CECALAIT'S NEWSLETTER

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EXPERTISE ET CONTROLE DES ANALYSES LAITIERES

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BEST WISHES FOR 2010 FROM ALL THE TEAM AT ACTILAIT 39

ARTICLE

EVALUATION OF THE ADVANCED LACTOSCOPE FTIR™ ANALYSER ON A CHEESE MATRIX

The Lactoscope FTIR™ analyser was evaluated by Actilait-Cecalait for the determination of fat, protein, dry matter and the freezing point in milk, and the fat content in cream. The results are presented in Cecalait's newsletter n°70. The present evaluation concerns the determination of fat, dry matter and total nitrogen in cheese.

The Lactoscope FTIR™ analyser, manufactured by Delta Instruments (Netherlands, Advanced group) and commercialized in France by Humeau Laboratories, is a mid infrared spectrophotometer used for the determination of the principal components (fat, protein, dry matter, freezing point and total nitrogen content) in milk, cream and cheese.

This instrument uses a mono-bundle Fourier transform-based infrared system (FTIR). The apparatus is connected to a computer that ensures the running and the signal treatment. Two mathematical calculations can be carried out: traditional PLS (MLR) and PLS.

Consumables:

The consumables used were:
- "Zero" solution: water + 0.1% triton
- Clean solution: Decon 90™ 4% aqueous solution

The tests:

The evaluation tests were performed in Actilait-Cecalait's physico-chemistry laboratory (reference and infrared analyses) in August and September 2009. The tests concerned the evaluation of repeatability and accuracy for the determination of fat, dry matter and total nitrogen in cheese.

The appreciation criteria of the estimated parameters were taken from ISO 9622 / IDF 141C:2000 "Guide for the operation of mid-infrared instruments", or from the CNIEL/IE handbook concerning the use of infrared apparatus within the context of milk payment and milk control in France.

The following instrumental parameters were used:
- Manual tests at a rate of about 120 samples / hour;
- No correction of contamination;
- MLR calibration (fat and total nitrogen);
- PLS calibration (dry matter).

The samples

The tests were performed on samples of cheese sold in supermarkets and hypermarkets. 20 samples were analysed: 6 samples of "fromage frais" (3 as is from the shops and 3 made up artificially), 7 samples of soft cheese and 7 samples of hard cheese. Bronopol was added to the "fromage frais" samples to give a final concentration of 0.02%. The samples were prepared in accordance with the manufacturer's recommendations: remove the rind and grind the test sample, make a 10% suspension with the "cheese" reagent and grind for one minute in a granulating mill.

ARTICLE

1- EVALUATION OF REPEATABILITY

1.1- Procedure

The repeatability of the instrument for fat, dry matter and protein was evaluated using all the cheese samples. The quantitative analyses were performed in consecutive duplicate (one suspension per sample).

1.2- Results

The results obtained are presented in the table below:

	n	min	max	M	Sx	Sr	Sr (%)	r
Fat (g/100g)	20	0.10	35.58	20.21	12.40	0.04	0.18	0.10
Dry matter (g/100g)	20	14.26	67.47	43.98	19.35	0.07	0.15	0.18
Total nitrogen (g/100g)	20	7.42	32.76	19.30	8.34	0.04	0.21	0.11

Table 1: repeatability criteria FTIR Lactoscope for fat, dry matter and total nitrogen in cheese samples

n: number of results; min and max: minimum and maximum values, M and Sx: mean and standard deviation of the results; Sr and Sr%: absolute and relative standard deviation of repeatability; r: maximum deviation of repeatability in 95% of cases.

1.3- Conclusion

With no standard criteria or recommendations from the manufacturer for fat, dry matter and total nitrogen content, the FTIR Lactoscope presents a standard deviation of repeatability lower than the maximum acceptable limit laid down in the ISO 1735, ISO 5534 and ISO 8968/IDF 20 (WD 2008) reference methods, equal to 0.07 g/100g, 0.13 g/100g and 0.05 g/100g respectively.

2- EVALUATION OF ACCURACY

2.1- Procedure

The accuracy of the analyser for fat, dry matter and protein was evaluated using all the cheese samples. The quantitative analyses were performed in compliance with the evaluation of repeatability (cf. 1.2). The instrumental values are from a calibration carried out by the manufacturer.

