+ 0,058	+ 0,068	+ 0,024	0,021	3,61
7	- 0,004	- 0,003	+ 0,009	+ 0,010	+ 0,012	+ 0,008	+ 0,010	+ 0,000	+ 0,007	+ 0,002	+ 0,005	0,006	2,82
8	- 0,008	- 0,008	- 0,001	- 0,001	- 0,000	- 0,002	+ 0,001	+ 0,004			- 0,002	0,004	1,30
9	- 0,009	+ 0,002	- 0,006	+ 0,009	- 0,000	- 0,003	- 0,004	- 0,002	- 0,010	+ 0,002	- 0,002	0,006	1,25
10	- 0,002	+ 0,003	+ 0,016	+ 0,009	+ 0,017	+ 0,003	+ 0,013	+ 0,016	+ 0,001	+ 0,024	+ 0,010	0,008	3,76
11	+ 0,013	+ 0,012	+ 0,014	+ 0,005	+ 0,016	+ 0,004	+ 0,018	+ 0,016	+ 0,004	+ 0,007	+ 0,011	0,005	6,30
12	+ 0,002	- 0,001	+ 0,009	+ 0,013	+ 0,006	+ 0,006	+ 0,009	+ 0,013	+ 0,035	+ 0,006	+ 0,010	0,010	3,09
13	- 0,013	- 0,011	- 0,030	- 0,022	- 0,037	+ 0,005	- 0,038	- 0,017	- 0,071	- 0,088	- 0,032	0,028	3,59
d	+ 0,001	+ 0,000	- 0,001	- 0,002	- 0,000	- 0,000	- 0,000	- 0,001	- 0,668	- 0,917	- 0,160	0,776	
Sd	0,013	0,010	0,020	0,021	0,020	0,006	0,028	0,017	1,483	2,036	0,797		

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

Upper limits : $\frac{-}{d} = +/-0.02 \text{ g} / 100 \text{ g}$ Sd = 0.03 g / 100g

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

Sample cab	1	2	3	4	5	6	7	8	FAT A	FAT B
1	+0,16	-0,03	+0,39	+0,43	+0,79	-1,76	+0,52	+0,98		
2	+0,91	-0,49	+0,30	+0,88	-0,96	-0,31	-1,27	-1,15	-0,00	-0,01
3	+2,37	+1,91	+0,94	-1,78	+0,31	-0,65	+1,77	-0,44	-2,46	-2,45
4	-0,99	-5,23	-2,26	-2,19	-1,41	-8,33	-1,99	-2,16	-2,48	-2,49
5	-0,92	-1,56	-1,31	-0,53	-1,04	-1,59	-0,13	-0,56	-0,02	-0,01
6	+0,98	+1,04	+0,62	+0,88	+1,42	+0,54	+0,54	+1,07	+0,04	+0,03
7	-0,28	-0,28	+0,44	+0,50	+0,61	+1,31	+0,36	+0,00	+0,00	+0,00
8	-0,62	-0,79	-0,05	-0,05	-0,01	-0,31	+0,03	+0,24		
9	-0,66	+0,17	-0,30	+0,43	-0,01	-0,57	-0,13	-0,15	-0,01	+0,00
10	-0,13	+0,33	+0,79	+0,45	+0,84	+0,54	+0,48	+0,92	+0,00	+0,01
11	+0,98	+1,24	+0,69	+0,23	+0,79	+0,63	+0,65	+0,95	+0,00	+0,00
12	+0,13	-0,13	+0,44	+0,62	+0,31	+1,1	+0,3	+0,74	+0,0	+0,00
13	-0,96	-1,10	-1,46	-1,04	-1,86	+0,8	-1,4	-1,03	-0,0	-0,04

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

n 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

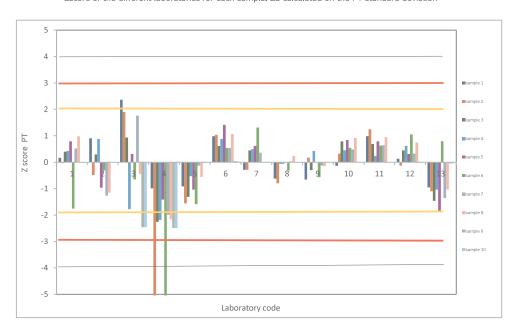


Table VII: Zscore of the different laboratories for each sample.

ZS calculated on the standard deviation of reproducibility of the method

Sample Lab	1	2	3	4	5	6	7	8	FAT A	FAT B
1	+0,11	-0,01	+0,40	+0,44	+0,79	-0,52	+0,72	+0,83		
2	+0,61	-0,24	+0,30	+0,92	-0,96	-0,09	-1,76	-0,97	-0,20	-0,61
3	+1,58	+0,94	+0,95	-1,86	+0,31	-0,19	+2,44	-0,37	-182,50	-249,71
4	-0,67	-2,56	-2,28	-2,28	-1,41	-2,44	-2,76	-1,82	-184,22	-253,71
5	-0,62	-0,76	-1,33	-0,56	-1,04	-0,47	-0,18	-0,47	-1,62	-1,41
6	+0,66	+0,51	+0,62	+0,92	+1,41	+0,16	+0,74	+0,90	+2,90	+3,39
7	-0,19	-0,14	+0,45	+0,52	+0,61	+0,38	+0,49	+0,00	+0,33	+0,12
8	-0,42	-0,39	-0,05	-0,06	-0,01	-0,09	+0,04	+0,20		
9	-0,44	+0,09	-0,30	+0,44	-0,01	-0,17	-0,18	-0,12	-0,50	+0,09
10	-0,09	+0,16	+0,80	+0,47	+0,84	+0,16	+0,67	+0,78	+0,03	+1,19
11	+0,66	+0,61	+0,70	+0,24	+0,79	+0,18	+0,89	+0,80	+0,20	+0,34
12	+0,08	-0,06	+0,45	+0,64	+0,31	+0,31	+0,4	+0,63	+1,75	+0,29
13	-0,64	-0,54	-1,48	-1,08	-1,86	+0,23	-1,9	-0,87	-3,55	-4,41

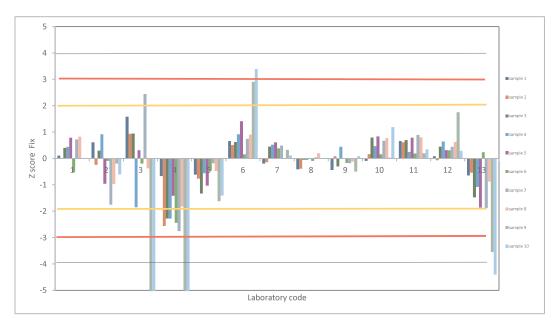
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR=0.02

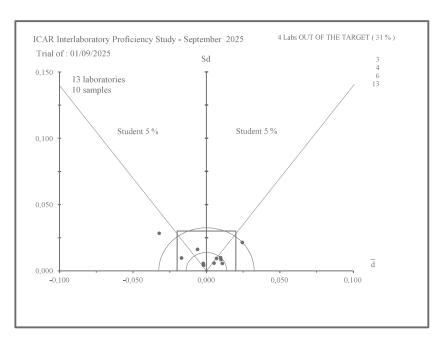
In yellow the values bigger or smaller than 2/-2

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





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



ICAR PROFICIENCY TESTING SCHEME

September 2025

Raw Milk

Determination of CRUDE PROTEIN CONTENT KJELDAHL Method

Sending date of statistical treatment : 25^{th} September 2025

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

ICAR Staff Silvia Orlandini pt@icar.org silvia@icar.org

Proficiency test accreditated ISO 17043





Nb	%	N°	d	Sd	D
1	8	8	- 0,001	0,005	0,005
2	15	1	- 0,006	0,004	0,008
3	23	6	- 0,006	0,008	0,010
4	31	11	+ 0,000	0,014	0,014
5	38	2	- 0,007	0,013	0,015
6	46	4	+ 0,014	0,006	0,015
7	54	12	+ 0,012	0,012	0,016
8	62	13	+ 0,009	0,014	0,016
9	69	5	- 0,016	0,007	0,017
10	77	7	- 0,017	0,009	0,019
11	85	10	+ 0,021	0,007	0,022
12	92	3	- 0,026	0,009	0,027
13	100	9	+ 0,041	0,021	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 :

+/- 0,025 g / 100 g for 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 $\,$ 13 laboratories using the reference method (ISO 8968-1 | IDF 20-1 and ISO 8968-3 | IDF 20-3), after outlier discarging 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)

 $(d\ et\ Sd\ :\ mean\ and\ standard\ deviation\ of\ the\ differences\ (laboratory\ \hbox{-reference}))$

(D : Euclidian distance to YX-axis origin $= SQUARE ROOT_*(d^2 + Sd^2))$

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

Repeatability standard deviation of this ICAR proficiency test (after Cochran elimination at 5 %) Sr_{PT} 0,006 Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) SR_{PT} 0,019

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

Sample cab	1	2	3	4	5	6	7	8	9	10	Sr	NL
1	0,001	0,004	0,005	0,014	0,001	0,009	0,010	0,002	0,003	0,009	0,005	20
2	0,000	0,019	0,038 *	0,006	0,019	0,000	0,006	0,000	0,006	0,006	0,011	20
3	0,016	0,006	0,003	0,016	0,014	0,004	0,028	0,018	0,006	0,003	0,010	20
4	0,004	0,014	0,005	0,013	0,001	0,006	0,002	0,011	0,006	0,023 *	0,008	20
5	0,005	0,000	0,003	0,003	0,006	0,001	0,003	0,003	0,002	0,004	0,002	20
6	0,003	0,005	0,004	0,006	0,001	0,003	0,005	0,002	0,007	0,009	0,004	20
7	0,000	0,000	0,006	0,006	0,006	0,006	0,013	0,000	0,013	0,000	0,005	20
8	0,013	0,000	0,013	0,013	0,006	0,000	0,006	0,006	0,000	0,000	0,006	20
9	0,004	0,002	0,009	0,037 *	0,020	0,010	0,013	0,010	0,011	0,004	0,011	20
10	0,007	0,007	0,008	0,007	0,003	0,011	0,011	0,004	0,010	0,009	0,006	20
11	**	**	**	**	**	**	**	**	**	**		
12	0,001	0,003	0,002	0,001	0,001	0,004	0,001	0,003	0,000	0,001	0,001	20
13	0,010	0,000	0,020	0,010	0,010	0,000	0,010	0,000	0,000	0,010	0,007	20
Sr	0,005	0,005	0,010	0,010	0,007	0,004	0,008	0,005	0,005	0,006		240
NE	24	24	24	24	24	24	24	24	24	24		
L	0,018	0,019	0,022	0,025	0,026	0,015	0,029	0,019	0,017	0,015		

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 $% \frac{1}{2}$ at 5 %

 $\ast \ast$: missing data

 $r: limit of \ repeatability, \ absolute \ difference \ betwen \ two \ replicates = 0,040 \ according ISO \ 8968-1 \ | \ IDF \ 20-1 \ | \ IDF$

