CECALAIT-S NEWSLETTER

ACTALIA
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ACTALIA Cecalait

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ARTICLE

EVALUATION OF THE PERKINELMER LACTOSCOPE™ 300 FT-IR INFRARED ANALYSER

The LactoScope™ 300 is an FTIR infrared analyser (400-4000 cm^{-1}) (interféromètre Dynascan™) manufactured and commercialised by PerkinElmer. It is used for the determination of the composition components (fat, protein, lactose, dry matter) in liquid dairy products as milk, cream and whey.
The aparatus is connected to a computer with a touch screen. All the operations (analysis, cleaning, calibration) are carried out through the ResultsPlus™ program. The modification of the prediction models can be done by adjusting the slope and/or the bias, the calculations are done directly by the software. The « zero » measurement is carried out automatically every hour, and the cleaning is automatic and programmable. Results can be exported in different file formats (.pdf; .xls; .csv).

The instrument used in this study was:
- LactoScope™ 300
- Serial number: 113207
- ResultsPlus™ version 3.20.21643.0

The instrument was installed by PerkinElmer in a temperature controlled room (20-23 °C – air conditioning), without direct sunlight.

Due to an apparatus problem, the tests on the cow milk matrix were carried out on a second apparatus after checking the short-term stability.
The characteristics of this instrument were:
- LactoScope™ 300
- Serial number: 300028

A cleaning solution [40 ml of Cleaning Solution GA00071042 in 1l of demineralised water (conductivity < 5 µS/cm), conservation 4 weeks at room temperature] and a zero solution [30 ml of ZERO standard GA00271012 in 1l of demineralised water (conductivity < 5 µS/cm), conservation 2 weeks at room temperature] are required.

THE TESTS

The evaluation tests were performed in ACTALIA Cecalait's physico-chemistry laboratory from April to June 2022. After preliminary tests of stability, the repeatability and accuracy on tank raw cow milk, tank raw goat milk, cream, whey and milk retentate for fat, true protein, dry matter and total nitrogen were evaluated.

The accuracy of the instrument was evaluated according to the following standardised methods:
- Fat in milk: Gerber method according to ISO 19662|IDF 238
- Fat in goat milk: Gerber method according to ISO 19662|IDF 238
- Fat in cream: Röse-Gottlieb method according to ISO 2450|IDF 16
- Fat in whey : Röse-Gottlieb method according to ISO 1211|IDF 1
- Protein in milk: Amido black method according to NF V04-216
- Total nitrogen: Kjeldahl method according to ISO 8968-1|IDF 20-1
- Dry matter: Oven method according to ISO 6731|IDF 21

ARTICLE

1. EVALUATION OF THE SHORT-TERM STABILITY

The short-term stability was by analysing 3 samples of raw milk with preservative (Bronopol 0.02 % final), with different concentration levels of fat and protein, in triplicate, every 15-20 minutes to obtain at least 20 sequences.

To evaluate the stability of the instrument, the repeatability and reproducibility were calculated by level.

	Level 1	Level 2	Level 3
Fat (g/l)	38	53	75
Protein (g/l)	32	40	55

Table 1: Content of the samples used for the short-term stability evaluation

The following table presents the results obtained:

		M	Sr	Sr(%)	SR	SR(%)	r	R
Fat (g/kg)	**Level 1**	37.302	0.1340	0.359%	0.1763	0.473%	0.371	0.488
	Level 2	53.058	0.0829	0.156%	0.1869	0.352%	0.230	0.518
	Level 3	73.872	0.0825	0.112%	0.2353	0.319%	0.228	0.652
Protein (g/kg)	**Level 1**	32.731	0.0444	0.136%	0.1453	0.444%	0.123	0.403
	Level 2	40.043	0.0730	0.182%	0.1604	0.401%	0.202	0.444
	Level 3	56.165	0.1281	0.228%	0.2020	0.360%	0.355	0.559
Dry matter (g/kg)	**Level 1**	124.347	0.2371	0.191%	0.3815	0.307%	0.657	1.057
	Level 2	147.864	0.2003	0.135%	0.3844	0.260%	0.555	1.065
	Level 3	185.287	0.2348	0.127%	0.4308	0.233%	0.650	1.193

Table 2: LactoScope™ 300 stability criteria for fat, protein and dry matter[1]

The results for levels 1 and 2 indicate that the standard deviations of repeatability for fat and protein are below the limits required in ISO 8196-3|IDF 128-3 standard for milk with an average fat and protein content (0.28 g/kg). For milk with a high fat and protein content (level 3), the results indicate that the standard deviations of repeatability for fat and protein are also below the limits required in ISO 8196-3|IDF 128-3 standard for milk with a high fat and protein content (0.56 g/kg).

