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of the International Dairy Federation

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Collaborative studies organized to include sheep and goat milk in the scope of joint standard
ISO 5764|IDF 108: 2002 Milk –
Determination of freezing point –
Thermistor cryoscope method
(Reference method)



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Collaborative studies organized to include sheep and goat milk in the scope of joint standard ISO 5764|IDF 108: 2002 Milk – Determination of freezing point – Thermistor cryoscope method (Reference method)

Foreword

This issue of the Bulletin presents the collaborative studies organized to include sheep and goat milk in the scope of joint standard ISO 5764|IDF 108: 2002 Milk – Determination of freezing point – Thermistor cryoscope method (Reference method).

As a work item in the IDF/ISO Joint Action Team (JAT) on “Water” under the IDF Standing Committee on Main components of milk, these collaborative studies were organized in cooperation between Associazione Italiana Allevatori (IT) and the Cyprus Milk Industry Organisation (CY), and have yielded precision figures for sheep and goat milk analysis which are now usable by the dairy sector.

IDF is most grateful to the members of the project group (Silvia Orlandini, Ugo Paggi, George Psathas, Luca Lattanzi, Harrie van den Bijgaart, Olivier Leray, Jan Floor, Jaap Evers, Jackie Page and all co-workers) for their efforts in producing this report, as well as to the JAT, the institutions that organized the collaborative studies and all laboratories participating.

Christian Robert
Director General
August 2007

1. Collaborative study on the freezing point of Goat Milk

S. Orlandini¹, L. Lattanzi¹, A. Carducci¹, G. Psathas², U. Paggi¹

Abstract

Precision was determined by a collaborative study conducted in 2004 in which 12 laboratories participated. Six goat milk samples with a freezing point level varying from $-0,597^{\circ}\text{C}$ to $-0,542^{\circ}\text{C}$ were analysed in blind duplicates. Of the 144 series of results received, 48 were eliminated because not in conformity with Standard ISO 5764|IDF 108: 2002. Hence 96 values were considered valid.

The statistical elaboration gave a repeatability value $r=0,004^{\circ}\text{C}$ and a reproducibility value $R=0,008^{\circ}\text{C}$.

1.1. Introduction

A first collaborative study conducted in March 2003 lead to unsatisfactory results. Hence in February 2004 a new collaborative study on goat milk was organized by Co-Project Leader Ugo Paggi.

1.2. Lay-out of the study

1.2.1. Organization and participating laboratories

During Autumn 2003, 36 international laboratories were contacted by C.M.I.O. inviting them to take part in the second Collaborative Study on goat milk.

The final list of participating laboratories list is (in alphabetical order):

AIA - LSL	Italy
Advanced Instruments	USA
AFSSA	France
Cecalait	France
CLO-DVK	Belgium
C.M.I.O.	Cyprus
Danish Vet. And Food Administration	Denmark
Istituto Zootecnico Caseario Sassari	Italy
Lab.Agroalimentario de Santander	Spain
MLUA	Germany
Netherlands Milk Control Station	Netherlands
Univ. of Ljubljana Biotech Fac. Zootech.	Slovenia

1.2.2. Samples

The samples were prepared and dispatched by AIA-LSL on the 24th of February by international express courier.

Sixteen samples were prepared in the following way:

- Six samples, in blind duplicates, of goat raw milk added with Bronopol 0,02%, ranging from $-0,597^{\circ}\text{C}$ to $-0,542^{\circ}\text{C}$;
- Two samples, in blind duplicates, of cow raw milk added with Bronopol 0,02%, with a freezing point of $-0,526^{\circ}\text{C}$ and $-0,530^{\circ}\text{C}$. The choice of inserting cow milk samples was taken

¹ A.I.A. Laboratorio Standard Latte, Maccaresse - Rome - Italy

² Cyprus Milk Industry Organisation, Nicosia - Cyprus

in order to evaluate the performance of laboratories.

All the samples were identified by numbers from 1 to 16, without differentiation between cow and goat milk samples.

Furthermore all the labs received 3 solutions to be used as pilot controls, and 3 standard solutions for instrument calibration.

Together with the samples, explanatory papers were sent about Package content, Instructions and Questionnaire. All the packages were delivered in about 24 hours (Advanced did not receive the package due to custom duty problems) at the average temperature of 3°C (each package included a water vial for measuring temperature upon package reception).

The quality of samples before the shipping was monitored by Total Bacterial Count and Somatic Cell Count by fluoro-opto-electronic instruments flow cytometry technique. See [Table 1](#).

The stability of the samples was checked by sending one package back and forth to our laboratory by air-mail, in order to simulate the delivery of samples. The package was received 48 hours after expedition, and the temperature on the water vial was 4,5°C.

These samples were processed through Bacterial Count and IR Instrument, and no significant differences were found after 48 hours.

1.2.3. Method

The method used was ISO 5764|IDF 108: 2002 (Thermistor cryoscope method).

1.2.4. Instructions

The following instructions were enclosed in the package.

Application of the method ISO 5764|IDF 108:2002 on samples n° 1- 16 (glass vials with black caps).

Prepare instrument by leaving power on at least for 1 hour for circuit stabilization.

The instrument should be connected on UPS power supplier.

Check position of thermistor probe and stirrer to be in accordance with the standard IDF 108:2002 (paragraph 6.1.2 and 6.1.3).

Check amplitude of stirring wire to be in accordance with the standard ISO 5764|IDF 108:2002.

Sample quantity 2,5±0,1ml.

Freezing pulse at -3,0 m°C.

Measurement cut-off when temperature rise has been maximal 0,5 m°C over last 20s (resolution better than 1 m°C) or the read out has been constant during last 20 s (resolution not better than 1 m°C).

Bring all standard solutions to room temperature.

Calibrate instrument with the standard solutions -0,408°C and -0,600°C according to instrument manufacturer instructions. Verify the instrument calibration with the standard solution -0,512°C. The results should not differ by more than 2 m°C from -0,512°C. If the difference is higher, recalibrate the instrument, otherwise do not make adjustments on the instrument.

Samples preparation: Shortly before determinations, warm the samples to 40°C in a water bath. Homogenize samples by inverting gently 6-8 times. Then cool to 20°C and homogenize again by inverting gently 6-8 times. Warning : avoid the incorporation of air. The samples are ready for testing: pipette 2,5±0,1ml.

Analyze the pilot sample and record the result (we suggest to change the pilot sample vial in the next steps of analysis).

Analyze once 8 samples in their respective order (1 to 8) and repeat analysis of the pilot sample (we suggest to use a brand new pilot sample).

Analyze once the rest of the 8 samples (9 to 16) in their respective order and repeat analysis of the pilot sample (we suggest to use a brand new pilot sample).