<u>Table III:</u> Means of the replicates in g / 100 g

Sample Lab	1	2	3	4	5	6	7	8	9	10
1	2,676	2,869	3,543	3,245	3,641	3,251	3,638	3,429	2,765	3,053
2	2,673	2,855	3,515	3,238	3,640	3,247	3,659	3,439	2,785	3,053
3	2,660	2,853	3,530	3,232	3,628	3,229	3,612	3,401	2,742	3,035
4	2,697	2,882	3,556	3,262	3,658	3,270	3,662	3,453	2,786	3,092
5	2,654	2,845	3,538	3,239	3,636	3,235	3,643	3,424	2,750	3,055
6	2,677	2,872	3,533	3,244	3,625	3,261	3,643	3,434	2,767	3,059
7	2,667	2,858	3,531	3,244	3,621	3,244	3,611	3,420	2,763	3,050
8	2,673	2,871	3,535	3,260	3,646	3,260	3,646	3,442	2,775	3,062
9	2,697	2,890	3,594	3,293	3,688	3,305	3,696	3,505 *	2,848 *	3,072
10	2,695	2,896	3,571	3,273	3,673	3,283	3,657	3,451	2,803	3,087
11	2,660	2,850	3,530	3,270	3,650	3,270	3,640	3,450	2,780	3,080
12	2,688	2,904	3,566	3,262	3,641	3,276	3,669	3,451	2,775	3,062
13	2,675	2,870	3,560	3,265	3,675	3,250	3,685	3,450	2,770	3,065
М	2,676	2,870	3,546	3,256	3,648	3,260	3,651	3,437	2,772	3,063
REF.	2,676	2,870	3,545	3,255	3,647	3,259	3,650	3,438	2,772	3,063
SD	0,014	0,018	0,022	0,018	0,020	0,021	0,025	0,016	0,016	0,016

M = mean per sample REF. = reference values

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

Table IV : Outlier identification

Sample	1	2	Т	3	П	4	П	5	1	6	П	7	1	8	П	9	П	10	
Outliers			Т		П		П		1		П		٦		П		٦		_
Cochran																		4	
Outlier			Τ		П				1		П		1	9	П	a	I		
Grubbs									ı					9		9			
sr	0,005	0,00	5	0,006		0,007		0,007		0,004		0,008		0,005		0,004		0,004	_
SR	0,015	0,01	3	0,021	П	0,015		0,022	1	0,022		0,026	1	0,017	П	0,017	1	0,014	

ncy Test September 2025

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

Sample cab	1	2	3	4	5	6	7	8	9	10	d	Sd _{lab}	t
1	+ 0,000	- 0,001	- 0,002	- 0,010	- 0,006	- 0,008	- 0,012	- 0,009	- 0,006	- 0,010	- 0,006	0,004	4,67
2	- 0,003	- 0,015	- 0,030	- 0,017	- 0,007	- 0,012	+ 0,009	+ 0,001	+ 0,013	- 0,011	- 0,007	0,013	1,81
3	- 0,016	- 0,017	- 0,015	- 0,024	- 0,019	- 0,030	- 0,039	- 0,037	- 0,030	- 0,029	- 0,026	0,009	9,52
4	+ 0,020	+ 0,012	+ 0,011	+ 0,007	+ 0,010	+ 0,011	+ 0,011	+ 0,015	+ 0,014	+ 0,028	+ 0,014	0,006	7,26
5	- 0,022	- 0,025	- 0,007	- 0,016	- 0,012	- 0,024	- 0,007	- 0,014	- 0,022	- 0,008	- 0,016	0,007	6,79
6	+ 0,001	+ 0,002	- 0,012	- 0,011	- 0,022	+ 0,002	- 0,007	- 0,004	- 0,004	- 0,005	- 0,006	0,008	2,58
7	- 0,009	- 0,012	- 0,014	- 0,011	- 0,027	- 0,015	- 0,039	- 0,018	- 0,009	- 0,014	- 0,017	0,009	5,64
8	- 0,003	+ 0,001	- 0,010	+ 0,005	- 0,001	+ 0,001	- 0,004	+ 0,004	+ 0,004	- 0,001	- 0,001	0,005	0,36
9	+ 0,021	+ 0,020	+ 0,049	+ 0,038	+ 0,040	+ 0,046	+ 0,045	+ 0,066	+ 0,076	+ 0,009	+ 0,041	0,021	6,26
10	+ 0,018	+ 0,026	+ 0,026	+ 0,018	+ 0,026	+ 0,024	+ 0,007	+ 0,013	+ 0,031	+ 0,023	+ 0,021	0,007	9,18
11	- 0,016	- 0,020	- 0,015	+ 0,015	+ 0,003	+ 0,011	- 0,010	+ 0,012	+ 0,008	+ 0,017	+ 0,000	0,014	0,08
12	+ 0,011	+ 0,034	+ 0,021	+ 0,007	- 0,006	+ 0,017	+ 0,018	+ 0,012	+ 0,004	- 0,001	+ 0,012	0,012	3,23
13	- 0,001	+ 0,000	+ 0,015	+ 0,010	+ 0,028	- 0,009	+ 0,035	+ 0,012	- 0,002	+ 0,002	+ 0,009	0,014	2,02
d	- 0,000	+ 0,000	+ 0,001	+ 0,001	+ 0,001	+ 0,001	+ 0,000	- 0,001	+ 0,000	+ 0,000	+ 0,001	0,020	
Sd	0,014	0,018	0,022	0,018	0,020	0,021	0,025	0,016	0,016	0,016	0,019		

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

Upper limits : d = +/- 0,025 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 cab										
code	1	2	3	4	5	6	7	8	9	10
1	+0,01	-0,05	-0,07	-0,58	-0,28	-0,37	-0,51	-0,57	-0,39	-0,63
2	-0,22	-0,82	-1,37	-0,98	-0,36	-0,55	+0,35	+0,04	+0,80	-0,67
3	-1,14	-0,95	-0,71	-1,34	-0,95	-1,44	-1,57	-2,31	-1,82	-1,82
4	+1,43	+0,65	+0,49	+0,42	+0,51	+0,53	+0,47	+0,95	+0,86	+1,79
5	-1,55	-1,38	-0,31	-0,93	-0,57	-1,16	-0,29	-0,85	-1,32	-0,51
6	+0,05	+0,09	-0,56	-0,64	-1,08	+0,11	-0,30	-0,26	-0,27	-0,30
7	-0,66	-0,64	-0,63	-0,62	-1,30	-0,71	-1,60	-1,15	-0,56	-0,87
8	-0,22	+0,06	-0,48	+0,29	-0,05	+0,05	-0,17	+0,24	+0,22	-0,06
9	+1,47	+1,09	+2,25	+2,18	+1,97	+2,21	+1,84	+4,13	+4,64	+0,54
10	+1,27	+1,40	+1,19	+1,03	+1,27	+1,16	+0,27	+0,79	+1,91	+1,46
11	-1,14	-1,09	-0,69	+0,85	+0,14	+0,52	-0,42	+0,74	+0,51	+1,05
12	+0,78	+1,84	+0,97	+0,42	-0,28	+0,81	+0,74	+0,77	+0,22	-0,06
13	-0,09	+0,00	+0,69	+0,56	+1,36	-0,43	+1,41	+0,74	-0,11	+0,10

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 cab	1	2	3	4	5	6	7	8	9	10
1	+0,00	-0,05	-0,09	-0,57	-0,32	-0,43	-0,69	-0,51	-0,35	-0,55
2	-0,17	-0,83	-1,65	-0,96	-0,41	-0,65	+0,48	+0,04	+0,73	-0,59
3	-0,91	-0,96	-0,85	-1,31	-1,08	-1,68	-2,14	-2,06	-1,65	-1,61
4	+1,14	+0,66	+0,59	+0,41	+0,58	+0,61	+0,64	+0,85	+0,78	+1,57
5	-1,24	-1,40	-0,37	-0,91	-0,64	-1,36	-0,39	-0,76	-1,20	-0,45
6	+0,04	+0,09	-0,67	-0,62	-1,23	+0,13	-0,41	-0,23	-0,24	-0,27
7	-0,53	-0,65	-0,76	-0,60	-1,48	-0,82	-2,18	-1,03	-0,51	-0,76
8	-0,17	+0,06	-0,58	+0,28	-0,06	+0,06	-0,23	+0,21	+0,20	-0,06
9	+1,17	+1,10	+2,71	+2,12	+2,25	+2,58	+2,52	+3,69	+4,22	+0,48
10	+1,01	+1,42	+1,44	+1,01	+1,45	+1,36	+0,37	+0,71	+1,74	+1,29
11	-0,91	-1,11	-0,83	+0,83	+0,15	+0,61	-0,57	+0,66	+0,46	+0,92
12	+0,62	+1,86	+1,17	+0,41	-0,32	+0,95	+1,01	+0,69	+0,20	-0,06
13	-0,07	+0,00	+0,83	+0,55	+1,54	-0,50	+1,93	+0,66	-0,10	+0,09

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

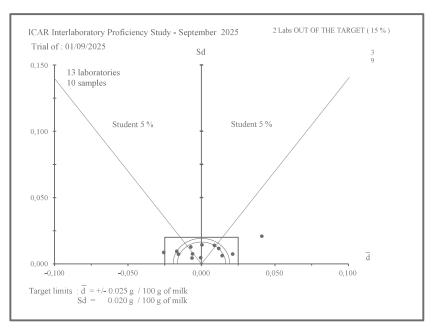
In yellow the values bigger or smaller than 2/-2

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





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

<u>Table VIII: Relative recovery of nitrogen on pure solutions</u>

N°	TRYP	GLY	SO4		
1	99,2	99,3	100,0		
2	99,1	99,1	100,4		
3	98,8	99,2	99,4		
4	99,4	99,3	100,0		
5	99,6	99,6	99,6		
6	99,1	99,7	100,4		
7	97,8	99,5	100,1		
8	99,2	99,8	100,6		
9	100,2	99,9	100,9		
10					
11	100,1	100,5	101,3		
12	98,2	100,6	100,0		
13					

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

GLY = Glycine solution to 5.60 g N/I

SO4 = Ammonium sulfate solution to 5.60 g N/l

TRYP: recovery 98 à 101 % GLY: recovery 99 à 101 % SO4: recovery 99 à 101 %



ICAR PROFICIENCY TESTING SCHEME

September 2025

Raw Milk

Determination of LACTOSE CONTENT

Sending date of statistical treatment : 25th September 2025

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

ICAR Staff Silvia Orlandini pt@icar.org silvia@icar.org





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

Nb	%	N°	d	Sd	D	Method
1	10	3	- 0,003	0,011	0,011	ISO 22662 / IDF 198
2	20	1	- 0,012	0,016	0,019	HPLC
3	30	8	+ 0,022	0,025	0,033	Enzymatic
4	40	6	+ 0,027	0,029	0,040	ISO 22662 / IDF 198
5	50	9	- 0,029	0,033	0,043	LC-PAD
6	60	10	- 0,046	0,010	0,047	N.C.
7	70	2	- 0,047	0,012	0,048	ISO 22662 / IDF 198
8	80	4	- 0,027	0,045	0,053	ISO 26462 / IDF 214
9	90	7	+ 0,055	0,016	0,058	Enzymatic
10	100	5	+ 0,057	0,017	0,060	Lane-Eynon

(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 :

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

 \underline{REF} : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 10 laboratories , after outliers discarging 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} 0,019 Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) SR_{PT} 0,048

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

Sample cab	11	12	13	14	15	16	17	18	19	20	Sr	NL
1	0,006	0,003	0,008	0,001	0,018	0,003	0,002	0,001	0,003	0,018	0,006	20
2	0,013	0,010	0,012	0,008	0,003	0,008	0,001	0,027	0,008	0,002	0,008	20
3	0,008	0,001	0,004	0,009	0,005	0,023	0,023	0,008	0,001	0,010	0,008	20
4	0,010	0,004	0,054 *	0,004	0,008	0,036	0,018	0,038	0,048	0,131	0,036	20
5	0,007	0,007	0,008	0,003	0,009	0,007	0,007	0,007	0,022	0,010	0,007	20
6	0,000	0,000	0,005	0,006	0,000	0,002	0,005	0,004	0,006	0,001	0,003	20
7	0,010	0,038	0,011	0,015	0,013	0,054	0,024	0,019	0,039	0,006	0,019	20
8	0,002	0,016	0,019	0,018	0,008	0,012	0,007	0,013	0,010	0,014	0,009	20
9	0,000	0,034	0,076 *	0,093 *	0,045	0,033	0,109 *	0,009	0,041	0,125	0,049	20
10	0,010	0,000	0,000	0,000	0,010	0,010	0,010	0,000	0,000	0,010	0,005	20
Sr	0,006	0,012	0,022	0,022	0,012	0,018	0,026	0,012	0,018	0,041		200
NE	20	20	20	20	20	20	20	20	20	20		
L	0,019	0,043	0,023	0,022	0,023	0,061	0,033	0,042	0,061	0,142		