As no standardised value exists for dry matter, it can be noted that the reproducibility of the instrument (R) is lower than the reproducibility of the reference value (2.00 g/kg).

Following a instrument problem, repeatability and accuracy on the cow's milk matrix were evaluated on the instrument No. 300028 The short-term stability of the apparatus used for the cow's milk evaluation was verified under the same conditions as for the instrument above. The results obtained are of the same order and comply with the requirements of standard ISO 8196-3IDF 128-3.

2. EVALUATION OF THE REPEATABILITY

The repeatability of the instrument was performed by analysing:

- for tank raw cow milk: 45 samples of tank raw milk from a French plant (West of France).
- for goat milk: 33 samples of goat milk from a French plant (South-West of France).
- for cream: 22 samples of cream from a French plant (West of France)
- for whey: 22 samples of whey from a 3 cheese factories (East of France).
- for retentate: 23 skim milk protein retentate samples. The samples were reconstituted from 5 samples of retentate from a dairy plant in the west of France and skimmed milk in order to obtain a range of 100 to 150 g/kg of dry matter.

Bronopol was added to the samples to give a final concentration of 0.02 %. They were analysed (after heating at 40 ± 2 °C and 37 ± 2 °C for cream) in consecutive duplicate.

For raw cow milk and goat milk, the instrument was precalibrated using ACTALIA Cecalait's mid-infrared median range standard reference materials (SRM (ETG 04 LMIR). For the other matrices, the samples were analysed without prior adjustment of the manufacturer's prediction model (slope at 1 and bias at 0). Repeatability is calculated from duplicate results obtained from the complete data set or after elimination of outliers (Cochran test at 5% threshold) for the criteria fat, protein and dry matter.

[1] M: mean; S_r and S_R (S_r% and S_R%): absolute (and relative) standard deviation of repeatability and reproductibility; r and R: maximum deviation of repeatability and reproductibility in 95 % of cases.

The following table presents the results obtained:

		n	min	max	M	S_r	S_r%	r
Tank raw cow milk	**Fat (g/kg)**	45	28.80	45.20	42.08	0.067	0.16%	0.185
	Protein (g/kg)	45	32.60	35.00	33.72	0.078	0.23%	0.217
	Dry matter (g/kg)	45	119.60	137.20	133.68	0.515	0.39%	1.427
Goat milk	**Fat (g/kg)**	32	35.70	41.40	38.69	0.139	0.36%	0.386
	Protein (g/kg)	33	31.90	35.00	33.37	0.059	0.18%	0.164
	Dry matter (g/kg)	33	118.30	126.50	122.64	0.267	0.22%	0.740
Cream	**Fat (g/kg)**	21	401.70	410.20	404.40	0.440	0.11%	1.218
	Dry matter (g/kg)	22	453.00	471.90	466.83	0.673	0.14%	1.863
Whey	**Fat (g/kg)**	22	1.10	8.00	5.00	0.080	1.60%	0.221
	Total nitrogen (g/kg)	22	8.40	12.70	10.10	0.161	1.59%	0.446
	Dry matter (g/kg)	21	56.70	71.30	66.53	0.389	0.58%	1.076
Retentate	**Total nitrogen (g/kg)**	23	50.50	101.90	81.17	0.181	0.22%	0.502
	Dry matter (g/kg)	23	103.10	151.30	132.24	0.354	0.27%	0.979

Table 3: LactoScope™ 300 repeatability criteria for fat, protein, dry matter and total nitrogen in tank raw cow milk, goat milk, cream, whey and retentate samples[2]

It can be noted that:

- for tank raw cow milk: for fat and true protein content, the standard deviations of repeatability are lower than the requirements of the ISO 8196-3|IDF 128-3 standard (Sr < 0.14 g/kg). For dry matter, the standard deviation of repeatability is higher that the results for the other components and higher than the repeatability standard deviation of the reference method (Sr = 0.36 g/kg).