Record the results to the fourth digit without rounding (or third digit depending on instrument's resolution) on the paper form and then in the excel file you received by email. Record any comments or observation you may have on the appropriate box.

Please complete the questionnaire accurately.

Return the excel file via email to dati.isl@aia.it

1.2.5. Application Questionnaire

The following questionnaire was sent with the samples. The results of this questionnaire have been utilized for laboratory scrutinization.

SAMPLES RECEPTION

1. Days between delivery and reception:
2. Samples temperature:.....
3. Samples condition (any leakage etc):
Storage of samples (if not analyzed immediately):
Temperature:..... Hours:.....

SAMPLES PREPARATION

1. Upon unscrewing the caps did you observe any fat stains on the samples surface?
YES NO
If yes, please state the identification number of samples(1-16):.....
Please state the temperature of warming up for homogenization:.....
2. Please state the temperature of samples drawing:.....
3. During the homogenization process, did you observe any kind of lypolysis in samples?
YES NO
If yes please state the identification number of samples (1-16).....

SAMPLES VOLUME

1. Please state the samples volume used:

INSTRUMENT SET UP

1. Please state the instrument model:.....
2. Please state the Freezing temperature prior to stirring:.....
3. Please state the plateau used:
 - a. Fixed plateau: - Time:
 - b. Seeking plateau: - Time:

INSTRUMENT CALIBRATION

1. Did you face any problem on calibration process with solutions received?
YES NO
If yes, please describe:
2. What were the duplicate values you received on verifying the calibration with the standard solution $-0,512^{\circ}\text{C}$: 1..... 2.....

SAMPLES MEASURING

1. Were all samples measured continuously?
YES NO
2. Did you have any preefreeze on any sample?
YES NO
If yes, please state the identification number of samples (1-16):.....

1.3. Returning of results

The results were registered on an excel file provided via e-mail to all the participants and sent back to AIA-LSL.

1.4. Analysis of results

The 16 samples results were paired in order to have 6 samples of goat milk (A, B, C, D, E, F) and 2 samples of cow milk (G and H). See [Table 1](#).

The answers to the questionnaire were collected by CMIO. Once elaborated, the results obtained were utilized for laboratory and/or sample scrutinization.

As a result, 3 labs were rejected due to lack of conformity with Standard ISO 5764|IDF 108: 2002; Laboratory n°2 commented that samples 1 and 16 were in bad condition, therefore sample G for lab 2 was rejected from the elaboration.

Statistical elaboration was conducted according to Standard ISO 5725-2:1994.

- Absolute difference between duplicates; the Cochran's Test was applied to target outlier values;
- Mean of duplicates; the Grubbs' Test sample was applied and then Zlab (one Zscore for each laboratory) was calculated and reported on a graphic.
- Difference from the Reference value, then plotted on a graphic; the reference values were calculated making the median of the values after outlier elimination. See [Table 3](#).

1.5. Results and conclusions

The unsuccessful collaborative study conducted in 2003, concluded the following figures:

- Repeatability r for goat milk of 0,004 with a minimum 0,003 of and a maximum of 0,007 (For cow milk $r = 0,004$ as reported on FIL-IDF 108:2002).
- Reproducibility R for goat milk is 0,008 with a minimum 0,007 of and a maximum of 0,011 (For cow milk $R=0,006$ as reported on FIL-IDF 108:2002).

In the Collaborative Study 2004, the results obtained on cow milk demonstrate the validity of the laboratories participating, and the figures for goat milk confirmed the results obtained in previous Collaborative Study 2003. See [Table 2](#).

The reproducibility value (0,008) obtained in this collaborative study on goat milk is higher than that in cow milk (0,006) while the repeatability value (0,004) is the same for both species.

1.6. Proposal for precision figures for goat milk

Precision. Precision data were determined from a collaborative study conducted in 2004 in which 12 laboratories participated. Six samples with a freezing point level varying from $-0,597^{\circ}\text{C}$ to $-0,542^{\circ}\text{C}$ were analyzed in duplicates (double blind). Of the 144 series of results received, 48 were eliminated because not in conformity with Standard ISO 5764|IDF 108:2002. Hence 96 values were considered valid.

Repeatability. The repeatability standard deviation, which expresses the variability of independent analytical results obtained by the same operator, using the same apparatus under the same conditions on the same test sample and in a short interval of time, should not exceed $0,002^{\circ}\text{C}$.

If two determinations are carried out under these conditions, the absolute difference between the two results should not be greater than $0,004^{\circ}\text{C}$.

Reproducibility. The relative standard deviation of the reproducibility, which expresses the variability of independent analytical results obtained by operators in different laboratories using different apparatus under different conditions for the analysis on the same test sample, should not exceed $0,003^{\circ}\text{C}$.

If two determinations are carried out under these conditions, the relative difference between the two results should not be greater than $0,008^{\circ}\text{C}$.

References

ISO 5725-1, Accuracy (trueness and precision) of measurement methods and results. Part 1: General principles and definitions.

ISO 5725-2, Accuracy (trueness and precision) of measurement methods and results. Part 2: A basic method for the determination of repeatability and reproducibility of a standard measurement method.

Table 1. Quality control of the samples

COLLABORATIVE STUDY ID SAMPLES (Freezing Point)								
SAMPLE	A	B	C	D	E	F	G**	H**
1° REP	8	9	3	4	5	6	1	2
2° REP	13	12	15	10	11	14	16	7
UFC/ml	91'000	155'000	299'000	142'000	320'000	225'000	90'000	108'000
SCC (cells/ml)	115'000	180'000	110'000	80'000	310'000	293'000	110'000	250'000

Table 2. Comparison with precision figures of previous studies

Goat Samples	Labs	Mean °C	r	R	Sr	SR
A	8	-0,542	0,002	0,009	0,001	0,003
B	8	-0,573	0,003	0,006	0,001	0,002
C	8	-0,550	0,005	0,008	0,002	0,003
D	8	-0,554	0,003	0,009	0,001	0,003
E	8	-0,562	0,005	0,008	0,002	0,003
F	8	-0,597	0,004	0,008	0,002	0,003
	TOTAL	-0,563	0,004	0,008	0,002	0,003
	CS 2003 on Goat Milk		0,004	0,009		
Cow Samples	Labs	Mean °C	r	R	Sr	SR
G	5	-0,521	0,001	0,003	0,000	0,001
H	7	-0,530	0,004	0,006	0,001	0,002
	TOTAL	-0,526	0,002	0,004	0,001	0,002
	ISO 5764 IDF Standard 108:2002 figures for cow milk		0,004	0,006		