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

NL : number of measurements per laboratory

 $\ensuremath{\mathsf{L}}$: 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 $% \frac{1}{2}$ at 5 %

** : missing data

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

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

Sample cab	11	12	13	14	15	16	17	18	19	20	LACT
1	5,181	5,039	4,960	4,911	5,069	4,861	4,787	4,827	4,883	4,825	4,913
2	5,143	5,025	4,923	4,848	5,065	4,823	4,735	4,781	4,839	4,807	4,890
3	5,173	5,030	4,956	4,902	5,122	4,886	4,781	4,839	4,892	4,851	4,905
4	5,086	4,948	4,922	4,877	5,079	4,882	4,782	4,796	4,916	4,895	4,927
5	5,208	5,115	5,011	4,941	5,191	4,942	4,868	4,896	4,951	4,910	4,977
6	5,125	5,107	4,979	4,942	5,143	4,887	4,818	4,879	4,942	4,904	4,951
7	5,250	5,100	5,029	4,967	5,176	4,890	4,845	4,880	4,950	4,924	4,902
8	5,239	5,069	5,013	4,938	5,152	4,897	4,768	4,836	4,916	4,849	
9	5,155	5,062	4,968	4,904	5,069	4,835	4,772	4,800	4,858	4,748	4,838
10	5,145	5,000	4,930	4,870	5,065	4,825	4,725	4,780	4,850	4,805	
М	5,170	5,049	4,969	4,910	5,113	4,873	4,788	4,831	4,900	4,852	
REF.	5,170	5,052	4,969	4,910	5,113	4,871	4,787	4,831	4,900	4,853	4,912
SD	0,051	0,052	0,039	0,037	0,050	0,037	0,045	0,043	0,042	0,057	

M = mean per sample

REF. = reference values

SD = standard deviation per sample

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

 $\overline{\text{REE}}$: Assigned values are robust average values per sample according to algorithm A of standard ISO 135 of 10 laboratories, after outliers discarging using Grubbs test at 5 % risk level.

Table IV: Outlier identification

Sample	11	12	13	14	15	16	17	18	19	20
Outliers			1 0	g						
Cochran			4.9	9			9			
Outlier										
Grubbs										
sr	0,006	0,012	0,007	0,006	0,007	0,018	0,010	0,012	0,018	0,041
SR	0,051	0,053	0,040	0,040	0,050	0,039	0,048	0,043	0,044	0,064

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

Sample cab	11	12	13	14	15	16	17	18	19	20	d	Sd _{lab}	t
1	+ 0,011	- 0,013	- 0,009	+ 0,001	- 0,044	- 0,011	- 0,000	- 0,005	- 0,017	- 0,028	- 0,012	0,016	2,35
2	- 0,028	- 0,027	- 0,046	- 0,062	- 0,048	- 0,048	- 0,053	- 0,051	- 0,061	- 0,046	- 0,047	0,012	12,68
3	+ 0,003	- 0,022	- 0,013	- 0,008	+ 0,009	+ 0,014	- 0,007	+ 0,008	- 0,008	- 0,002	- 0,003	0,011	0,76
4	- 0,084	- 0,104	- 0,047	- 0,033	- 0,034	+ 0,011	- 0,005	- 0,035	+ 0,016	+ 0,042	- 0,027	0,045	1,92
5	+ 0,037	+ 0,063	+ 0,042	+ 0,031	+ 0,078	+ 0,070	+ 0,080	+ 0,064	+ 0,051	+ 0,057	+ 0,057	0,017	10,74
6	- 0,045	+ 0,055	+ 0,010	+ 0,032	+ 0,030	+ 0,016	+ 0,030	+ 0,048	+ 0,042	+ 0,050	+ 0,027	0,029	2,89
7	+ 0,080	+ 0,048	+ 0,060	+ 0,057	+ 0,063	+ 0,019	+ 0,058	+ 0,048	+ 0,050	+ 0,071	+ 0,055	0,016	10,72
8	+ 0,069	+ 0,017	+ 0,044	+ 0,028	+ 0,039	+ 0,026	- 0,020	+ 0,004	+ 0,016	- 0,004	+ 0,022	0,025	2,75
9	- 0,015	+ 0,010	- 0,001	- 0,006	- 0,044	- 0,037	- 0,016	- 0,032	- 0,042	- 0,106	- 0,029	0,033	2,80
10	- 0,025	- 0,052	- 0,039	- 0,040	- 0,048	- 0,046	- 0,062	- 0,051	- 0,050	- 0,048	- 0,046	0,010	14,98
d	- 0,000	- 0,002	- 0,000	+ 0,000	- 0,000	+ 0,001	+ 0,001	+ 0,000	- 0,000	- 0,001	- 0,000	0,044	
Sd	0,051	0,052	0,039	0,037	0,050	0,037	0,045	0,043	0,042	0,057	0,046		

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

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

Sr = 0.022 g / 100 g SR = 0.047 g / 100 g ISO 22662 IDF 198: Precision of the method:

28,

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

Sample cab	11	12	13	14	15	16	17	18	19	20
1	+0,21	-0,25	-0,23	+0,02	-0,88	-0,29	-0,00	-0,11	-0,41	-0,49
2	-0,55	-0,51	-1,19	-1,65	-0,97	-1,30	-1,18	-1,19	-1,45	-0,81
3	+0,05	-0,42	-0,34	-0,22	+0,17	+0,38	-0,15	+0,19	-0,19	-0,04
4	-1,66	-1,99	-1,22	-0,88	-0,68	+0,28	-0,11	-0,83	+0,39	+0,73
5	+0,73	+1,20	+1,09	+0,82	+1,57	+1,88	+1,79	+1,51	+1,23	+1,01
6	-0,89	+1,06	+0,25	+0,86	+0,61	+0,42	+0,68	+1,13	+1,02	+0,89
7	+1,56	+0,93	+1,54	+1,52	+1,26	+0,50	+1,29	+1,14	+1,20	+1,25
8	+1,35	+0,33	+1,13	+0,76	+0,79	+0,69	-0,44	+0,10	+0,39	-0,07
9	-0,30	+0,20	-0,02	-0,17	-0,89	-0,99	-0,35	-0,74	-1,00	-1,86
10	-0,50	-0,99	-1,01	-1,06	-0,96	-1,25	-1,39	-1,20	-1,18	-0,85

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

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

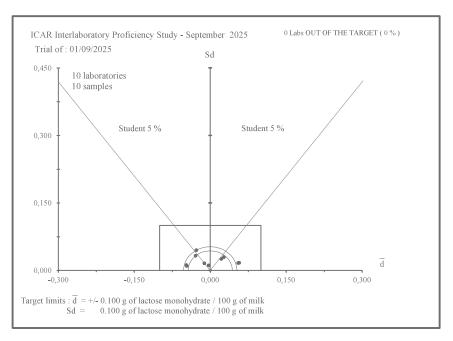
Sample cab	11	12	13	14	15	16	17	18	19	20
1	+0,23	-0,28	-0,19	+0,02	-0,93	-0,23	-0,00	-0,10	-0,36	-0,60
2	-0,59	-0,57	-0,98	-1,31	-1,03	-1,03	-1,12	-1,08	-1,29	-0,98
3	+0,06	-0,47	-0,28	-0,18	+0,18	+0,30	-0,14	+0,17	-0,17	-0,04
4	-1,80	-2,21	-1,00	-0,70	-0,72	+0,22	-0,11	-0,75	+0,35	+0,88
5	+0,79	+1,34	+0,89	+0,65	+1,65	+1,49	+1,71	+1,37	+1,10	+1,21
6	-0,97	+1,18	+0,20	+0,69	+0,64	+0,33	+0,65	+1,02	+0,90	+1,07
7	+1,69	+1,03	+1,27	+1,21	+1,33	+0,39	+1,23	+1,03	+1,06	+1,51
8	+1,46	+0,37	+0,93	+0,60	+0,83	+0,54	-0,42	+0,09	+0,35	-0,09
9	-0,33	+0,22	-0,02	-0,13	-0,94	-0,79	-0,33	-0,67	-0,89	-2,24
10	-0,54	-1,10	-0,83	-0,85	-1,02	-0,99	-1,32	-1,09	-1,05	-1,02

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





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



ICAR PROFICIENCY TESTING SCHEME

September 2025

Raw Milk

Determination of UREA CONTENT

Sending date of statistical treatment : 25th September 2025

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

ICAR Staff Silvia Orlandini pt@icar.org silvia@icar.org





Table I: Ranking of the laboratories Units : mg / dl

Nb	%	N°	d	Sd	D	Method
1	14	4	+ 0,62	0,47	0,78	Continous flow analyser
2	29	6	+ 1,04	0,34	1,09	ISO 14637 / IDF 195
3	43	5	- 1,22	0,41	1,29	ISO 14637 / IDF 195
4	57	3	- 1,96	1,19	2,30	Enzymatic UV Method
5	71	1	- 3,27	0,59	3,32	ISO 14637 / IDF 195
6	86	2	+ 3,45	1,12	3,63	ISO 14637 / IDF 195
7	100	7	- 3,63	1,37	3,88	Enzymatic

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 / dl for d and 1,50 mg / dl for Sd

REF: Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 4 laboratories using reference method (ISO 14637 | IDF 195 or V 04-217), after outlier discarging using Grubbs test at 5% risk level

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

(Nb : laboratory rank; % : relative rank)

(N°: laboratory identification number)

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

(D : Euclidian distance to YX-axis origin = SQUARE ROOT.($d^2 + Sd^2$))

<u>Note</u>: 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} 0,40 Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) SR_{PT} 2,79

<u>Table II :</u> REPEATABILITY - Absolute difference between replicates in mg / dl

Sample cab	21	22	23	24	25	26	27	28	29	30	Sr	NL
1	0,160	0,390	0,230	0,030	0,750	0,500	0,110	0,000	0,230	0,050	0,24	20
2	0,370	0,120	0,400	0,020	0,550	0,230	0,050	0,410	0,120	0,440	0,23	20
3	2,500 *	0,500	0,400	0,000	1,800	2,000 *	2,300 *	0,800	3,000	3,400	1,42	20
4	0,100	0,400	0,770	1,180	0,030	0,350	0,640	0,360	0,340	0,270	0,39	20
5	0,800	0,500	0,200	0,500	0,500	0,300	0,100	0,500	0,500	0,700	0,36	20
6	0,800	0,200	0,600	0,600	1,100	0,100	1,200	0,800	0,700	0,100	0,51	20
7	0,140	0,470	0,280	0,290	0,010	0,230	0,250	0,270	0,410	0,040	0,20	20
Sr	0,74	0,28	0,32	0,39	0,63	0,57	0,72	0,37	0,85	0,94		140
NE	14	14	14	14	14	14	14	14	14	14		
L	1,07	0,89	1,02	1,23	2,01	0,67	1,23	1,18	0,92	0,78		

Sr: repeatability standard deviation of each laboratory $\,$ limit 0,54 $\,$ mg/dl $\,$