- for goat milk: for fat and true protein content, the standard deviations of repeatability are lower than the requirements of the ISO 8196-3|IDF 128-3 standard (Sr < 0.14 g/kg). For dry matter, the standard deviation of repeatability is higher that the results for the other components and lower than the repeatability standard deviation of the reference method (Sr = 0.36 g/kg).

- for cream: as no standardised value exists for cream, it can be noted that the standard deviations of repeatability for fat and dry matter obtained at with the instrument are lower than the standard deviations obtained with the reference methods: Sr = 0.44 g/kg vs 0.72 g/kg for fat; and 0.67 g/kg vs 0.72 g/kg for dry matter.

- for whey: as no standardised value exists for whey, it can be noted that the standard deviations of repeatability for fat and total nitrogen obtained with the instrument are lower than the standard deviations obtained with the reference methods: Sr = 0.080 g/kg vs 0.13 g/kg for fat; and 0.161 g/kg vs 0.18 g/kg for total nitrogen. For dry matter, the standard deviation obtained with the instrument is close to the standard deviation obtained with the reference method (Sr = 0.389 g/kg vs 0.36 g/kg).

- for retentate: as no standardised value exists for retentate, it can be noted that the standard deviation of repeatability for total nitrogen obtained with the instrument is équivalent to the standard deviation obtained with the reference method (Sr = 0.181 g/kg vs 0.18 g/kg). For dry matter, the standard deviation obtained with the instrument is lower than the standard deviation obtained with the reference method (Sr = 0.354 g/kg vs 1.08 g/kg).

3. EVALUATION OF THE ACCURACY

The accuracy of the instrument was evaluated by using the same samples than those used for the repeatablity evaluation. The mean of the duplicates of the results obtained in the repeatability evaluation was used for the calculation of the results. Outliers samples (samples whose regression residues are greater than 2 times the standard deviation of deviations: P at 5 %) have been discarded.

The following table presents the results obtained:

		n	min	max	X	Sx	d	Sd	Sy,x	Sy,x%	b	a
Tank raw cow milk	**Fat (g/kg)**	43	28.80	45.20	42.03	2.32	1.083	0.279	0.280	0.68%	0.984	-0.408
	Protein (g/kg)	43	32.60	34.90	33.74	0.51	1.314	0.269	0.229	0.71%	0.715	8.305
	Dry matter (g/kg)	45	119.70	137.10	133.68	2.57	3.229	0.417	0.378	0.29%	0.928	6.415

[2] N: number of the results; min and max: minimum and maximum values; M: mean of the results; Sr (Sr%): absolute (and relative) standard deviation; r: maximum deviation of repeatability in 95 % of cases

Goat milk	**Fat (g/kg)**	30	35.75	41.40	38.66	1.24	1.672	0.332	0.337	0.91%	0.984	-1.053
	Protein (g/kg)	29	31.95	35.00	33.44	0.80	1.378	0.152	0.100	0.31%	0.855	3.464
	Dry matter (g/kg)	32	118.35	126.45	122.70	2.00	-0.689	0.457	0.356	0.29%	0.853	18.729
Cream	**Fat (g/kg)**	19	402.00	410.20	404.27	2.28	-21.474	3.412	3.135	0.74%	0.325	294.323
	Dry matter (g/kg	20	454.20	470.20	466.56	3.18	-11.503	3.534	1.361	0.28%	-0.029	491.722
Whey	**Fat (g/kg)**	21	1.10	7.15	4.86	1.71	0.445	0.597	0.605	13.72%	0.920	-0.055
	Total nitrogen (g/kg)	20	8.60	12.40	10.27	1.18	0.497	0.597	0.467	4.78%	0.670	2.889
	Dry matter (g/kg)	19	56.80	70.75	66.23	4.19	-3.154	0.950	0.678	0.98%	0.836	13.991
Retentate	**Total nitrogen (g/kg)**	20	50.55	101.65	83.46	15.84	4.158	1.164	0.336	0.42%	0.929	1.728
	Dry matter (g/kg)	22	103.40	150.85	132.99	14.99	-3.561	0.513	0.505	0.37%	1.009	2.299

Table 4: LactoScope™ 300 accuracy criteria for fat, protein, dry matter and total nitrogen in tank raw cow milk, goat milk, cream, whey and retentate samples [3]