Table 3. Statistical elaboration



**COLLABORATIVE STUDY ON GOAT MILK FEBRUARY 2004
FREEZING POINT °C
AFTER SCRUTINIZATION**



COLLABORATIVE STUDY ID SAMPLES								
SAMPLE	A	B	C	D	E	F	G**	H*
1 st REP	8	9	3	4	5	6	1	2
2 nd REP	13	12	15	10	11	14	16	7

COMPLETED DATA RESULTS

	LAB1	LAB2	LAB5	LAB6
A	-0.542	-0.542	-0.546	-0.546
B	-0.571	-0.570	-0.576	-0.578
C	-0.549	-0.549	-0.552	-0.555
D	-0.554	-0.552	-0.558	-0.557
E	-0.561	-0.560	-0.566	-0.566
F	-0.597	-0.597	-0.600	-0.602
G**	-0.521	-0.521	-0.530*	-0.532*
H*	-0.530	-0.529	-0.533	-0.534

	LAB7	LAB8	LAB11	LAB13
A	-0.540	-0.541	-0.539	-0.540
B	-0.571	-0.573	-0.572	-0.572
C	-0.549	-0.548	-0.550	-0.548
D	-0.553	-0.554	-0.554	-0.548
E	-0.569	-0.568	-0.562	-0.568
F	-0.594	-0.592	-0.592	-0.596
G**	-0.519	-0.520	-0.522	-0.522
H*	-0.526	-0.528	-0.531	-0.528

*lab2 sample G: samples in bad conditions, results rejected

ABSOLUTE DIFFERENCE BETWEEN DUPLICATES

	LAB1	LAB2	LAB5	LAB6	LAB7	LAB8	LAB11	LAB13
A	0.000	0.000	0.002	0.001	0.001	0.001	0.001	0.002
B	0.001	0.002	0.001	0.001	0.002	0.000	0.001	0.002
C	0.000	0.003	0.003	0.001	0.001	0.002	0.003	0.004
D	0.002	0.001	0.001	0.001	0.001	0.000	0.002	0.001
E	0.001	0.000	0.000	0.005	0.001	0.004	0.002	0.003
F	0.001	0.002	0.002	0.000	0.002	0.004	0.001	0.003
G**	0.000		0.001	0.000	0.001	0.000	0.000	0.006
H*	0.001	0.001	0.003	0.001	0.002	0.003	0.001	0.011

REPEATABILITY AND REPRODUCIBILITY ON GOAT MILK

Sample	Labs*	Mean	r	R	Sr	SR
A	8	-0.542	0.002	0.009	0.001	0.003
B	8	-0.573	0.003	0.006	0.001	0.002
C	8	-0.550	0.005	0.008	0.002	0.003
D	8	-0.554	0.003	0.009	0.001	0.003
E	8	-0.562	0.005	0.008	0.002	0.003
F	8	-0.597	0.004	0.008	0.002	0.003
TOTAL		-0.563	0.004	0.008	0.002	0.003

REPEATABILITY AND REPRODUCIBILITY ON COW MILK

Sample	Labs*	Mean	r	R	Sr	SR
G**	5	-0.521	0.001	0.003	0.000	0.001
H*	7	-0.530	0.004	0.006	0.001	0.002
TOTAL		-0.526	0.002	0.004	0.001	0.002

MEANS OF DUPLICATES

	LAB1	LAB2	LAB5	LAB6	LAB7	LAB8	LAB11	LAB13	MEAN	MIN	MAX	SD	REF.
A	-0.542	-0.542	-0.542	-0.544	-0.541	-0.540	-0.536	-0.546	-0.542	-0.546	-0.536	0.003	-0.542
B	-0.571	-0.577	-0.571	-0.574	-0.572	-0.572	-0.570	-0.574	-0.572	-0.577	-0.570	0.002	-0.572
C	-0.549	-0.554	-0.549	-0.553	-0.549	-0.549	-0.546	-0.553	-0.550	-0.554	-0.546	0.003	-0.549
D	-0.553	-0.553	-0.553	-0.555	-0.554	-0.554	-0.546	-0.556	-0.554	-0.558	-0.546	0.003	-0.554
E	-0.561	-0.566	-0.561	-0.565	-0.560	-0.560	-0.558	-0.563	-0.562	-0.566	-0.558	0.003	-0.561
F	-0.597	-0.601	-0.597	-0.599	-0.593	-0.594	-0.594	-0.598	-0.596	-0.601	-0.593	0.003	-0.597
m lab	-0.562	-0.567	-0.562	-0.565	-0.561	-0.561	-0.558	-0.565	-0.563	-0.601	-0.536	0.003	-0.562

MEANS OF DUPLICATES (COW MILK SAMPLES)

	LAB1	LAB2	LAB5	LAB6	LAB7	LAB8	LAB11	LAB13	MEAN	MIN	MAX	SD	REF.
G**	-0.521		-0.522		-0.520	-0.522	-0.522	-0.526	-0.521	-0.522	-0.520	0.001	-0.522
H*	-0.530	-0.534	-0.529	-0.531	-0.527	-0.530	-0.529	-0.528	-0.530	-0.534	-0.527	0.002	-0.530
m lab	-0.525	-0.534	-0.526	-0.531	-0.523	-0.526	-0.525	-0.527	-0.525	-0.534	-0.523	0.002	-0.526

PILOT SAMPLES ANALYSES				
	PS1	PS2	PS3	SD LAB
LAB1	-0.561	-0.561	-0.559	0.001
LAB2	-0.559	-0.563	-0.561	0.002
LAB5	-0.560	-0.560	-0.559	0.001
LAB6	-0.557	-0.558	-0.557	0.001
LAB7	-0.559	-0.558	-0.559	0.001
LAB8	-0.558	-0.558	-0.558	0.000
LAB11	-0.557	-0.557	-0.557	0.000
LAB13	-0.558	-0.561	-0.563	0.003
SD P S	0.002	0.002	0.002	

* : number of labs after outlier elimination
 value : outlier value by Cochran Test
 r : repeatability
 R : reproducibility
 Sr : repeatability standard deviation
 SR : reproducibility standard deviation
 RSDr : repeatability relative standard deviation
 RSDR : reproducibility relative standard deviation
 RSDL : the relative standard deviation between laboratories

RANKING OF LABS ON D VALUE (EUCLIDIAN DISTANCE FROM REF VALUES)

RANK	GOAT MILK SAMPLES			COW MILK SAMPLES		
	LAB CODE	D	DR%	LAB CODE	D	DR%
I	LAB 5	0.0006	12%	LAB 1	0.0000	0%
II	LAB 1	0.0007	16%	LAB 2	0.0004	12%
III	LAB 8	0.0015	32%	LAB 7	0.0006	18%
IV	LAB 7	0.0017	36%	LAB 13	0.0009	24%
V	LAB 6	0.0027	59%	LAB 8	0.0025	68%
VI	LAB 13	0.0030	65%	LAB 11	0.0037	100%
VII	LAB 11	0.0045	98%	LAB 9		
VIII	LAB 2	0.0046	100%	LAB 6		