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 $% \frac{1}{2}$ at 5 %

** : missing data

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

Table III: Means of the replicates in mg / dl

Sample cab	21	22	23	24	25	26	27	28	29	30
1	15,37	24,88	34,33	43,99	53,74	58,95	49,23	40,08	30,05	25,41
2	20,04	30,92	41,03	52,11	63,13	67,44	57,28	45,77	35,09	30,41
3	15,05	26,75	35,90	43,80	55,90	60,30	52,85	40,60	31,70	26,20
4	17,77	28,37	38,25	48,73	59,10	64,05	53,66	43,60	33,24	28,13
5	17,30	26,95	36,60	46,35	56,05	61,25	51,05	42,15	31,65	27,15
6	19,50	28,90	38,50	49,20	59,25	63,35	53,00	44,30	33,85	29,25
7	16,31	25,74	34,34	43,80	52,58	56,98	47,91	39,56	30,01	25,23
М	17,33	27,50	36,99	46,85	57,10	61,76	52,14	42,29	32,23	27,40
REF.	18,05	27,91	37,61	47,91	58,04	62,75	52,64	43,07	32,66	28,05
SD	1,93	2,05	2,44	3,26	3,63	3,49	3,10	2,35	1,92	1,97

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

<u>Table IV:</u> Outlier identification

Sample	21	22	23	24	25	26	27	28	29	30
Outliers	_									
Cochran	3					3	3			
Outlier										
Grubbs										
sr	0,35	0,28	0,32	0,39	0,63	0,22	0,40	0,37	0,30	0,25
SR	1.82	2.06	2,45	3,27	3,66	3,76	3,39	2,36	2.10	2.08

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

Sample cab	21	22	23	24	25	26	27	28	29	30	d	Sd _{lab}	t
1	- 2,68	- 3,04	- 3,29	- 3,93	- 4,31	- 3,80	- 3,41	- 2,99	- 2,61	- 2,65	- 3,27	0,59	17,64
2	. , , , ,	+ 3,01	+ 3,42	+ 4,20	+ 5,09	+ 4,69	+ 4,64	+ 2,69	+ 2,43		+ 3,45	1,12	9,72
3	- 3,00	- 1,16	- 1,71	- 4,11	- 2,14	- 2,45	+ 0,21	- 2,47	- 0,96	- 1,85	- 1,96	1,19	5,24
4	- 0,28	+ 0,46	+ 0,63	+ 0,82	+ 1,06	+ 1,30	+ 1,02	+ 0,53	+ 0,58	+ 0,07	+ 0,62	0,47	4,15
5	- 0,75	- 0,96	- 1,01	- 1,56	- 1,99	- 1,50	- 1,59	- 0,92	- 1,01	- 0,90	- 1,22	0,41	9,50
6	+ 1,45	+ 0,99	+ 0,89	+ 1,29	+ 1,21	+ 0,60	+ 0,36	+ 1,23	+ 1,19	+ 1,20	+ 1,04	0,34	9,79
7	- 1,74	- 2,18	- 3,27	- 4,12	- 5,47	- 5,77	- 4,73	- 3,52	- 2,65	- 2,82	- 3,63	1,37	8,39
d	- 0,72	- 0,41	- 0,62	- 1,06	- 0,94	- 0,99	- 0,50	- 0,78	- 0,43	- 0,66	- 0,71	2,52	
Sd	1 93	2.05	2 44	3.26	3.63	3.49	3 10	2 35	1 92	1 97	2 69		

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

Upper limits : d = +/- 2,50 mg / dl Sd = 1,50 mg / dl

ISO 14637 IDF 195: Precision of the method:

Sr = 0.54 mg / dl SR = 1.81 mg / dl

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

Sample cab	21	22	23	24	25	26	27	28	29	30
1	-1,39	-1,48	-1,35	-1,20	-1,18	-1,09	-1,10	-1,28	-1,36	-1,35
2	+1,03	+1,47	+1,40	+1,29	+1,40	+1,34	+1,50	+1,15	+1,26	+1,20
3	-1,56	-0,57	-0,70	-1,26	-0,59	-0,70	+0,07	-1,05	-0,50	-0,94
4	-0,15	+0,22	+0,26	+0,25	+0,29	+0,37	+0,33	+0,22	+0,30	+0,04
5	-0,39	-0,47	-0,42	-0,48	-0,55	-0,43	-0,51	-0,39	-0,52	-0,46
6	+0,75	+0,48	+0,36	+0,40	+0,33	+0,17	+0,12	+0,52	+0,62	+0,61
7	-0,90	-1,06	-1,34	-1,26	-1,50	-1,65	-1,53	-1,50	-1,38	-1,44

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

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 cab	21	22	23	24	25	26	27	28	29	30
1	-1,48	-1,68	-1,82	-2,17	-2,38	-2,10	-1,89	-1,65	-1,44	-1,46
2	+1,10	+1,66	+1,89	+2,32	+2,81	+2,59	+2,56	+1,49	+1,34	+1,30
3	-1,66	-0,64	-0,95	-2,27	-1,18	-1,35	+0,12	-1,37	-0,53	-1,02
4	-0,16	+0,25	+0,35	+0,45	+0,58	+0,72	+0,56	+0,29	+0,32	+0,04
5	-0,42	-0,53	-0,56	-0,86	-1,10	-0,83	-0,88	-0,51	-0,56	-0,50
6	+0,80	+0,55	+0,49	+0,71	+0,67	+0,33	+0,20	+0,68	+0,66	+0,66
7	-0,96	-1,20	-1,81	-2,27	-3,02	-3,19	-2,61	-1,94	-1,47	-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=1,81 $\,$

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

Figure 3:

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



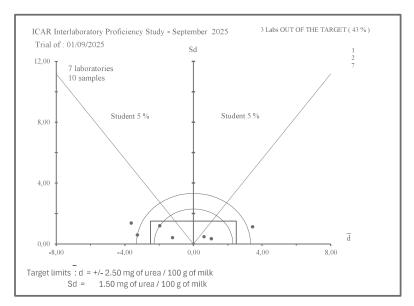


Figure 1: ACCURACY - Evaluation of the individual performances (to see table I).



ICAR PROFICIENCY TESTING SCHEME

September 2025

Raw Milk

Enumeration of SOMATIC CELLS

Sending date of statistical treatment : 25th September 2025

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

Silvia Orlandini pt@icar.org silvia@icar.org

Proficiency test accreditated ISO 17043





Table I: Ranking of the laboratories in %

Nb	%	Ν°	d	Sd	D	Method
1	2	2	+ 0%	1%	1%	В
2	3	37	+ 1%	1%	1%	В
3	5	23	- 1%	1%	1%	В
4	6	8	+ 1%	1%	1%	В
5	8	41	- 0%	1%	2%	В
6	9	7	+ 1%	1%	2%	В
7	11	16	- 1%	1%	2%	В
8	13	28	+ 0%	2%	2%	В
9	14	42	- 1%	2%	2%	В
10	16	4	+ 1%	2%	2%	В
11	17	25	- 1%	1%	2%	В
12	19	49	- 1%	1%	2%	В
13	20	58	+ 1%	1%	2%	В
14	22	9	+ 1%	2%	2%	В
15	23	36	- 1%	1%	2%	В
16	25	22	- 1%	2%	2%	В
17	27	12	+ 1%	2%	2%	В
18	28	62	+ 1%	2%	2%	В
19	30	32	- 1%	2%	2%	В
20	31	15	- 1%	2%	2%	В
21	33	45	- 2%	1%	2%	В
22	34	64	+ 1%	2%	2%	в
23	36	51	+ 2%	2%	3%	В
24	38	21	+ 1%	2%	3%	В
25	39	24	- 2%	2%	3%	В
26	41	19	- 2%	2%	3%	В
27	42	20	- 2%	2%	3%	В
28	44	27	- 2%	3%	3%	В
29	45	40	- 2%	2%	3%	В
30	47	50	+ 2%	3%	3%	В

(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

 $\label{eq:REE:assigned} \begin{array}{l} \text{REE: Assigned values are robust average values per sample according to} \\ \text{algorithm A of standard ISO 13528, of } 63 \text{ laboratories using reference metod} \\ \text{ISO 13366-1} \\ \text{IDF 148-1} \\ \text{and alternative method ISO 13366-2} \\ \text{IDF 148-2} \\ \text{after outlier discarging using Grubbs test at } 5\% \\ \text{risk level} \\ \end{array}$

Nh 0/0 Ν° А Sd D Method 11 32 + 3% В 33 52 29 - 2% 3% 4% В 34 53 13 - 1% 4% 4% В 35 55 10 + 3% 2% 4% 36 37 58 17 - 2% 4% В 38 59 - 3% 3% 4% В 39 61 46 + 3% 3% 4% В 40 55 - 0% 4% 4% В В 41 64 35 + 3% 4% 5% 66 В 42 38 - 4% 3% 5% 43 44 + 4% 4% 5% В 67 69 - 4% 4% 5% В 44 61 45 70 + 4% В 1 4% 6% + 4% 4% 6% В 46 72 31 В - 5% 47 73 26 3% 6% 48 75 3 - 5% 5% 7% В + 5% В 49 77 4% 7% 14 50 78 56 - 6% 7% 4% В 51 80 30 + 6% 6% 8% В 52 54 + 7% 8% 81 53 8% 84 53 + 7% 10% В 55 10% 56 88 57 9% 6% 11% В В 57 89 48 + 10% 6% 11% 58 91 59 8% 12% + 8% В 59 92 63 + 11% 9% 14% - 13% 8% 15% В 60 94 18 95 11% В 61 33 - 11% 16% 62 97 34 - 13% 11% 17% В 63 98 43 + 18% 13% 22% В 100 80% 100% 64 60

A ISO 13366-1 IDF 148-1

B ISO 13366-2 IDF 148-2

C Image Cytometry

Repeatability standard deviation of this ICAR proficiency test (after Cochran elimination at 5 %) Sr_{PT} 13 2% Repr<u>Note</u>: Limits are only indicative and so far do not constitute standard values; they indicate what is norm SR_{PT} 39 6% reachable by labs for their self evaluation.