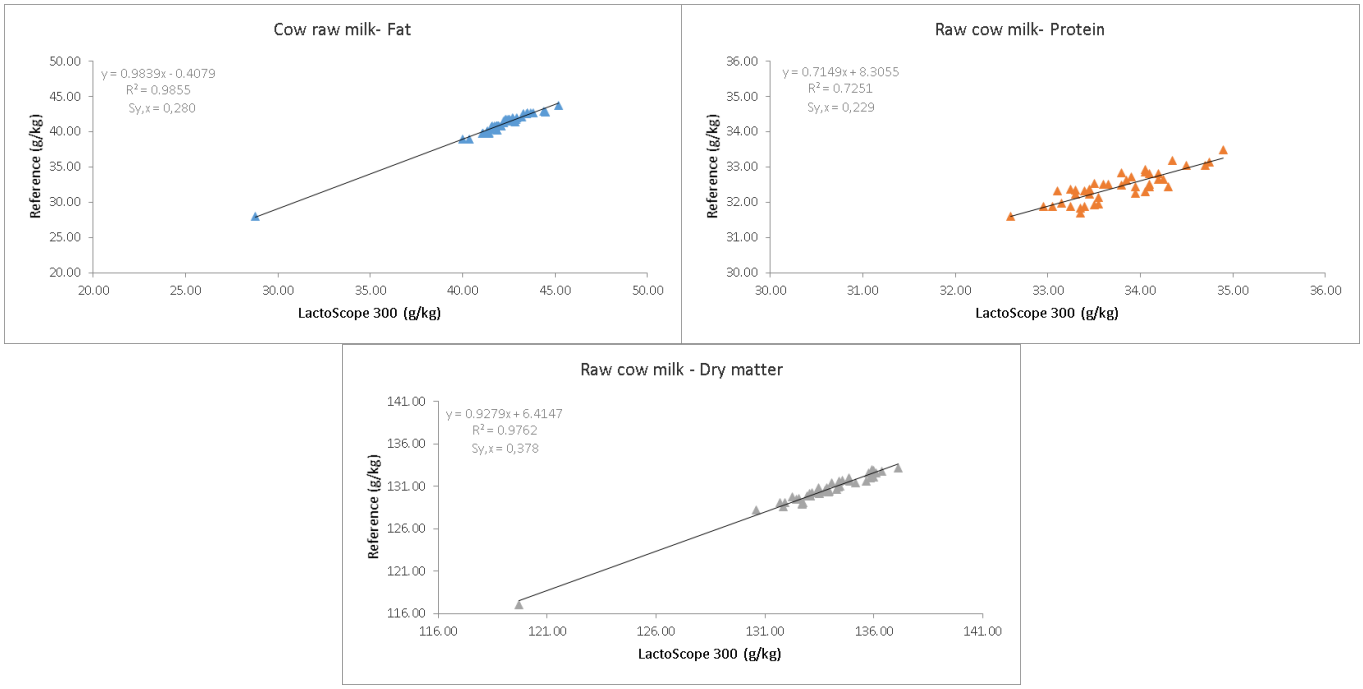
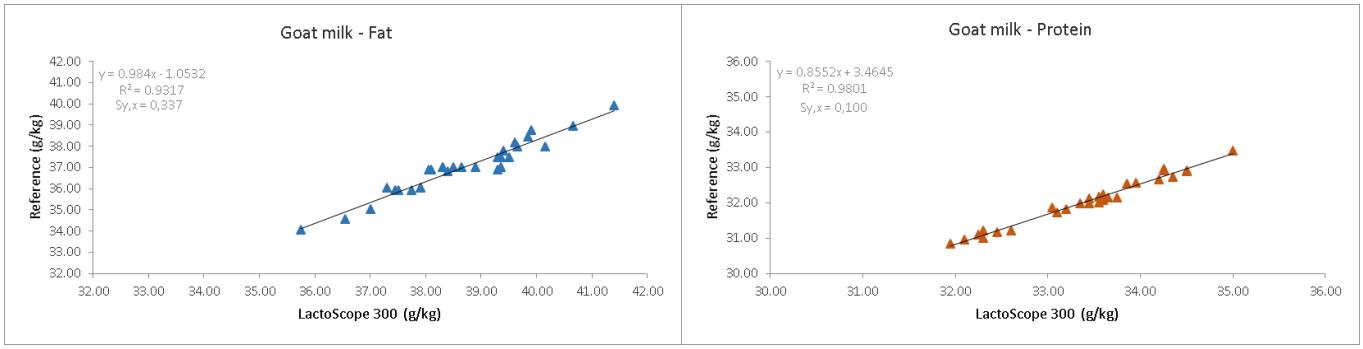