Table 3. Statistical elaboration - continued

Z SCORE CALCULATED ON REFERENCE VALUE								
	LAB1	LAB2	LAB5	LAB6	LAB7	LAB8	LAB11	LAB13
-B CAMP.A	-0,073	-1,249	0,073	-0,514	0,267	0,661	1,690	-1,323
-B CAMP.B	0,662	-2,207	0,441	-0,662	0,000	0,000	0,893	-0,794
-B CAMP.C	0,000	-1,626	0,000	-1,265	0,181	0,000	1,266	-1,482
-B CAMP.D	0,224	-1,119	0,224	-0,522	0,075	-0,075	2,239	-0,612
-B CAMP.E	0,000	-2,009	0,000	-1,461	0,265	0,183	0,913	-0,968
-B CAMP.F	0,000	-1,636	0,000	-0,909	1,273	0,909	0,909	-0,473
-B LAB	0,115	-1,678	0,115	-0,861	0,373	0,287	1,362	-0,941

Z SCORE CALCULATED ON REFERENCE VALUE (COM MILK SAMPLES)								
	LAB1	LAB2	LAB5	LAB6	LAB7	LAB8	LAB11	LAB13
-B CAMP.G**	0,913		0,000		2,282	0,000	0,000	-3,423
-B CAMP.H**	0,000	-2,007	0,251	-0,502	1,254	0,000	0,376	0,562
-B LAB	0,311	-4,819	0,155	-2,953	1,654	0,000	0,233	-0,824

DIFFERENCES FROM THE REFERENCE VALUE								
	LAB1	LAB2	LAB5	LAB6	LAB7	LAB8	LAB11	LAB13
A	0,000	-0,004	0,000	-0,002	0,001	0,002	0,006	-0,005
B	0,001	-0,005	0,001	-0,001	0,000	0,000	0,002	-0,002
C	0,000	-0,005	0,000	-0,003	0,001	0,000	0,003	-0,004
D	0,001	-0,004	0,001	-0,002	0,000	0,000	0,008	-0,002
E	0,000	-0,005	0,000	-0,004	0,001	0,000	0,002	-0,003
F	0,000	-0,004	0,000	-0,002	0,004	0,003	0,002	-0,001
mdiff	0,000	-0,005	0,000	-0,003	0,001	0,001	0,004	-0,003
dsdiff	0,001	0,001	0,000	0,001	0,001	0,001	0,002	0,001
D	0,001	0,005	0,001	0,003	0,002	0,001	0,005	0,003
SLOPE	1,006	0,992	0,999	0,999	1,043	1,018	0,932	1,057
BIAS	0,003	0,000	-0,001	0,002	0,023	0,009	-0,042	0,035
CORREL	0,999	1,000	1,000	0,999	0,999	0,998	0,996	0,999

DIFFERENCES FROM THE REFERENCE VALUE (COM MILK SAMPLES)								
	LAB1	LAB2	LAB5	LAB6	LAB7	LAB8	LAB11	LAB13
G**	0,001		0,000		0,002	0,000	0,000	-0,004
H**	0,000		0,001		0,003	0,000	0,001	0,001
mdiff	0,001		0,000		0,003	0,000	0,000	-0,001
dsdiff	0,001		0,000		0,003	0,000	0,001	0,003
D	0,001		0,000		0,003	0,000	0,001	0,004
SLOPE	0,882		1,071		1,000	1,000	1,111	2,830
BIAS	-0,062		0,037		-0,002	0,000	0,058	0,066
CORREL	1,000		1,000		1,000	1,000	1,000	1,000

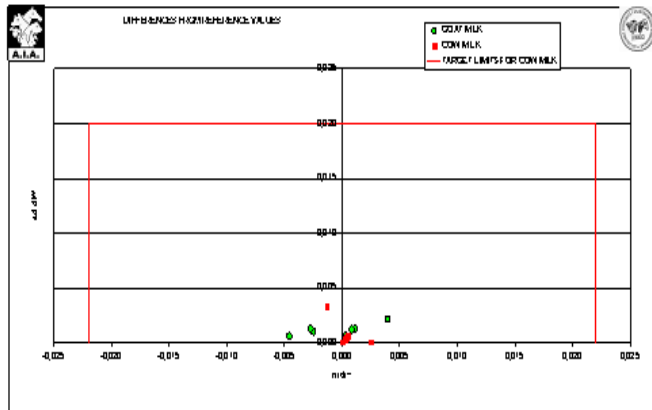
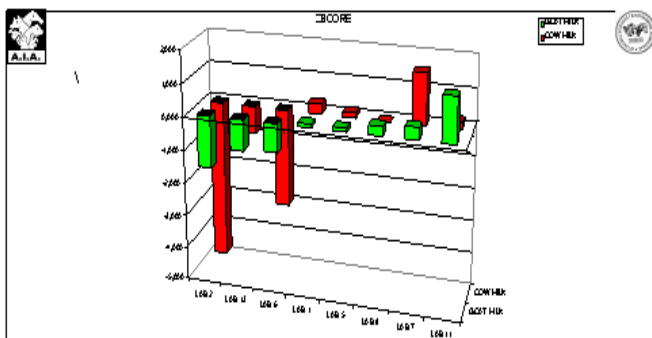


Table 4. Table of Precision per level goat milk

Samples	A	B	C	D	E	F	Grand Mean ^a
No of participating laboratories after eliminating outliers	8	8	8	8	8	8	
Mean value, m °C	-542,0	-573,0	-550,0	-554,0	-562,0	-597,0	-563,0
Repeatability standard deviation, s _r , m °C	1,0	1,0	2,0	1,0	2,0	2,0	1,6
Repeatability limit r (2,8 s _r), m °C	2,8	2,8	5,6	2,8	5,6	5,6	4,4
Coefficient of variation of repeatability, %	-0,52	-0,49	-1,02	-0,51	-1,00	-0,84	-0,74
Reproducibility standard deviation, s _R , m °C	3,0	2,0	3,0	3,0	3,0	3,0	2,9
Reproducibility limit R (2,8 s _R), m °C	8,4	5,6	8,4	8,4	8,4	8,4	8,0
Coefficient of variation of reproducibility, %	-1,55	-0,98	-1,53	-1,52	-1,49	-1,41	-1,41

^a Grand mean values were calculated using only sample data with outliers removed. Grand sample mean value and coefficient of variation were calculated by averaging. All other statistical means were calculated from the square root of the average of the square deviation.