 $\underline{\textbf{Table IIa:}} \qquad \qquad \textbf{REPEATABILITY - Absolute difference between replicates in 10} \ ^3 \, \text{cells / ml}$

Sample Lab	31	32	33	34	35	36	37	38	39	40	Sr	NL
1	5	11	16	27	3	10	7	1	1	1	8	20
2	4	16	16	17	23	4	15	7	6	6	9	20
3	15	17	7	7	39	19	7	3	9	3	11	20
4	18	1	28	36	4	11	0	3	0	4	11	20
5	16	21	13	11	5	21	7	19	5	2	10	20
6	19	8	12	59	8	2	5	4	9	5	15	20
7	19	13	8	1	27	1	2	1	2	0	8	20
8	12	5	21	18	10	3	11	2	1	5	8	20
9	5	7	6	4	6	5	6	7	4	2	4	20
10	45	12	17	69	60	32	20	8	0	11 9	25	20
11 12	11 24	11 13	2	69 15	10 9	10 1	25 3	12 10	5 7	7	17 8	20 20
13	5	30	37	5	17	8	7	2	3	2	12	20
14	27	16	0	44	16	14	1	6	6	6	13	20
15	31	55	0	23	19	33	7	19	11	2	18	20
16	4	1	4	8	2	11	3	4	6	3	4	20
17	25	14	6	4	21	49	6	4	5	7	14	20
18	0	6	5	9	80	10	1	2	1	6	18	20
19	4	4	0	2	17	27	12	2	10	15	9	20
20	10	4	14	27	13	33	9	0	16	0	11	20
21	15	16	6	34	24	40	5	16	9	6	15	20
22	1	7	36	4	14	3	3	2	2	1	9	20
23	13	13	11	19	8	2	6	6	0	0	7	20
24	13	12	21	1	9	0	11	2	7	2	7	20
25	6	22	5	32	18	4	3	7	2	1	10	20
26	1	3	3	29	4	8	4	7	3	6	7	20
27	13	30	26	31	8	29	7	14	5	6	14	20
28 29	21 2	4 33	28 48	3 13	22 21	10 7	3 6	0 4	6 22	3 2	10 15	20 20
30	25	1	48	3	57	5	6	2	6	4	18	20
31	1	8	6	14	14	2	20	1	0	4	7	20
32	11	18	42	5	24	11	1	4	3	7	12	20
33	17	3	6	14	1	22	27	9	17	2	10	20
34	3	33	26	23	39	8	33	0	10	6	16	20
35	10	20	27	8	11	5	5	3	6	4	9	20
36	2	11	14	8	6	0	4	2	1	9	5	20
37	19	1	25	2	7	3	6	1	5	3	7	20
38	7	6	1	13	12	3	4	2	1	0	5	20
39	19	29	15	8	22	12	15	1	13	4	11	20
40	13	6	28	6	3	11	14	14	6	4	9	20

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

Sample Lab	31	32	33	34	35	36	37	38	39	40	Sr	NL
41	18	15	30	22	58	31	24	2	11	1	19	20
42	11	11	5	19	7	10	20	8	10	6	8	20
43	15	16	34	1	100	10	12	1	0	11	24	20
44	16	24	1	15	6	2	17	10	1	1	9	20
45	11	16	20	27	21	0	6	6	2	1	10	20
46	44	17	14	56	72	10	13	6	9	6	24	20
47	22	16	43	22	28	14	3	15	5	5	15	20
48	2	4	13	12	10	5	2	0	1	1	5	20
49	46	35	25	5	40	7	19	8	21	0	18	20
50	20	28	29	37	28	19	32	8	3	11	17	20
51	4	11	10	7	27	2	5	11	2	1	8	20
52	4	28	18	33	78	7	17	4	2	14	21	20
53	23	5	44	13	51	3	1	5	0	7	16	20
54	47	14	6	22	6	17	16	1	8	3	13	20
55	0	0	0	0	0	0	0	0	0	0	0	20
56	0	0	0	0	0	0	0	0	0	0	0	20
57	4	0	1	46	0	2	5	0	7	2	11	20
58	11	19	6	13	29	11	3	7	7	4	9	20
59	30	2	15	22	1	19	27	7	10	9	12	20
60	4	68 *	38	107 *	107	16	18	16	19	3	39	20
61	3	29	34	53	56	14	5	2	11	3	20	20
62	42	18	9	44	49	15	13	6	6	5	19	20
63	7	2	22	22	77	1	7	22	14	7	20	20
64	4	10	16	33	12	14	26	14	5	3	12	20
Sr	13	14	15	21	25	11	9	6	6	4		1280
r	63	126	126	126	126	63	50	42	25	25		
NE	128	128	128	128	128	128	128	128	128	128		\square
L	59	56	69	84	112	49	41	25	26	17		

 $Sr: \ repeatability \ standard \ deviation \ of \ each \ laboratory \ \ limit: \ \textbf{C}f \ up \ down$

NL : number of measurements per laboratory

 $\ensuremath{\mathsf{L}}$: 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 $% \frac{1}{2}$ at 5 %

** : missing data

r : limit of repeatability, absolute difference betwen 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

<u>Table III:</u> Means of the replicates in 10 ³ cells / ml

Sample Lab	31	32	33	34	35	36	37	38	39	40
1	633	850	1148	1351	1612	479	324	220	113	61
2	621	808	1126	1296	1534	475	322	215	116	66
3	606	765	1002	1252	1500	442	303	208	96	63
4	609	823	1116	1318	1542	473	316	217	119	72
5	596	774	1086	1216	1500	445	217 *	306 *	121	74
6	594	785	1068	1252	1489	442	312	208	119	65
7	624	813	1112	1303	1555	487	324	222	123	75
8	617	809	1128	1293	1549	475	328	226	119	68
9	618	816	1121	1315	1562	470	311	218	122	71
10 11	647 649	827 824	1123 1141	1339 1320	1570 1567	496 476	343 329	227 226	116 124	77 75
12	630	804	1130	1320	1551	457	329	220	123	75 78
13	632	787	1112	1256	1479	482	334	216	123	75
14	641	855	1178	1365	1610	481	329	231	124	76
15	604	788	1084	1270	1515	465	316	215	131	73
16	613	791	1106	1275	1519	478	318	211	121	72
17	599	813	1072	1255	1480	472	302	204	118	74
18	511	720	976	1131	1378	398	277	175	96	54
19	618	796	1072	1277	1502	442	313	214	119	74
20	603	779	1087	1245	1523	455	307	206	119	72
21	627	836	1103	1264	1555	482	330	214	131	75
22	611	797	1122	1270	1512	457	307	209	117	68
23	615	784	1099	1291	1542	462	312	211	120	78
24	585	790	1072	1273	1523	455	320	210	113	69
25	595	788	1094	1289	1531	448	312	215	116	66
26	578	766	1048	1240	1481	434	300	208	114	69
27 28	596 601	816 807	1070 1107	1292 1277	1499 1532	449 487	315 330	206 225	119 121	70 75
29	586	772	1096	1249	1520	440	321	220	118	82
30	651	842	1180	1379	1645	494	334	221	123	76
31	638	855	1150	1351	1608	488	324	222	123	69
32	605	803	1103	1281	1499	455	314	211	116	69
33	526	763	998	1144	1304 *	437	284	191	99	60
34	538	654 *	883 *	1158	1439	388	277	184	105	65
35	631	830	1142	1339	1605	484	314	224	116	68
36	591	796	1097	1267	1526	459	320	214	122	74
37	600	811	1115	1300	1550	477	320	226	119	71
38	586	773	1066	1227	1497	458	306	205	119	72
39	579	787	1093	1272	1475	449	318	209	113	71
40	612	772	1085	1250	1517	453	312	215	121	65

M = mean per sample

REF. = reference values

SD = standard deviation per sample

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

Table IV : Outlier identification

Sample	31	32	33	34	35	36	37	38	39	40
Outliers				60						
Cochran	60			60						
Outlier Grubbs	60	34; 43 60	34; 60	43; 60	33; 43 60	60	5; 43 60	5; 60	43; 48 56; 60	48; 60
sr	13	12	15	19	22	11	9	5	6	4
SR	36	40	55	63	63	28	17	15	8	7
sr %	2%	1%	1%	1%	1%	2%	3%	2%	5%	6%
SR %	6%	5%	5%	5%	4%	6%	5%	7%	7%	10%

<u>Table III:</u> Means of the replicates in 10 ³ cells / ml

Sample Lab Code	31	32	33	34	35	36	37	38	39	40	1
41	620	795	1097	1283	1520	473	317	214	124	80	٦
42	611	787	1101	1290	1517	463	316	218	125	80	1
43	721	956 *	1277	1527 *	1808 ^k	536	376 *	258	156 *	91	1
44	635	836	1150	1361	1594	482	330	222	122	71	1
45	595	784	1099	1280	1514	443	316	209	112	68	1
46	647	846	1129	1319	1595	477	321	221	121	68	1
47	630	843	1128	1328	1573	482	325	220	115	70	1
48	663	893	1229	1419	1626	513	358	241	147 *	98	*
49	594	788	1096	1272	1530	465	310	210	120	71	1
50	607	820	1115	1309	1596	479	326	224	117	77	1
51	619	834	1114	1315	1562	492	317	221	120	75	1
52	554	764	1027	1172	1431	425	304	198	116	74	1
53	639	883	1212	1381	1651	500	345	227	125	70	1
54	656	859	1171	1364	1651	491	342	236	140	82	1
55	648	769	1149	1285	1573	452	296	187	123	54	1
56	560	698	1068	1276	1522	422	292	181	88 *	53	1
57	558	718	1021	1182	1429	393	287	174	114	64	1
58	613	817	1114	1306	1570	465	323	224	123	70	١
59	647	864	1198	1432	1694	492	338	228	131	76	١
60	95 *	157 *	260 *	256 *	388 ⊧	34 *	36 *	34 *	22 *	10	*
61	595	774	1081	1232	1465	450	309	196	115	66	١
62	642	811	1095	1306	1562	467	331	219	119	66	1
63	668	895	1238	1449	1685	520	349	240	128	73	1
64	612	843	1121	1308	1551	464	330	204	111	65	
М	612	805	1110	1289	1541	465	317	214	119	71	٦
REF.	613	806	1109	1290	1540	466	318	215	119	71	
SD	34	39	54	62	61	27	16	15	8	7]
AVT											1

M = mean per sample

REF, = reference values

SD = standard deviation per sample

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

AVT = Assign value traceable to the ERM BD001

Table IV : Outlier identification

Sample	31	32	33	34	35	36	37	38	39	40
Outliers	60			60						
Cochran	60			60						
Outlier		34; 43	24: 60	42. 60	33; 43		5; 43	F. CO	43; 48	40. 60
Grubbs	60	60	34; 60	43; 60	60	60	60	5; 60	56; 60	48; 60
sr	13	12	15	19	22	11	9	5	6	4
SR	36	40	55	63	63	28	17	15	8	7
sr %	2%	1%	1%	1%	1%	2%	3%	2%	5%	6%
SR %	6%	5%	5%	5%	4%	6%	5%	7%	7%	10%
SR Method										
for AVT	43	48	67	77	92	33	25	19	11	6
values										