Figure 1: Relation between LactoScope™ 300 and reference results for fat, protein and dry matter in raw cow milk



[3] n, min, max: number of results, minimum and maximum values ; Y: mean of the results using the reference method ; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x (Sy,x%): absolute (and relative) residual standard deviation; b,a: slope and intercept of the linear regression

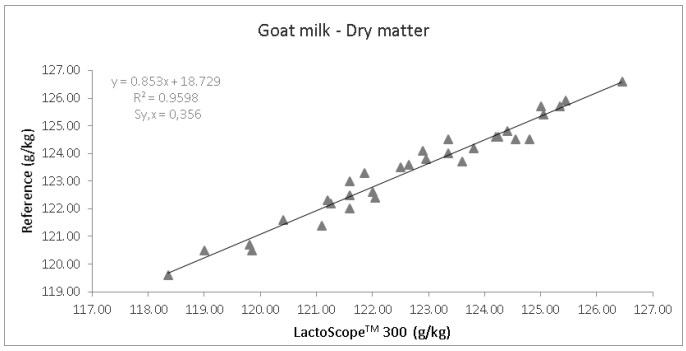


Figure 2: Relation between LactoScope™ 300 and reference results for fat, protein and dry matter in goat milk

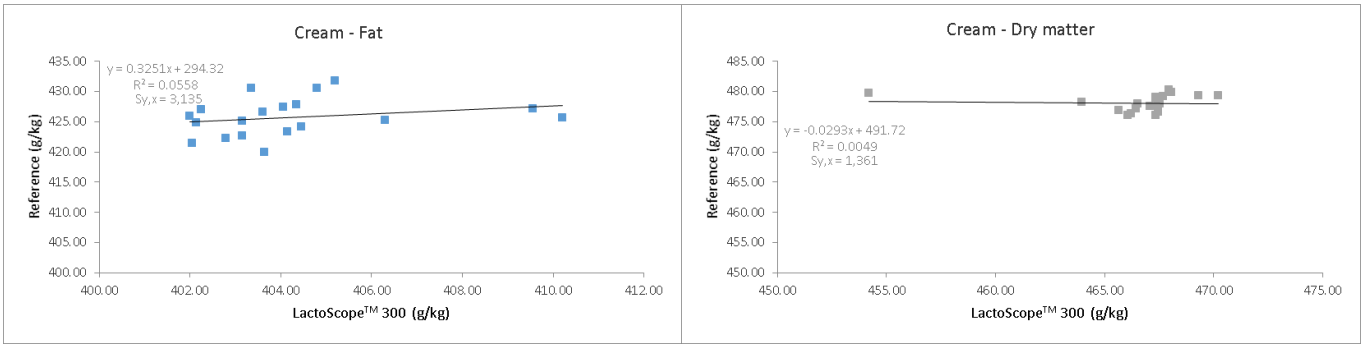


Figure 3: Relation between LactoScope™ 300 and reference results for fat and dry matter in cream