2. Collaborative study on the freezing point of Sheep Milk

S. Orlandini¹, L. Lattanzi¹, A. Carducci¹, G. Psathas², U. Paggi¹

Abstract

Precision data were determined by a collaborative study conducted in 2004 in which 12 laboratories participated. Six sheep milk samples with a freezing point level varying from $-0,490^{\circ}\text{C}$ to $-0,584^{\circ}\text{C}$ were analysed in blind duplicates. Of the 144 series of results received, 48 were eliminated because they were not in conformity with Standard ISO 5764|IDF 108: 2002, and one value was eliminated as outlier. 95 values were therefore considered as valid results.

The statistical elaboration gave a repeatability value $r=0,006^{\circ}\text{C}$ and a reproducibility value $R=0,010^{\circ}\text{C}$

2.1. Introduction

A first collaborative study conducted in April 2003 lead to unsatisfactory results. Hence a new collaborative study on sheep milk was organized in March 2004 by Co-Project Leader Ugo Paggi.

2.2. Lay-out of the study

2.2.1. Organization and participating laboratories

During Autumn 2003, 36 international laboratories were contacted by C.M.I.O. inviting them to take part in the second collaborative study on sheep milk.

The final list of participating laboratories is as follows (in alphabetical order):

AIA - LSL	Italy
Advanced Instruments	USA
AFSSA	France
Cecalait	France
CLO-DVK	Belgium
C.M.I.O.	Cyprus
Danish Vet. And Food Administration	Denmark
Lab.Agroalimentario de Santander	Spain
Lab. de Lactologia e Sanidad animal	Spain
MLUA	Germany
Netherlands Milk Control Station	Netherlands
Univ. of Ljubljana Biotech Fac. Zootech.	Slovenia

2.2.2. Samples

The samples were prepared and dispatched by AIA-LSL on the 25th of March by international express courier.

Sixteen samples were prepared in the following way:

- Six samples, in blind duplicates, of sheep raw milk with 0,02% Bronopol added, ranging from $-0,491^{\circ}\text{C}$ to $-0,583^{\circ}\text{C}$;
- Two samples, in blind duplicates, of cow raw milk with 0,02% Bronopol added, with a freezing point of $-0,522^{\circ}\text{C}$ and $-0,534^{\circ}\text{C}$.

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² Cyprus Milk Industry Organisation, Nicosia - Cyprus

The choice of inserting cow milk samples was taken in order to evaluate the performance of the laboratories.

All the samples were identified by numbers from 1 to 16, with no differentiation between cow and sheep milk samples.

Furthermore all the labs received 3 solutions to be used as pilot controls, and 3 standard solutions for instrument calibration.

Together with the samples explanatory papers were sent concerning Package content, Instructions, and Questionnaire.

All the packages were delivered in about 24 hours at the average temperature of 3°C (each package included a water vial for measuring temperature upon package reception).

The quality of samples before the shipping was monitored by Total Bacterial Count and Somatic Cell Count by fluoro-opto-electronic instruments flow cytometry technique. See [Table 1](#).

The stability of the samples was checked by sending a package back and forth to our laboratory by airmail, in order to simulate the delivery of samples. The package was received 48 hours after expedition, and the temperature on the water vial was 4,5°C.

These samples were processed through Bacterial Count and IR Instrument, and no significant differences were found in results obtained after 48 hours.

2.2.3. Method

The method used was ISO 5764|IDF 108: 2002 (Thermistor cryoscope method).

2.2.4. Instructions

The following instructions were enclosed in the package.

Application of the method ISO 5764|IDF 108:2002 on samples n° 1- 16 (glass vials with black caps). Prepare instrument by leaving power on at least for 1 hour for circuit stabilization.

The instrument should be connected on UPS power supplier.

Check position of the thermistor probe and stirrer to be in accordance with the standard ISO 5764|IDF 108:2002 (paragraph 6.1.2 and 6.1.3).

Check amplitude of stirring wire to be in accordance with the standard ISO 5764|IDF 108:2002.

Sample quantity 2,5±0,1ml.

Freezing pulse at -3,0 m°C.

Measurement cut-off when temperature rise has been maximal 0,5 m°C over last 20s (resolution better than 1 m°C) or the read out has been constant during last 20 s (resolution not better than 1 m°C).

Bring all standard solutions to room temperature.

Calibrate instrument with the standard solutions -0,408°C and -0,600°C according to instrument manufacturer instructions. Verify the instrument calibration with the standard solution -0,512°C. The results should not differ by more than 2 m°C from -0,512°C. If the difference is higher, recalibrate the instrument, otherwise do not make adjustments on the instrument.

Samples preparation: Shortly before determinations, warm the samples to 40°C in a water bath. Homogenize samples by inverting gently 6-8 times. Then cool to 20°C and homogenize again by inverting gently 6-8 times. Warning : avoid the incorporation of air. The samples are ready for testing: pipette 2,5±0,1ml.

Analyze the pilot sample and record the result (we suggest to change the pilot sample vial in the next steps of analysis).

Analyze once 8 samples in their respective order (1 to 8) and repeat analysis of the pilot sample (we suggest to use a brand new pilot sample).

Analyze once the rest of the 8 samples (9 to 16) in their respective order and repeat analysis of the pilot sample (we suggest to use a brand new pilot sample).

Record the results to the fourth digit without rounding (or third digit depending on instrument's resolution) on the paper form and then in the excel file you received by email. Record any comments or observation you may have on the appropriate box.

Please complete the questionnaire accurately.

Return the excel file via email to dati.lsl@aia.it

2.2.5. Application Questionnaire

The following questionnaire was sent with the samples. The results of this questionnaire have been utilized for laboratory scrutinization.

SAMPLES RECEPTION

1. Days between delivery and reception:
2. Samples temperature:.....
3. Samples condition (any leakage etc):
Storage of samples (if not analyzed immediately):
Temperature:..... Hours:.....

SAMPLES PREPARATION

1. Upon unscrewing the caps did you observe any fat stains on the samples surface?
YES NO
If yes, please state the identification number of samples(1-16):.....
Please state the temperature of warming up for homogenization:.....
2. Please state the temperature of samples drawing:.....
3. During the homogenization process, did you observe any kind of lypolysis in samples?
YES NO
If yes please state the identification number of samples (1-16).....

SAMPLES VOLUME

1. Please state the samples volume used:

INSTRUMENT SET UP

1. Please state the instrument model:.....
2. Please state the Freezing temperature prior to stirring:.....
3. Please state the plateau used:
 - a. Fixed plateau: - Time:
 - b. Seeking plateau: - Time:

INSTRUMENT CALIBRATION

1. Did you face any problem on calibration process with solutions received?
YES NO
If yes, please describe:
2. What were the duplicate values you received on verifying the calibration with the standard solution $-0,512^{\circ}\text{C}$: 1..... 2.....