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	+ 3%	+ 5%	+ 4%	+ 5%	+ 5%	+ 3%	+ 2%	+ 2%	- 5%	- 15%	+ 4%	4%	2,70
2	+ 1%	+ 0%	+ 2%	+ 0%	- 0%	+ 2%	+ 1%	- 0%	- 2%	- 7%	+ 0%	1%	1,38
3	- 1%	- 5%	- 10%	- 3%	- 3%	- 5%	- 5%	- 4%	- 20%	- 12%	- 5%	5%	3,32
4	- 1%	+ 2%	+ 1%	+ 2%	+ 0%	+ 1%	- 1%	+ 1%	+ 0%	+ 2%	+ 1%	2%	1,87
5	- 3%	- 4%	- 2%	- 6%	- 3%	- 5%	- 32%	+ 42%	+ 1%	+ 5%	- 3%	8%	1,33
6	- 3%	- 3%	- 4%	- 3%	- 3%	- 5%	- 2%	- 3%	- 0%	- 9%	- 3%	3%	3,96
7	+ 2%			+ 1%	+ 1%	+ 4%	+ 2%	+ 3%	+ 3%	+ 6%	+ 1%	1%	5,02
8	+ 1%	+ 0%	+ 2%	+ 0%	+ 1%	+ 2%	+ 3%	+ 5%	- 0%	- 5%	+ 1%	1%	3,15
9	+ 1%	+ 1%	+ 1%	+ 2%	+ 1%	+ 1%	- 2%	+ 1%	+ 3%	+ 0%	+ 1%	2%	2,44
10	+ 5%	+ 3%	+ 1%	+ 4%	+ 2%	+ 7%	+ 8%	+ 6%		+ 8%	+ 3%	2%	4,57
11	+ 6%	+ 2% - 0%	+ 3%	+ 2%	+ 2%	+ 2%	+ 3%	+ 5%		+ 5% + 9%	+ 3%	2%	4,85
12 13	+ 3% + 3%	- 2%	+ 2% + 0%	+ 2% - 3%	+ 1%	- 2% + 4%	+ 1% + 5%	+ 3% + 0%	l l	+ 9% + 6%	+ 1% - 1%	2% 4%	2,45 0,67
14	+ 4%	+ 6%	+ 6%	+ 6%	+ 5%	+ 3%	+ 3%	+ 7%	+ 4%	+ 7%	+ 5%	4%	3,78
15	- 2%	- 2%	- 2%	- 2%	- 2%	- 0%	- 1%	- 0%	+ 10%	+ 3%	- 1%	2%	2,20
16	- 0%	- 2%	- 0%	- 1%	- 1%	+ 3%	- 0%	- 2%	+ 2%	+ 1%	- 1%	1%	1,40
17	- 2%	+ 1%	- 3%	- 3%	- 4%	+ 1%	- 5%	- 5%	- 1%	+ 4%	- 2%	3%	2,28
18	- 17%	- 11%	- 12%	- 12%	- 11%	- 15%	- 13%	- 19%	- 20%	- 24%	- 13%	8%	4,83
19	+ 1%	- 1%	- 3%	- 1%	- 2%	- 5%	- 2%	- 1%	+ 0%	+ 4%	- 2%	2%	2,39
20	- 2%	- 3%	- 2%	- 4%	- 1%	- 2%	- 4%	- 4%	+ 0%	+ 2%	- 2%	2%	3,50
21	+ 2%	+ 4%	- 1%	- 2%	+ 1%	+ 4%	+ 4%	- 1%	+ 10%	+ 6%	+ 1%	2%	1,45
22	- 0%	- 1%	+ 1%	- 2%	- 2%	- 2%	- 4%	- 3%	- 2%	- 5%	- 1%	2%	2,24
23	+ 0%	- 3%	- 1%	+ 0%	+ 0%	- 1%	- 2%	- 2%	+ 1%	+ 10%	- 1%	1%	1,29
24	- 5%	- 2%	- 3%	- 1%	- 1%	- 2%	+ 0%	- 2%	- 5%	- 3%	- 2%	2%	3,62
25	- 3%	- 2%	- 1%	- 0%	- 1%	- 4%	- 2%	- 0%	- 2%	- 7%	- 1%	1%	4,11
26	- 6%	- 5%	- 6%	- 4%	- 4%	- 7%	- 6%	- 4%	- 5%	- 3%	- 5%	3%	4,45
27	- 3%	+ 1%	- 3%	+ 0%	- 3%	- 4%	- 1%	- 4%	- 0%	- 1%	- 2%	3%	2,15
28	- 2%	+ 0% - 4%	- 0%	- 1% - 3%	- 1%	+ 5% - 6%	+ 4% + 1%	+ 5%		+ 5%	+ 0%	2% 3%	0,42
29	- 4%	+ 4%	- 1% + 6%	+ 7%	- 1% + 7%	+ 6%	+ 1% + 5%	+ 2% + 3%	- 1% + 3%	+ 16% + 7%	- 2% + 6%		2,50
30 31	+ 6% + 4%	+ 6%			I	II .		ll l	ll l	1. , ,,	, ,,,	6%	3,45
	ll l			+ 5%	+ 4%	+ 5%	ll .	+ 3%	+ 3%	3 / 0	+ 4%	4%	3,51
32	- 1%	- 0%	- 1%	- 1%	- 3%	- 2%	- 1%	- 2%	- 3%	- 3%	- 1%	2%	2,54
33	- 14%	- 5%	- 10%	- 11%	- 15%	- 6%	- 11%	- 11%	- 17%	- 15%	- 11%	11%	3,25
34	- 12%	- 19%	- 20%	- 10%	- 7%	- 17%	- 13%	- 14%	- 12%	- 8%	- 13%	11%	3,92
35	+ 3%	+ 3%	+ 3%	+ 4%	+ 4%	+ 4%	- 1%	+ 4%	- 2%	- 4%	+ 3%	4%	2,80
36	- 4%	- 1%	- 1%	- 2%	- 1%	- 1%	+ 1%	- 1%	+ 2%	+ 4%	- 1%	1%	2,64
37	- 2%	+ 1%	+ 1%		+ 1%	+ 2%	+ 1%	+ 5%	- 0%	- 0%	+ 1%	1%	1,66
38	- 4%	- 4%	- 4%	- 5%	- 3%	- 2%	- 4%	- 5%		+ 2%	- 4%	3%	3,52
39	- 6%	- 2%	- 1%	- 1%	- 4%	- 4%	- 0%	- 3%		+ 0%	- 3%	3%	2,98
40	- 0%	- 4%	- 2%	- 3%	- 2%	- 3%	- 2%	- 0%	+ 2%	- 8%	- 2%	2%	3,10

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

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

Sample Lab code	31	32	33	34	35	36	37	38	39	40	d	Sd _{lab}	t
41	+ 1%	- 1%	- 1%	- 1%	- 1%	+ 1%	- 0%	- 1%	+ 4%	+ 12%	- 0%	1%	0,80
42	- 0%	- 2%	- 1%	- 0%	- 2%	- 1%	- 1%	+ 1%	+ 5%	+ 13%	- 1%	2%	1,22
43	+ 18%	+ 19%	+ 15%	+ 18%	+ 17%	+ 15%	+ 18%	+ 20%	+ 31%	+ 28%	+ 18%	13%	4,21
44	+ 4%	+ 4%	+ 4%	+ 5%	+ 4%	+ 4%	+ 4%	+ 3%	+ 2%	- 0%	+ 4%	4%	3,44
45	- 3%	- 3%	- 1%	- 1%	- 2%	- 5%	- 1%	- 3%	- 6%	- 5%	- 2%	1%	4,58
46	+ 6%	+ 5%	+ 2%	+ 2%	+ 4%	+ 2%	+ 1%	+ 3%	+ 1%	- 4%	+ 3%	3%	3,23
47	+ 3%	+ 5%	+ 2%	+ 3%	+ 2%	+ 4%	+ 2%	+ 2%	- 4%	- 2%	+ 3%	2%	3,37
48	+ 8%	+ 11%	+ 11%	+ 10%	+ 6%	+ 10%	+ 13%	+ 12%	+ 23%	+ 38%	+ 10%	6%	5,20
49	- 3%	- 2%	- 1%	- 1%	- 1%	- 0%	- 3%	- 2%	+ 0%	+ 0%	- 1%	1%	3,76
50	- 1%	+ 2%	+ 1%	+ 1%	+ 4%	+ 3%	+ 3%	+ 4%	- 2%	+ 8%	+ 2%	3%	2,25
51	+ 1%	+ 3%	+ 0%	+ 2%	+ 1%	+ 6%	- 0%	+ 3%	+ 1%	+ 5%	+ 2%	2%	3,30
52	- 10%	- 5%	- 7%	- 9%	- 7%	- 9%	- 5%	- 8%	- 2%	+ 5%	- 7%	7%	3,54
53	+ 4%	+ 10%	+ 9%	+ 7%	+ 7%	+ 7%	+ 8%	+ 5%	+ 5%	- 2%	+ 7%	7%	3,59
54	+ 7%	+ 7%	+ 6%	+ 6%	+ 7%	+ 5%	+ 8%	+ 9%	+ 18%	+ 15%	+ 7%	5%	4,49
55	+ 6%	- 5%	+ 4%	- 0%	+ 2%	- 3%	- 7%	- 13%	+ 3%	- 24%	- 0%	4%	0,11
56	- 9%	- 13%	- 4%	- 1%	- 1%	- 9%	- 8%	- 16%	- 26%	- 25%	- 6%	4%	4,46
57	- 9%	- 11%	- 8%	- 8%	- 7%	- 16%	- 10%	- 19%	- 5%	- 10%	- 9%	6%	4,94
58	- 0%	+ 1%	+ 0%	+ 1%	+ 2%	- 0%	+ 1%	+ 4%	+ 3%	- 1%	+ 1%	1%	2,52
59	+ 6%	+ 7%	+ 8%	+ 11%	+ 10%	+ 6%	+ 6%	+ 6%	+ 10%	+ 7%	+ 8%	8%	3,17
60	- 85%	- 81%	- 77%	- 80%	- 75%	- 93%	- 89%	- 84%	- 82%	- 87%	- 80%	59%	4,28
61	- 3%	- 4%	- 2%	- 5%	- 5%	- 3%	- 3%	- 9%	- 4%	- 7%	- 4%	4%	3,62
62	+ 5%	+ 1%	- 1%	+ 1%	+ 1%	+ 0%	+ 4%	+ 2%	+ 0%	- 7%	+ 1%	2%	1,71
63	+ 9%	+ 11%	+ 12%	+ 12%	+ 9%	+ 12%	+ 10%	+ 12%	+ 8%	+ 2%	+ 11%	9%	3,82
64	- 0%	+ 5%	+ 1%	+ 1%	+ 1%	- 0%	+ 4%	- 5%	- 7%	- 9%	+ 1%	2%	1,32
d	- 0%	- 0%	+ 0%	- 0%	+ 0%	- 0%	- 0%	- 0%	- 0%	- 0%	- 1%	12%	
Sd	6%	5%	5%	5%	4%	6%	5%	7%	6%	9%			

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

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

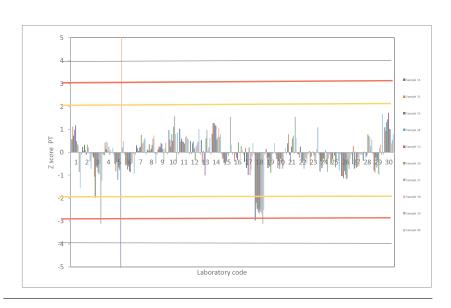
<u>Table VI:</u> Zscore of the different laboratories for each sample.

ZS calculated on the PT standard deviation

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

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

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



<u>Table VI:</u> Zscore of the different laboratories for each sample.