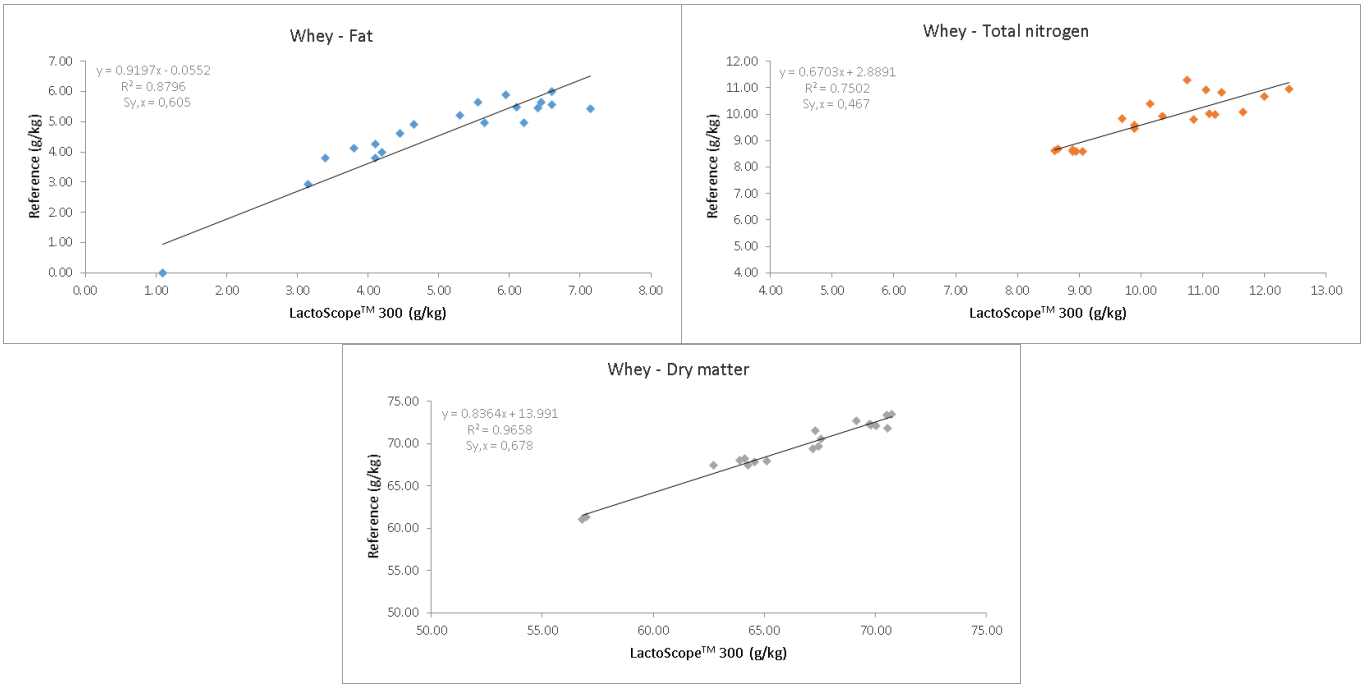


Figure 4: Relation between LactoScope™ 300 and reference results for fat, total nitrogen and dry matter in whey

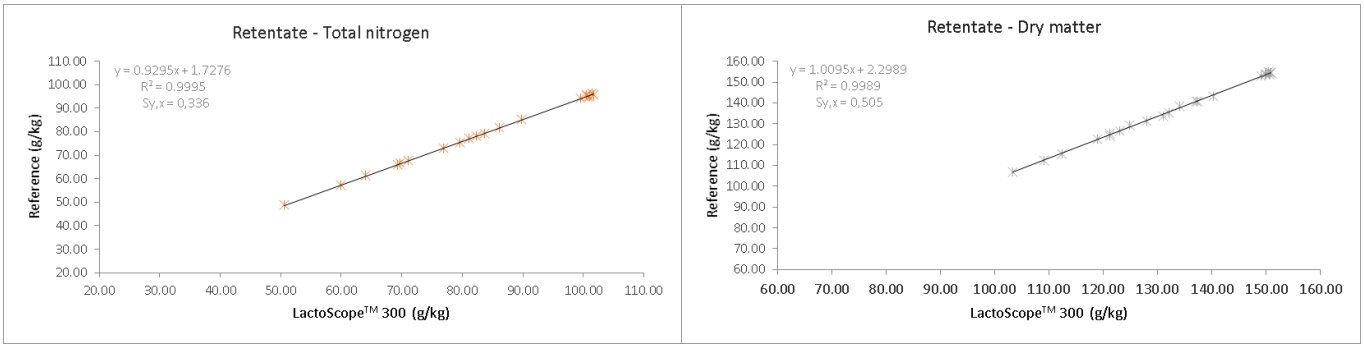


Figure 5: Relation between LactoScope™ 300 and reference results for total nitrogen and dry matter in retentate

Concerning the relation between LactoScope™ 300 and reference method results, it can be noted that:

- for tank raw cow milk:

The residual standard deviations of linear regression obtained are equal to 0.280 g of fat/kg, 0.229 g of protein/kg, and 0.378 g of dry matter/kg. The estimation precision of the instrument is therefore ± 0.56 g/kg (± 2 x 0.280 g/kg) for fat, ± 0.46 g/kg (± 2 x 0.229 g/kg) for protein, and ± 0.76 g/kg (± 2 x 0.378 g/kg) for dry matter.