SAMPLES MEASURING

1. Were all samples measured continuously?
YES NO
2. Did you have any prefreeze on any sample?
YES NO
If yes, please state the identification number of samples (1-16):.....

2.3. Returning of results

The results were registered on an excel file provided via e-mail to all the participants and sent back to AIA-LSL.

2.4. Analysis of results

The 16 samples results were paired in order to have 6 samples of sheep milk (A, B, C, D, E, F) and 2 samples of cow milk (G and H). See [Table 1](#).

The answers to the questionnaire were collected by CMIO. Once elaborated, the results obtained were utilized for laboratory and/or sample scrutinization.

As a result, 3 labs (lab n° 8, 9 and 15) were rejected due to lack of conformity with Standard ISO 5764/IDF 108: 2002; Laboratory n°7 commented that samples 1, 2, 5, 8, 9 and 13 were in bad condition (fat stains on surface), therefore lab n° 7 was also excluded from figures calculation.

Statistical elaboration was conducted according to Standard ISO 5725-2:1994.

- Absolute difference between duplicates; the Cochran's Test was applied to target outlier values;
- Mean of duplicates; the Grubbs' Test sample was applied and then Zlab (one Zscore for each laboratory) was calculated and reported on a graphic.
- Difference from the Reference value, then plotted on a diagram; the reference values were calculated making the median of the values after outlier elimination. See [Table 3](#).

2.5. Results and conclusions

The unsuccessful collaborative study conducted in 2003, concluded the following figures:

- Repeatability r for sheep milk of 0,006 with a minimum 0,002 of and a maximum of 0,001 (For cow milk r = 0,004 as reported on ISO 5764/IDF 108:2002).
- Reproducibility R for sheep milk is 0,010 with a minimum 0,009 of and a maximum of 0,017 (For cow milk R=0,006 as reported on FIL-IDF 108:2002).

In the Collaborative Study, the results obtained on cow milk demonstrate the validity of the laboratories participating, and the figures for sheep milk confirmed the results obtained previously in the Collaborative Study of 2003 regarding the repeatability figure, while a major improvement was found in reproducibility value (0,010 vs. 0,012), probably due to scrutinization of results. See [Table 2](#).

2.6. Proposal for precision figures for sheep milk

Precision. Precision data were determined from a collaborative study conducted in 2004 in which 12 laboratories participated. Six samples with a freezing point level varying from $-0,490^{\circ}\text{C}$ to $-0,584^{\circ}\text{C}$ were analyzed in duplicate (double blind). Of the 144 series of results received, 48 were eliminated because not in conformity with standard ISO 5764/IDF 108: 2002, and one value was eliminated as outlier. As a result 95 values were considered as valid results.

Repeatability. The repeatability standard deviation, which expresses the variability of independent analytical results obtained by the same operator, using the same apparatus under the same conditions on the same test sample and in a short interval of time, should not exceed $0,002^{\circ}\text{C}$.

If two determinations are carried out under these conditions, the absolute difference between the two results should not be greater than $0,006^{\circ}\text{C}$.

Reproducibility. The relative standard deviation of the reproducibility, which expresses the variability of independent analytical results obtained by operators in different laboratories using different apparatus under different conditions for the analysis on the same test sample, should not exceed $0,004^{\circ}\text{C}$.

If two determinations are carried out under these conditions, the relative difference between the two results should not be greater than $0,010^{\circ}\text{C}$. See [Table 4](#).

References

ISO 5725-1, Accuracy (trueness and precision) of measurement methods and results. Part 1: General principles and definitions.

ISO 5725-2, Accuracy (trueness and precision) of measurement methods and results. Part 2: A basic method for the determination of repeatability and reproducibility of a standard measurement method.

Table 1. Quality control of the samples

SAMPLE	COLLABORATIVE STUDY ID SAMPLES (Freezing Point)							
	A	B	C	D	E	F	G**	H**
1° REP	1	5	3	7	2	8	4	6
2° REP	11	10	15	16	14	13	12	9
UFC/ml	320'000	710'000	788'000	620'000	623'000	820'000	210'000	40'000
SCC (cells/ml)	225'000	347'000	522'000	426'000	313'000	580'000	120'000	80'000

Table 2. Comparison with precision figures of previous studies

Sheep Samples	Labs	Mean °C	r	R	Sr	SR
A	7	-0,533	0,002	0,005	0,001	0,002
B	8	-0,572	0,007	0,012	0,002	0,004
C	8	-0,516	0,003	0,006	0,001	0,002
D	8	-0,490	0,005	0,010	0,002	0,003
E	8	-0,584	0,005	0,010	0,002	0,004
F	8	-0,572	0,009	0,014	0,003	0,005
	TOTAL	-0,545	0,006	0,010	0,002	0,004
	CS 2003 on Sheep Milk		0,005	0,0118		

Table 3. Statistical elaboration



COLLABORATIVE STUDY ON SHEEP MILK MARCH 2004



FREEZING POINT °C

AFTER CENTRIFUGATION

SAMPLE	COLLABORATIVE STUDY 10 SAMPLES							
	A	B	C	D	E	F	G**	H**
1 st REP	1	5	3	7	2	8	4	6
2 nd REP	11	10	15	16	14	13	12	9

COMPLETE DATA RESULTS								
	LAB 1	LAB 2	LAB 3	LAB 5	LAB 10	LAB 11	LAB 13	LAB 14
A	-0.531	-0.530	-0.534	-0.535	-0.532	-0.532	-0.532	-0.533
B	-0.570	-0.566	-0.574	-0.575	-0.570	-0.572	-0.577	-0.577
C	-0.515	-0.514	-0.517	-0.518	-0.514	-0.514	-0.517	-0.516
D	-0.487	-0.486	-0.491	-0.492	-0.491	-0.489	-0.491	-0.490
E	-0.581	-0.579	-0.584	-0.585	-0.583	-0.582	-0.583	-0.583
F	-0.566	-0.566	-0.578	-0.577	-0.569	-0.569	-0.572	-0.580
G**	-0.519	-0.521	-0.521	-0.522	-0.520	-0.522	-0.523	-0.523
H**	-0.533	-0.534	-0.536	-0.535	-0.532	-0.531	-0.534	-0.532
A	-0.532	-0.532	-0.531	-0.532	-0.534	-0.535	-0.536	-0.542
B	-0.566	-0.572	-0.569	-0.569	-0.575	-0.570	-0.578	-0.579
C	-0.517	-0.515	-0.517	-0.515	-0.517	-0.517	-0.520	-0.522
D	-0.494	-0.490	-0.488	-0.489	-0.493	-0.490	-0.496	-0.499
E	-0.584	-0.582	-0.582	-0.585	-0.583	-0.586	-0.589	-0.594
F	-0.570	-0.572	-0.565	-0.570	-0.576	-0.572	-0.571	-0.579
G**	-0.522	-0.522	-0.521	-0.518	-0.522	-0.523	-0.524	-0.526
H**	-0.529	-0.531	-0.535	-0.533	-0.533	-0.534	-0.537	-0.536