ZS calculated on the PT standard deviation

Sample Lab	31	32	33	34	35	36	37	38	39	40
31	+0,71	+1,27	+0,76	+0,99	+1,12	+0,82	+0,38	+0,43	+0,54	-0,27
32	-0,25	-0,07	-0,11	-0,15	-0,67	-0,41	-0,28	-0,27	-0,45	-0,34
33	-2,55	-1,11	-2,05	-2,35	-3,88	-1,06	-2,16	-1,65	-2,72	-1,62
34	-2,20	-3,92	-4,18	-2,14	-1,67	-2,86	-2,60	-2,09	-1,85	-0,87
35	+0,52	+0,63	+0,61	+0,79	+1,06	+0,66	-0,28	+0,56	-0,39	-0,42
36	-0,64	-0,26	-0,22	-0,37	-0,23	-0,25	+0,13	-0,07	+0,34	+0,41
37	-0,40	+0,13	+0,11	+0,16	+0,16	+0,40	+0,13	+0,70	-0,05	-0,04
38	-0,80	-0,84	-0,80	-1,02	-0,70	-0,30	-0,75	-0,68	-0,05	+0,18
39	-1,01	-0,49	-0,30	-0,29	-1,07	-0,61	-0,03	-0,44	-0,85	+0,03
40	-0,05	-0,87	-0,44	-0,64	-0,38	-0,49	-0,37	-0,01	+0,28	-0,87
41	+0,20	-0,29	-0,22	-0,11	-0,33	+0,25	-0,06	-0,07	+0,61	+1,31
42	-0,08	-0,49	-0,15	-0,00	-0,38	-0,10	-0,12	+0,20	+0,81	+1,38
43	+3,13	+3,87	+3,12	+3,82	+4,41	+2,59	+3,64	+2,85	+4,94	+2,96
44	+0,64	+0,78	+0,76	+1,14	+0,89	+0,60	+0,72	+0,46	+0,34	-0,04
45	-0,54	-0,56	-0,18	-0,17	-0,43	-0,83	-0,12	-0,41	-0,92	-0,49
46	+0,99	+1,03	+0,38	+0,47	+0,91	+0,42	+0,16	+0,40	+0,21	-0,42
47	+0,49	+0,96	+0,35	+0,62	+0,55	+0,60	+0,41	+0,30	-0,59	-0,19
48	+1,45	+2,25	+2,22	+2,09	+1,42	+1,72	+2,51	+1,74	+3,67	+4,00
49	-0,56	-0,47	-0,24	-0,30	-0,16	-0,04	-0,53	-0,34	+0,08	+0,03
50	-0,18	+0,37	+0,11	+0,30	+0,92	+0,47	+0,51	+0,60	-0,32	+0,86
51	+0,17	+0,72	+0,10	+0,40	+0,36	+0,97	-0,09	+0,36	+0,15	+0,56
52	-1,72	-1,07	-1,51	-1,91	-1,79	-1,52	-0,90	-1,15	-0,39	+0,48
53	+0,74	+1,98	+1,91	+1,46	+1,82	+1,24	+1,66	+0,77	+0,81	-0,19
54	+1,24	+1,37	+1,15	+1,20	+1,83	+0,91	+1,51	+1,37	+2,81	+1,61
55	+1,02	-0,94	+0,75	-0,08	+0,55	-0,50	-1,37	-1,88	+0,54	-2,52
56	-1,55	-2,77	-0.75	-0,22	-0,29	-1.61	-1,63	-2,29	-4,12	-2,67
57	-1,60	-2,26	-1,63	-1,74	-1,82	-2,67	-1,97	-2,76	-0,72	-1,02
58	-0,02	+0,28	+0,10	+0,25	+0,49	-0,04	+0,29	+0,56	+0,48	-0,12
59	+0,99	+1,50	+1,64	+2,30	+2,53	+0,95	+1,23	+0,83	+1,61	+0,71
60	-15,09	-16,70	-15,71	-16,70	-18,94	-15,88	-17,67	-12,15	-12,97	-9,19
61	-0,54	-0,83	-0,51	-0,94	-1,23	-0,58	-0,59	-1,28	-0,59	-0,79
62	+0,84	+0,14	-0,26	+0,26	+0,36	+0,03	+0,79	+0,26	+0,01	-0,79
63	+1,59	+2,30	+2,39	+2,57	+2,38	+1,98	+1,91	+1,67	+1,21	+0,26
64	-0,03	+0,96	+0,23	+0,29	+0,18	-0,06	+0,76	-0,74	-1,12	-0,94

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

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

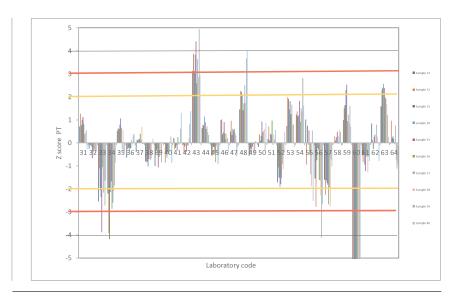


Table VII: Zscore FIX of the different laboratories for each sample,
ZS calculated on Assign value(REF) and standard deviation of reproducibility

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

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

Figure 3:

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

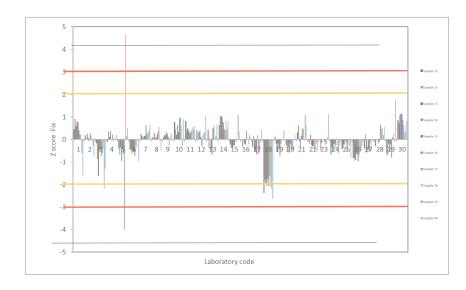


Table VII: Zscore FIX of the different laboratories for each sample.

ZS calculated on Assign value(REF) and standard deviation of reproducibility of the method

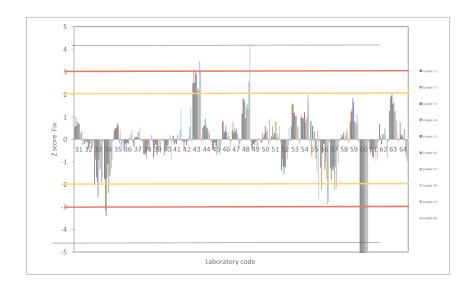
			netnod							
Sample	31	32	33	34	35	36	37	38	39	40
31	+0,57	+1,02	+0,62	+0,79	+0,74	+0,68	+0,24	+0,33	+0,38	-0,28
32	-0,20	-0,05	-0,09	-0,12	-0,44	-0,34	-0,17	-0,21	-0,32	-0,36
33	-2,04	-0,89	-1,66	-1,88	-2,56	-0,88	-1,35	-1,27	-1,91	-1,69
34	-1,76	-3,15	-3,39	-1,71	-1,10	-2,38	-1,63	-1,61	-1,30	-0,91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

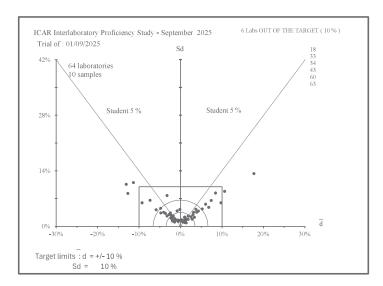
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR: Cf page 7 and 8/13

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

Figure 3:

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





 $\underline{ \mbox{Figure 1}}: \quad \mbox{ ACCURACY - Evaluation of the individual performances (to see table I).}$



ICAR PROFICIENCY TESTING SCHEME

September 2025

Raw Milk

Enumeration of SOMATIC CELLS

Sending date of statistical treatment : 25th September 2025

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

ICAR Staff Silvia Orlandini pt@icar.org silvia@icar.org

Proficiency test accreditated ISO 17043





Table I: Ranking of the laboratories in %

Nb	%	Ν°	d	Sd	D	Method	
1	2	9	- 0%	1%	1%	В	
2	4	4	+ 0%	1%	1%	В	
3	6	53	+ 0%	1%	1%	В	
4	8	29	+ 1%	1%	1%	В	
5	10	51	- 1%	1%	1%	В	
6	12	11	+ 1%	0%	1%	В	
7	14	19	+ 1%	1%	1%	В	
8	16	18	- 1%	1%	1%	В	
9	18	42	- 0%	2%	2%	В	
10	20	57	- 1%	2%	2%	В	
11	22	64	+ 2%	0%	2%	В	
12	24	38	- 2%	0%	2%	В	
13	25	50	+ 2%	0%	2%	В	
14	27	55	- 2%	1%	2%	В	
15	29	20	- 1%	2%	2%	В	
16	31	35	+ 1%	2%	2%	В	
17	33	40	- 2%	2%	2%	В	
18	35	22	- 0%	2%	3%	В	
19	37	37	- 2%	2%	3%	В	
20	39	7	- 1%	3%	3%	В	
21	41	41	- 0%	3%	3%	В	
22	43	44	- 3%	2%	3%	В	
23	45	17	- 0%	3%	3%	В	
24	47	32	- 2%	3%	3%	В	
25	49	21	+ 1%	3%	3%	В	
26	51	1	+ 3%	2%	3%	В	
27	53	14	+ 2%	3%	4%	В	
28	55	56	+ 3%	2%	4%	В	
29	57	12	+ 3%	3%	4%	В	
30	59	45	- 2%	4%	4%	В	

Nb	%	Ν°	d	Sd	D	Method
31	61	34	- 5%	0%	5%	В
32	63	62	- 4%	2%	5%	В
33	65	8	- 3%	4%	5%	В
34	67	63	+ 5%	0%	5%	В
35	69	49	+ 2%	5%	5%	В
36	71	48	+ 5%	3%	6%	В
37	73	31	+ 2%	5%	6%	В
38	75	36	- 3%	5%	6%	В
39	76	15	- 3%	6%	7%	В
40	78	33	- 6%	2%	7%	B B
41 42	80 82	54 10	+ 4%	6% 2%	7% 7%	B
43	84	61	- 8%	6%	10%	B
44	86	58	- 10%	4%	11%	В
45	88	59	+ 11%	2%	11%	B
46	90	2	- 8%	9%	12%	В
47	92	43	+ 11%	5%	12%	В
48	94	30	+ 9%	9%	13%	В
49	96	60	+ 13%	9%	16%	С
50	98	13	- 4%	27%	27%	A
51	100	52	- 6%	96%	97%	A
N.C.		3				В
N.C.		5				В
N.C.		6				В
N.C.		16				В
N.C.		23				В
N.C.		24				В
N.C.		25				В
N.C.		26				В
N.C.		27				В
N.C.		28				В
N.C.		39				В
N.C.		46				В
N.C.		47				В

(NC : OUT of RANKING because of insufficient data 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 :

A ISO 13366-1 IDF 148-1 B ISO 13366-2 IDF 148-2

C Image Cytometry

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

REE : Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 48 laboratories using reference metod ISO 13366-1 | IDF 148-1 and alternative method ISO 13366-2 | IDF 148-2 after outlier discarging 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 %) 11 2% Reproducibility standard deviation of this ICAR proficiency test (after Cochran and Grubbs elimination at 5 %) SR_{PT}

⁽Nb : laboratory rank; % : relative rank)

⁽N° : laboratory identification number)

<u>Table II:</u> REPEATABILITY - Absolute difference between replicates in 10 ³ cells / ml

Sample Lab	А	В	Sr	NL
1	4	1	2	4
2	8	5	5	4
3	**	**	-	
4	6	10	6	4
5	**	**	•	
6	**	**		
7	3	12	6	4
8	0	20	10	4
9	12	0	6	4
10	43	12	22	4
11	9	11	7	4
12	38	43	29	4
13	10	1	5	4
14	13	7	7	4
15	19	38	21	4
16	**	**		
17	17	26	16	4
18	1	4	2	4
19	5	3	3	4
20	4	1	2	4
21	6	9	5	4
22	10	10	7	4
23	**	**		
24	**	**		
25	**	**		
26	**	**		
27	**	**		
28	**	**		
29	14	16	11	4
30	18	24	15	4

Sample Lab		_		
code	Α	В	Sr	NL
31	2	7	4	4
32	21	18	14	4
33	2	2	1	4
34	15	21	13	4
35	3	9	5	4
36	14	12	9	4
37	9	5	5	4
38	4	0	2	4
39	**	**		
40	25	11	14	4
41	24	8	13	4
42	21	4	11	4
43	3	0	2	4
44	22	3	11	4
45	2	2	1	4
46	**	**		
47				
48	5	5	4	4
49	6	22	11	4
50	10	6	6	4
51	19	6	10	4
52	1	31	16	4
53	46	1	23	4
54	12	17	10	4
55	0	0	0	4
56	0	0	0	4
57	21	4	11	4
58	5	9	5	4
59	13	16	10	4
60	85 *	13	43	4
61	37	20	21	4
62	2	16	8	4
63	1	2	1	4
64	2	33	17	4
Sr	14	11		204
r	126	50		
NE	102	102		
L	51	47		

 ${\tt Sr: repeatability \, standard \, deviation \, of \, each \, laboratory \, \, limit: \, \textbf{C}f \, \, up \, down}$

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 $% \frac{1}{2}$ at 5 %

** : missing data

r: limit of repeatability, absolute difference betwen two replicates according ISO 13366-2 / IDF 148-2: Cf up down and the control of the c

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	А	В
1	885	402
2	773	381
3		
4	857	396
5		
6		200
7	844	398
8	825	394
9	854	392
10 11	911 869	428 397
12	890	398
13	715 *	483 *
14	887	389
15	816	401
16	010	101
17	871	375
18	857	380
19	860	401
20	845	396
21	877	381
22	868	378
23		
24		
25		
26		
27		
28	050	200
29	859	399
30	954	406