- for goat milk:

The residual standard deviations of linear regression obtained are equal to 0.337 g of fat/kg, 0.100 g of protein/kg, and 0.356 g of dry matter/kg. The estimation precision of the instrument is therefore ± 0.67 g/kg (± 2 x 0.337 g/kg) for fat, ± 0.20 g/kg (± 2 x 0.100 g/kg) for protein, and ± 0.712 g/kg (± 2 x 0.356 g/kg) for dry matter.

- for cream:

The residual standard deviations of linear regression obtained are equal to 3.135 g of fat/kg and 1.361 g of dry matter/kg. The estimation precision of the instrument is therefore ± 6.27 g/kg (± 2 x 3.135 g/kg) for fat, and ± 2.72 g/kg (± 2 x 1.361 g/kg) for dry matter.

- for whey:

The residual standard deviations of linear regression obtained are equal to 0.605 g of fat/kg, 0.467 g of total nitrogen/kg, and 0.678 g of dry matter/kg. The estimation precision of the instrument is therefore ± 1.21 g/kg (± 2 x 0.605 g/kg) for fat, ± 0.934 g/100 g (± 2 x 0.467 g/kg) for total nitrogen, and ± 1.356 g/kg (± 2 x 0.678 g/kg) for dry matter.

- for retentate:

The residual standard deviations of linear regression obtained are equal to 0.336 g of total nitrogen/kg, and 0.505 g of dry matter/kg. The estimation precision of the instrument is therefore ± 0.672 g/kg (± 2 x 0.336 g/kg) for total nitrogen, and ± 1.01 g/kg (± 2 x 0.505 g/kg) for dry matter.

4. CONCLUSION

We can conclude that the short-term stability of the instrument is in accordance with the requirements of the ISO 8196-3|IDF 128-3 standard.

Concerning the repeatability of the instrument, the results for fat and protein in milk are in conformity with limits of the ISO 9622IDF 141 standard. For the other products and criteria, the results obtained are in accordance with the recommendations of the ISO 8196-3|IDF 128-3 standard, or lower or near to the repeatability limits of the corresponding reference method.

Concerning the precision, no standardised requirements exist for the products tested during this evaluation (tank raw milk, goat milk, cream, whey and milk retentate).

According to the evaluation report of the LactoScope™ 300 – M. ESTEVES, A. OUDOTTE et Ph. TROSSAT – April-June2022

STANDARDS, DRAFT STANDARDS

ISO standards under development

MICROBIOLOGY	
ISO/DIS 7581 November 2022	Method for the evaluation of basic bactericidal activity of a non-porous surface
ISO/DIS 7251/DAmd 1 November 2022	MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS Horizontal method for the detection and enumeration of presumptive *Escherichia coli*- Most probable number technique – Amendment 1: Inclusion of performance testing of culture media and reagents
ISO/DIS 15213-2 December 2022	MICROBIOLOGY OF THE FOOD CHAIN Horizontal method for the detection and enumeration of Clostridium spp. – Part 2: Enumeration of Clostridium perfringens by colony-count technique
ISO/DIS 17468 December 2022	MICROBIOLOGY OF THE FOOD CHAIN Technical requirements and guidance on establishment or revision of a standardized reference method
VITAMINS	
ISO/DIS 20631 November 2022	INFANT FORMULA AND ADULT NUTRITIONALS Determination of total folates content by trienzyme extraction and ultra-performance liquid chromatography tandem mass spectrometry (UPLC-MS/MS)

ISO published standards

SAMPLING	
ISO 3951-1 August 2022	SAMPLING PROCEDURES FOR INSPECTION BY VARIABLES Part 1: Specification for single sampling plans indexed by acceptance quality limit (AQL) for lot-by-lot inspection for a single quality characteristic and a single AQL *Replace ISO 3951-1:2014*
METROLOGY	
ISO 23783-2 August 2022	AUTOMATED LIQUID HANDLING SYSTEMS Part 2: Measurement procedures for the determination of volumetric performance
ISO 23783-3 August 2022	AUTOMATED LIQUID HANDLING SYSTEMS Part 3: Determination, specification and reporting of volumetric performance *Replace IWA 15:2015*
ISO 24185 August 2022	Evaluation of the uncertainty of measurements from a stationary autocorrelated process
STATISTICS	
ISO 10576 August 2022	STATISTICAL METHODS Guidelines for the evaluation of conformity with specified requirements *Replace ISO 10576-1:2003*
ISO 13528 August 2022	STATISTICAL METHODS Statistical methods for use in proficiency testing by interlaboratory comparison *Replace ISO 13528:2015*