PILOT SAMPLES ANALYSES				
	PS1	PS2	PS3	SD LAB
LAB 1	-0.559	-0.559	-0.558	0.001
LAB 2	-0.558	-0.560	-0.560	0.001
LAB 3	-0.559	-0.560	-0.557	0.002
LAB 5	-0.559	-0.560	-0.561	0.001
LAB 7	-0.556	-0.558	-0.558	0.001
LAB 8	-0.557	-0.558	-0.558	0.001
LAB 9	-0.558	-0.559	-0.557	0.001
LAB 10	-0.557	-0.557	-0.557	0.000
LAB 11	-0.560	-0.559	-0.559	0.001
LAB 13	-0.559	-0.560	-0.559	0.001
LAB 14	-0.558	-0.560	-0.562	0.002
SD PS	0.001	0.001	0.002	

ABSOLUTE DIFFERENCE BETWEEN DUPLICATES								
	LAB 1	LAB 2	LAB 3	LAB 5	LAB 10	LAB 11	LAB 13	LAB 14
A	0.001	0.001	0.000	0.001	0.000	0.001	0.000	0.006
B	0.005	0.001	0.002	0.000	0.006	0.000	0.005	0.001
C	0.001	0.001	0.000	0.001	0.002	0.001	0.000	0.002
D	0.001	0.001	0.002	0.001	0.004	0.001	0.004	0.003
E	0.002	0.001	0.001	0.000	0.002	0.003	0.003	0.005
F	0.001	0.001	0.000	0.000	0.002	0.005	0.004	0.008
G**	0.002	0.001	0.002	0.000	0.000	0.004	0.001	0.002
H**	0.001	0.001	0.001	0.002	0.002	0.001	0.001	0.001

* : number of flabs after outlier elimination
 value : outlier value by Cochran Test
 r : repeatability
 R : reproducibility
 Sr : repeatability standard deviation
 SR : reproducibility standard deviation
 RSDr : repeatability relative standard deviation
 RSDR : reproducibility relative standard deviation
 RSDL : the relative standard deviation between laboratories

REPEATABILITY AND REPRODUCIBILITY ON SHEEP MILK						
Sample	Lab**	Mean	r	R	Sr	SR
A	7	-0.533	0.002	0.005	0.001	0.002
B	8	-0.572	0.007	0.012	0.002	0.004
C	8	-0.516	0.003	0.006	0.001	0.002
D	8	-0.490	0.005	0.010	0.002	0.003
E	8	-0.584	0.005	0.010	0.002	0.004
F	8	-0.572	0.009	0.014	0.003	0.005
TOTAL		-0.545	0.006	0.010	0.002	0.004

REPEATABILITY AND REPRODUCIBILITY ON COW MILK						
Sample	Lab**	Mean	r	R	Sr	SR
G**	9	-0.522	0.004	0.006	0.001	0.002
H**	9	-0.534	0.003	0.006	0.001	0.002
TOTAL		-0.528	0.003	0.006	0.001	0.002

RANKING OF LABS ON D VALUE (EUCLIDIAN DISTANCE FROM REF VALUES)						
RANK	SHEEP MILK SAMPLES			COW MILK SAMPLES		
	LAB CODE	D	CV%	LAB CODE	D	CV%
I	LAB 10	0.0011	23%	LAB 10	0.0008	42%
II	LAB 13	0.0011	23%	LAB 13	0.0008	42%
III	LAB 2	0.0020	41%	LAB 1	0.0012	62%
IV	LAB 5	0.0021	43%	LAB 2	0.0014	70%
V	LAB 3	0.0026	53%	LAB 5	0.0017	87%
VI	LAB 11	0.0036	74%	LAB 3	0.0020	100%
VII	LAB 1	0.0048	100%	LAB 11	0.0020	100%

MEANS OF DUPLICATES													
	LAB 1	LAB 2	LAB 3	LAB 5	LAB 10	LAB 11	LAB 13	LAB 14	MEAN	MIN	MAX	SD	MEDIAN
A	-0.531	-0.535	-0.532	-0.533	-0.534	-0.531	-0.534	-0.535	-0.533	-0.536	-0.531	0.002	-0.533
B	-0.568	-0.575	-0.571	-0.577	-0.573	-0.569	-0.573	-0.574	-0.572	-0.577	-0.568	0.003	-0.573
C	-0.515	-0.518	-0.514	-0.517	-0.517	-0.516	-0.517	-0.519	-0.516	-0.519	-0.514	0.002	-0.517
D	-0.487	-0.492	-0.490	-0.491	-0.491	-0.488	-0.491	-0.493	-0.490	-0.493	-0.487	0.002	-0.491
E	-0.580	-0.585	-0.583	-0.583	-0.585	-0.583	-0.585	-0.588	-0.584	-0.588	-0.580	0.002	-0.584
F	-0.566	-0.578	-0.569	-0.576	-0.574	-0.567	-0.574	-0.571	-0.572	-0.578	-0.566	0.004	-0.573
m lab	-0.541	-0.547	-0.543	-0.546	-0.546	-0.542	-0.546	-0.547	-0.545	-0.588	-0.487	0.003	-0.545

MEANS OF DUPLICATES (COW MILK SAMPLES)													
	LAB 1	LAB 2	LAB 3	LAB 5	LAB 10	LAB 11	LAB 13	LAB 14	MEAN	MIN	MAX	SD	MEDIAN
G**	-0.520	-0.522	-0.521	-0.523	-0.522	-0.519	-0.522	-0.523	-0.522	-0.523	-0.519	0.001	-0.522
H**	-0.534	-0.536	-0.532	-0.533	-0.534	-0.534	-0.534	-0.536	-0.534	-0.536	-0.532	0.001	-0.534
m lab	-0.527	-0.529	-0.526	-0.528	-0.528	-0.527	-0.528	-0.530	-0.528	-0.529	-0.526	0.001	-0.528