Sample Lab	А	В
31	896	380
32	837	392
33	809	363
34	833	362
35	876	390
36	817	393
37	841	390
38	846	380
39		
40	857	374
41	845	401
42	852	399
43	948	438
44	836	383
45	832	399
46 47		
48	878	436
49	l	
	891	380
50	874	402
51	861	383
52	393	* 778 *
53	865	389
54	911	394
55	843	386
56	890	400
57	847	392
58 59	779 940	347 450
60	904	513 *
61	786	369
62	825	374
63	893	421
64	873	402
M	861	393
REF.	859	393
SD SD	38	19
	38	1 19
AVT		

M = mean per sample

REF. = reference values

SD = standard deviation per sample

*: discarded data using the test of Grubbs 5 %

AVT = Assign value traceable to the ERM BD001

REF: Assigned values are robust average values per sample according to algorithm A of standard ISO 13528, of 48 laboratories using the reference method ISO 13366 | IDF 148-1 and alternative method ISO 13366-2 | IDF 148-2, after outlier discarging using Grubbs test at 5% risk level

Table IV: Outlier identification

Sample	Α		В	
Outliers	60			
Cochran	00			
Outlier	13; 52		13; 52	
Grubbs	13, 32		60	
sr	12		10	
SR	39		20	
sr %	1%		3%	
SR %	5%		5%	

SR Method			Г
for AVT	52	31	
values			

<u>Table V :</u> ACCURACY - differences (laboratory - reference) in %

Sample Lab code	А	В	d	Sd _{lab}	t
1	+ 3%	+ 3%	+ 3%	2%	2,32
2	- 10%	- 3%	- 8%	9%	1,29
3					
4	- 0%	+ 1%	+ 0%	1%	0,33
5					
6	201	201	40/	201	0.40
7	- 2%	+ 2%	- 1%	3%	0,40
8 9	- 4%	+ 1%	- 3%	4%	0,85
10	- 1% + 6%		- 0% + 7%	1% 2%	0,76 6,09
11	+ 6% + 1%	+ 9% + 1%	+ 7% + 1%	0%	3,65
12	+ 4%	+ 2%	+ 3%	3%	1,51
13	- 17%	+ 23%	- 4%	27%	0,23
14	+ 3%	- 1%	+ 2%	3%	0,81
15	- 5%	+ 2%	- 3%	6%	0,64
16					'
17	+ 1%	- 4%	- 0%	3%	0,19
18	- 0%	- 3%	- 1%	1%	1,68
19	+ 0%	+ 2%	+ 1%	1%	1,03
20	- 2%	+ 1%	- 1%	2%	0,55
21	+ 2%	- 3%	+ 1%	3%	0,24
22	+ 1%	- 3%	- 0%	2%	0,21
23					
24					
25					
26					
27					
28	- 0%	+ 2%	+ 1%	1%	0,91
29 30	+ 11%	+ 4%	+ 1%	9%	
30	H 11%	 + 4%	+ 9%	9%	1,37

Sample Lab	А	В	d	Sd _{lab}	t
31	+ 4%	- 3%	+ 2%	5%	0,51
32	- 3%	+ 0%	- 2%	3%	0,94
33	- 6%	- 7%	- 6%	2%	3,56
34	- 3%	- 8%	- 5%	0%	19,29
35	+ 2%	- 0%	+ 1%	2%	0,80
36	- 5%	+ 0%	- 3%	5%	0,92
37	- 2%	- 0%	- 2%	2%	1,21
38	- 2%	- 3%	- 2%	0%	11,93
39					
40	- 0%	- 5%	- 2%	2%	1,38
41	- 2%	+ 2%	- 0%	3%	0,19
42 43	- 1% + 10%	+ 2% + 12%	- 0% + 11%	2% 5%	0,01 3,25
44	- 3%	- 2%	- 3%	2%	2,21
45	- 3%	+ 2%	- 2%	4%	0,56
46	3 /0	270	- 270	770	0,50
47					
48	+ 2%	+ 11%	+ 5%	3%	2,39
49	+ 4%	- 3%	+ 2%	5%	0,47
50	+ 2%	+ 3%	+ 2%	0%	6,45
51	+ 0%	- 2%	- 1%	1%	0,76
52	- 54%	+ 99%	- 6%	96%	0,09
53	+ 1%	- 1%	+ 0%	1%	0,34
54	+ 6%	+ 1%	+ 4%	6%	1,09
55	- 2%	- 1%	- 2%	1%	1,96
56	+ 4%	+ 2%	+ 3%	2%	1,79
57	- 1%	+ 0%	- 1%	2%	0,89
58 59	- 9% + 9%	- 11% + 15%	- 10% + 11%	4% 2%	3,48 6,48
60	+ 5%	+ 31%	+ 13%	9%	2,15
61	- 9%	- 6%	- 8%	6%	1,86
62	- 4%	- 4%	- 4%	2%	3,03
63	+ 4%	+ 8%	+ 5%	0%	18,30
64	+ 2%	+ 3%	+ 2%	0%	6,95
d	+ 0%	+ 0%	- 1%	13%	0,55
Sd	4%	5%			

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:

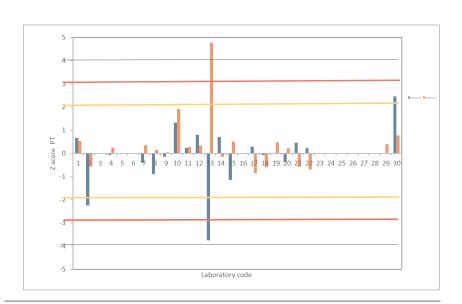
Level SCC *10 ³ /ml	Sr %	r	SR %	R
150	6	25	9	38
200	5	42	8	67
450	4	50	7	88
750	3	63	6	126
1500	3	126	6	252

<u>Table VI:</u> Zscore of the different laboratories for each sample, ZS calculated on the PT standard deviation

Sample Lab		
code	A	В
1	+0,67	+0,53
2	-2,25	-0,56
3		
4	-0,06	+0,25
5		
6		
7	-0,41	+0,35
8	-0,89	+0,14
9	-0,14	+0,04
10	+1,33	+1,92
11	+0,24	+0,27
12	+0,80	+0,33
13	-3,75	+4,78
14	+0,71	-0,15
15	-1,14	+0,51
16		
17	+0,29	-0,85
18	-0,07	-0,59
19	+0,00	+0,48
20	-0,37	+0,22
21	+0,46	-0,56
22	+0,22	-0,70
23		
24		
25		
26		
27		
28		
29	-0,01	+0,40
30	+2,46	+0,77

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

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

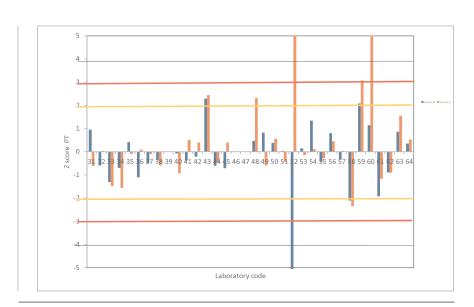


<u>Table VI:</u> Zscore of the different laboratories for each sample, ZS calculated on the PT standard deviation

Sample Lab	A	В
code	- ' '	
31	+0,95	-0,62
32	-0,59	+0,04
33	-1,31	-1,48
34	-0,70	-1,56
35	+0,42	-0,09
36	-1,10	+0,09
37	-0,49	-0,09
38	-0,35	-0,59
39		
40	-0,07	-0,93
41	-0,37	+0,51
42	-0,20	+0,40
43	+2,29	+2,45
44	-0,61	-0,46
45	-0,71	+0,40
46		
47		
48	+0,47	+2,31
49	+0,82	-0,59
50	+0,38	+0,56
51	+0,03	-0,43
52	-12,14	+20,22
53	+0,15	-0,15
54	+1,34	+0,12
55	-0,43	-0,28
56	+0,80	+0,46
57	-0,33	+0,04
58	-2,10	-2,34
59	+2,08	+3,07
60	+1,15	+6,35
61	-1,92	-1,17
62	-0,89	-0,90
63	+0,86	+1,56
64	+0,36	+0,53

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

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



<u>Table VII:</u> Zscore FIX of the different laboratories for each sample. ZS calculated on Assign value(REF) and standard deviation of reproducibility

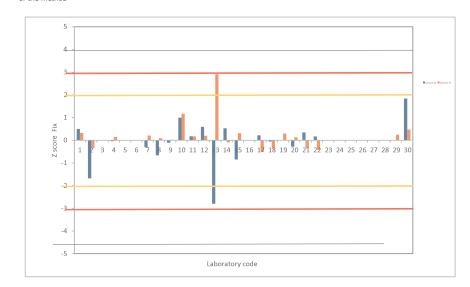
Cample		1
Sample and code	A	В
1	+0,50	+0,33
2	-1,67	-0,34
3		
4	-0,05	+0,15
5		
6		
7	-0,31	+0,21
8	-0,67	+0,09
9	-0,10	+0,02
10	+0,99	+1,17
11	+0,18	+0,17
12	+0,59	+0,20
13	-2,80	+2,91
14	+0,53	-0,09
15	-0,85	+0,31
16		
17	+0,22	-0,52
18	-0,06	-0,36
19	+0,00	+0,29
20	-0,28	+0,13
21	+0,34	-0,34
22	+0,17	-0,42
23		
24		
25		
26		
27		
28		
29	-0,01	+0,25
30	+1,84	+0,47

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

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

Figure 3:

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



<u>Table VII:</u> Zscore FIX of the different laboratories for each sample. ZS calculated on Assign value (REF) and standard deviation of reproducibility

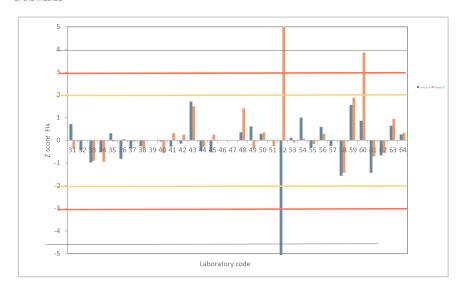
Sample and	Α	В
31	+0,71	-0,38
32	-0,44	+0,02
33	-0,98	-0,90
34	-0,52	-0,95
35	+0,31	-0,06
36	-0,82	+0,05
37	-0,37	-0,06
38	-0,26	-0,36
39		
40	-0,06	-0,57
41	-0,28	+0,31
42	-0,15	+0,25
43	+1,71	+1,49
44	-0,45	-0,28
45	-0,53	+0,25
46 47		
	10.25	11.41
48 49	+0,35 +0,61	+1,41
50	+0,28	+0,34
51	+0,02	-0,26
52	-9,05	+12,34
53	+0,11	-0,09
54	+1,00	+0,07
55	-0,32	-0,17
56	+0,59	+0,28
57	-0,25	+0,02
58	-1,57	-1,43
59	+1,55	+1,88
60	+0,86	+3,87
61	-1,43	-0,71
62 63	-0,67 +0,64	-0,55 +0,95
64	1 1	l '
04	+0,26	+0,33

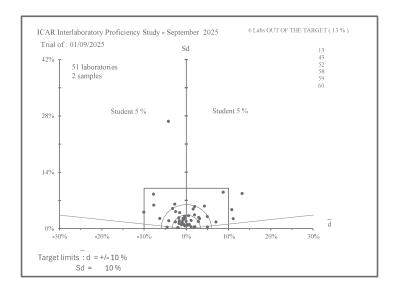
This table will allows to compare your ZSCORE from one PT to an other because the standard deviation has always the value of SR of the method SR : Cf page 7 and $8\,/13$

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

Figure 3:

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





 $\underline{ \mbox{Figure 1}}: \quad \mbox{ACCURACY - Evaluation of the individual performances (to see table I).}$