NEW EU REGULATIONS

CONTAMINANTS

O.J.E.U. L 221, 26th August 2022 – Commission Implementing Regulation (EU) 2022/1428 of 24 August 2022 laying down methods of sampling and analysis for the control of perfluoroalkyl substances in certain foodstuffs
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2022.221.01.0066.01.ENG

O.J.E.U. L 221, 26th August 2022 – Commission Recommendation (EU) 2022/1431 of 24 August 2022on the monitoring of perfluoroalkyl substances in food
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2022.221.01.0105.01.ENG

P.D.O. / T.S.G.

O.J.E.U. L 196, 25th July 2022 – Commission Implementing Regulation (EU) 2022/1291 of 22 July 2022 approving a non-minor amendment to the product specification for a name entered in the register of traditional specialities guaranteed [Mozzarella (cheese) (TSG)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2022.196.01.0115.01.ENG

O.J.E.U. C 286, 27th July 2022 – Publication of the amended single document following the approval of a minor amendment pursuant to the second subparagraph of Article 53(2) of Regulation (EU) No 1151/2012 [Idiazabal (cheese) (PDO)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2022.286.01.0054.01.ENG

O.J.E.U. L 201, 1st August 2022 – Commission Implementing Regulation (EU) 2022/1332 of 26 July 2022 approving a non-minor amendment to the product specification for a name entered in the register of traditional specialities guaranteed [Beurre Charentes-Poitou / Beurre des Charentes / Beurre des Deux-Sèvres (butter) (PDO)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2022.201.01.0023.01.ENG

O.J.E.U. C 312, 17th August 2022 – Publication of the amended single document following the approval of a minor amendment pursuant to the second subparagraph of Article 53(2) of Regulation (EU) No 1151/2012 [Rigotte de Condrieu (cheese) (PDO)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2022.312.01.0005.01.ENG

O.J.E.U. C 349, 12th September 2022 – Publication of an application for approval of a non-minor amendment to a product specification pursuant to Article 50(2)(a) of Regulation (EU) No 1151/2012 of the European Parliament and of the Council on quality schemes for agricultural products and foodstuffs [Salers (cheese) (PDO)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2022.349.01.0011.01.ENG

PESTICIDES

O.J.E.U. L 200, 29th July 2022 – Commission Regulation (EU) 2022/1321 of 25 July 2022 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for fluoride ion, oxyfluorfen, pyroxsulam, quinmerac and sulfuryl fluoride in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2022.200.01.0001.01.ENG

O.J.E.U. L 200, 29th July 2022 – Commission Regulation (EU) 2022/1324 of 28 July 2022 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for benzovindiflupyr, boscalid, fenazaquin, fluazifop-P, flupyradifurone, fluxapyroxad, fosetyl-Al, isofetamid, metaflumizone, pyraclostrobin, spirotetramat, thiabendazole and tolclofos-methyl in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2022.200.01.0068.01.ENG

O.J.E.U. L 202, 2nd August 2022 – Commission Regulation (EU) 2022/1343 of 29 July 2022 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for acequinocyl, chlorantraniliprole and emamectin in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2022.202.01.0001.01.ENG

O.J.E.U. L 202, 2nd August 2022 – Commission Regulation (EU) 2022/1346 of 1 August 2022 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for 1,4-dimethylnaphthalene, 8-hydroxyquinoline, pinoxaden and valifenalate in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2022.202.01.0031.01.ENG

O.J.E.U. L 205, 5th August 2022 – Commission Regulation (EU) 2022/1363 of 3 August 2022 amending Annex II to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for 2,4-D, azoxystrobin, cyhalofop-butyl, cymoxanil, fenhexamid, flazasulfuron, florasulam, fluroxypyr, iprovalicarb and silthiofam in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2022.205.01.0207.01.ENG

O.J.E.U. L 215, 18th August 2022 – Commission Regulation (EU) 2022/1406 of 3 August 2022 amending Annex II, III and V to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for methoxyfenozide, propoxur, spinosad and thiram in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2022.205.01.0001.01.ENG

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