MEANS OF DUPLICATES (at the values do not take part Mean, Min, Max, Sd calculation)													
	LAB 1	LAB 2	LAB 3	LAB 5	LAB 10	LAB 11	LAB 13	LAB 14	MEAN	MIN	MAX	SD	REF. VALUE
A	-0.531	-0.535	-0.532	-0.533	-0.534	-0.531	-0.534	-0.535	-0.533	-0.536	-0.531	0.002	-0.533
B	-0.568	-0.575	-0.571	-0.577	-0.573	-0.569	-0.573	-0.574	-0.572	-0.577	-0.568	0.003	-0.573
C	-0.515	-0.518	-0.514	-0.517	-0.517	-0.516	-0.517	-0.519	-0.516	-0.519	-0.514	0.002	-0.517
D	-0.487	-0.492	-0.490	-0.491	-0.491	-0.488	-0.491	-0.493	-0.490	-0.493	-0.487	0.002	-0.491
E	-0.580	-0.585	-0.583	-0.583	-0.585	-0.583	-0.585	-0.588	-0.584	-0.588	-0.580	0.002	-0.584
F	-0.566	-0.578	-0.569	-0.576	-0.574	-0.567	-0.574	-0.571	-0.572	-0.578	-0.566	0.004	-0.573
m lab	-0.541	-0.547	-0.543	-0.546	-0.546	-0.542	-0.546	-0.547	-0.545	-0.588	-0.487	0.003	-0.545

MEANS OF DUPLICATES (COW MILK SAMPLES) (at the values do not take part Mean, Min, Max, Sd calculation)													
	LAB 1	LAB 2	LAB 3	LAB 5	LAB 10	LAB 11	LAB 13	LAB 14	MEAN	MIN	MAX	SD	REF. VALUE
G**	-0.520	-0.522	-0.521	-0.523	-0.522	-0.519	-0.522	-0.523	-0.522	-0.523	-0.519	0.001	-0.522
H**	-0.534	-0.536	-0.532	-0.533	-0.534	-0.534	-0.534	-0.536	-0.534	-0.536	-0.532	0.001	-0.534
m lab	-0.527	-0.529	-0.526	-0.528	-0.528	-0.527	-0.528	-0.530	-0.528	-0.529	-0.526	0.001	-0.528

Table 3. Statistical elaboration - continued

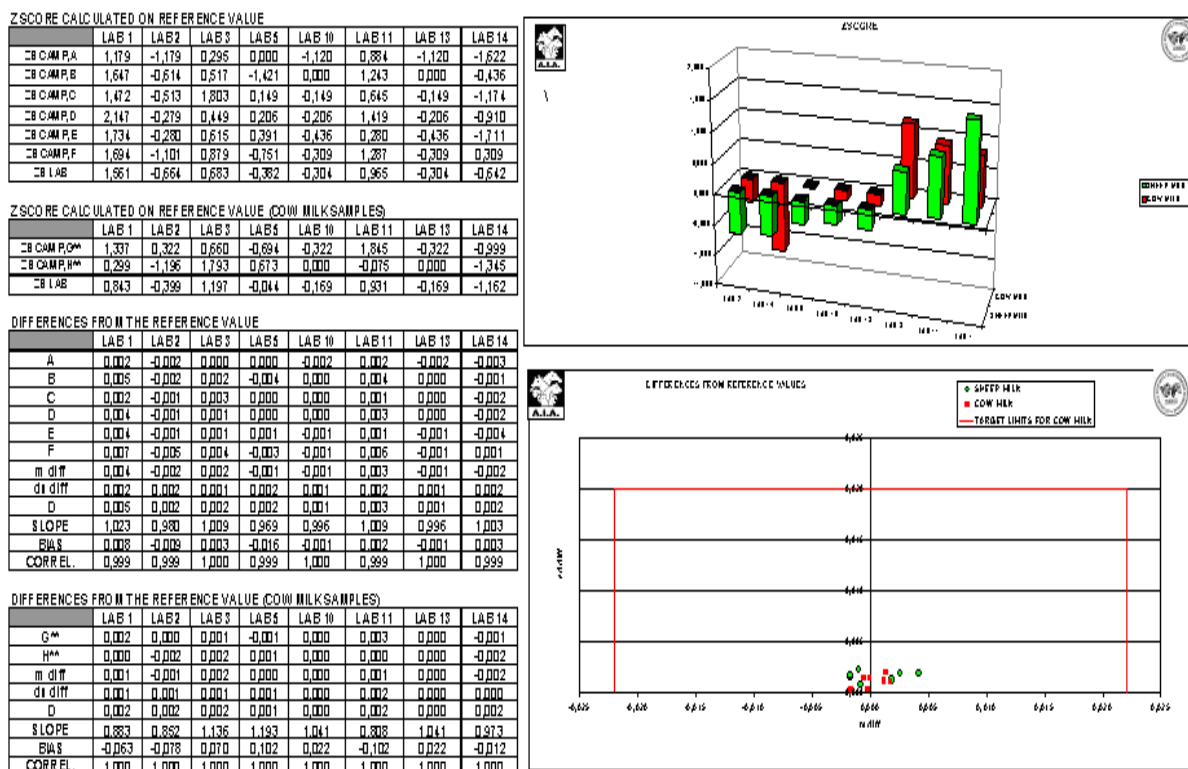


Table 4. Table of Precision per level sheep milk

Samples	A	B	C	D	E	F	Grand Mean ^a
No of participating laboratories after eliminating outliers	7	8	8	8	8	8	
Mean value, m °C	-532,8	-572,2	-516,3	-490,3	-583,8	-571,9	-544,5
Repeatability standard deviation, s _r , m °C	1,0	2,0	1,0	2,0	2,0	3,0	2,0
Repeatability limit r (2,8* s _r) m °C	2,8	5,6	2,8	5,6	5,6	8,4	5,5
Coefficient of variation of repeatability, %	-0,53	-0,98	-0,54	-1,14	-0,96	-1,47	-0,94
Reproducibility standard deviation, s _R , m °C	2,0	4,0	2,0	3,0	4,0	5,0	3,5
Reproducibility limit R (2,8* s _R) m °C	5,6	11,2	5,6	8,4	11,2	14,0	9,8
Coefficient of variation of reproducibility, %	-1,05	-1,96	-1,08	-1,71	-1,92	-2,45	-1,70

^aGrand mean values were calculated using only sample data with outliers removed. Grand sample mean value and coefficient of variation were calculated by averaging. All other statistical means were calculated from the square root of the average of the square deviation.

COLLABORATIVE STUDIES ORGANIZED TO INCLUDE SHEEP AND GOAT MILK
IN THE SCOPE OF JOINT STANDARD ISO 5764|IDF 108: 2002 MILK – DETER-
MINATION OF FREEZING POINT – THERMISTOR CRYOSCOPE METHOD (REF-
ERENCE METHOD)

ABSTRACT

Bulletin presenting the collaborative studies organized to include sheep and goat milk in the scope of joint standard ISO 5764|IDF 108: 2002 Milk – Determination of freezing point – Thermistor cryoscope method (Reference method).

Keywords: : analysis, ewe, freezing point, goat, milk, precision, repeatability, reproducibility, sampling, sheep, standard, thermistor cryoscope

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e.g., i.e.,...Spelled out in English - for example, that is
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