The following reference methods were used:
- Fat using SBR extraction method according to ISO 1735 (single test);
- Dry matter using drying method according to ISO 5534 (single test);
- Total nitrogen using Kjeldahl method according to ISO 8968 (single test). Conversion using the following calculation: MAT = NT x 6.38

2.2- Results

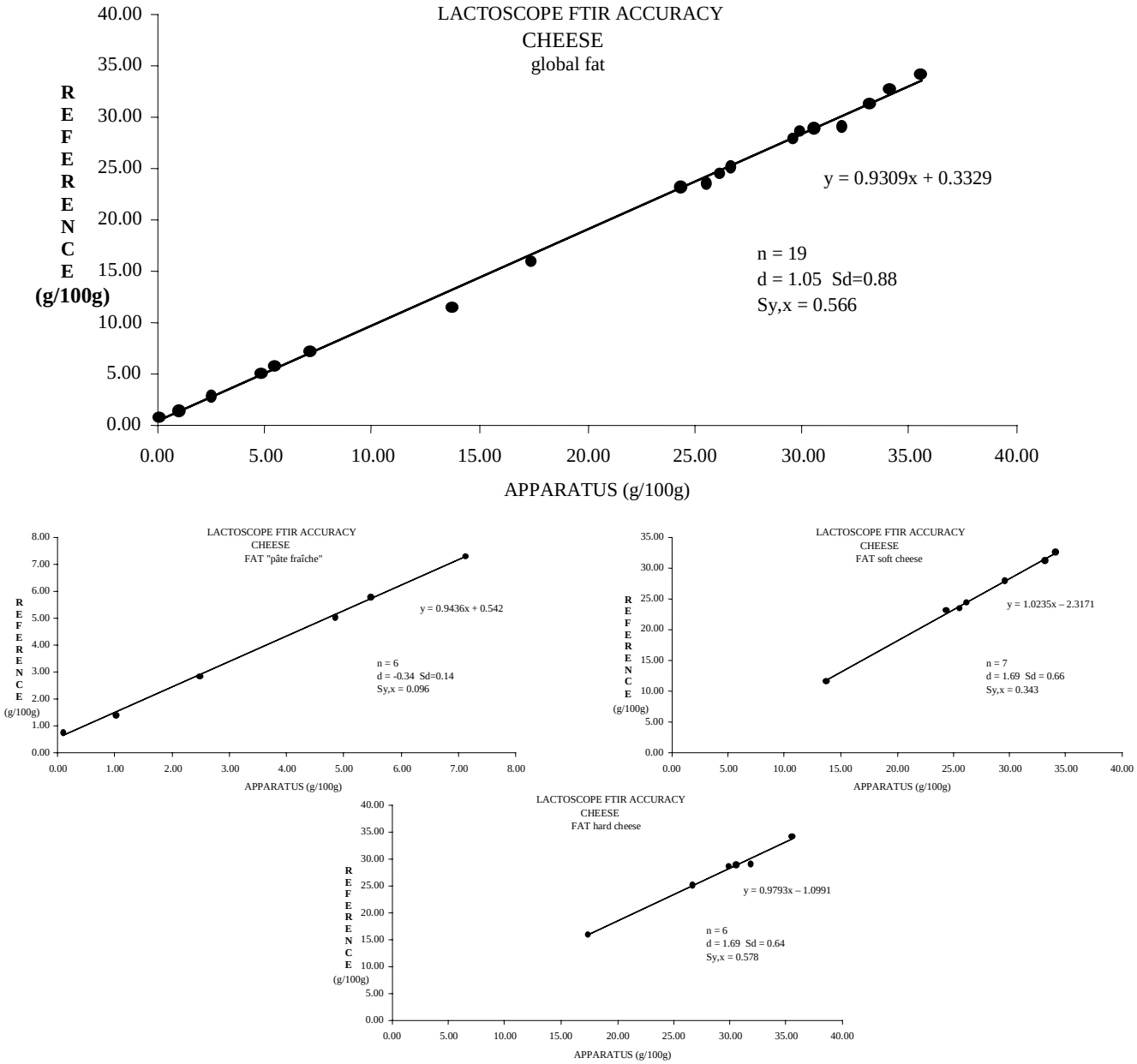
2.2.1- Fat

The results obtained are presented in the following tables and figures.

	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x %	b	a
Global Fat (g/100g)	19	0.75	34.08	18.93	11.83	1.05	0.88	0.566	3.0	0.931	0.33
Fat (g/100g) "Pâte fraîche"	6	0.75	7.29	3.85	2.59	-0.34	0.14	0.096	2.5	0.944	0.54
Fat (g/100g) Soft cheese	7	11.59	32.68	24.96	6.99	1.69	0.66	0.343	1.4	1.024	-2.32
Fat (g/100g) Hard cheese	6	15.91	34.08	26.97	6.12	1.69	0.64	0.578	2.1	0.979	-1.10

Table 2: Accuracy criteria of the FTIR Lactoscope for fat in cheese samples

n, min, max: number of results, minimum and maximum values; Y: mean results using the reference; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation; b, a: slope and intercept of the linear regression.



Figures 1, 2, 3 and 4: Relationship between the FTIR Lactoscope and the reference results for fat in "pâte fraîche", soft and hard cheese samples

It can be noted that:
- Overall, the mean and standard deviation of deviations are respectively 1.05 and 0.88 g/100g. The regression slope obtained is significantly different from 1 (P = 1%) and the intercept is significantly different from zero (P = 1%). The residual standard deviation of regression is equal to 0.566 g/100g.
- the slope varies between 0.94 and 1.02 according to the type of cheese, which indicates the necessity for specific adjustments.

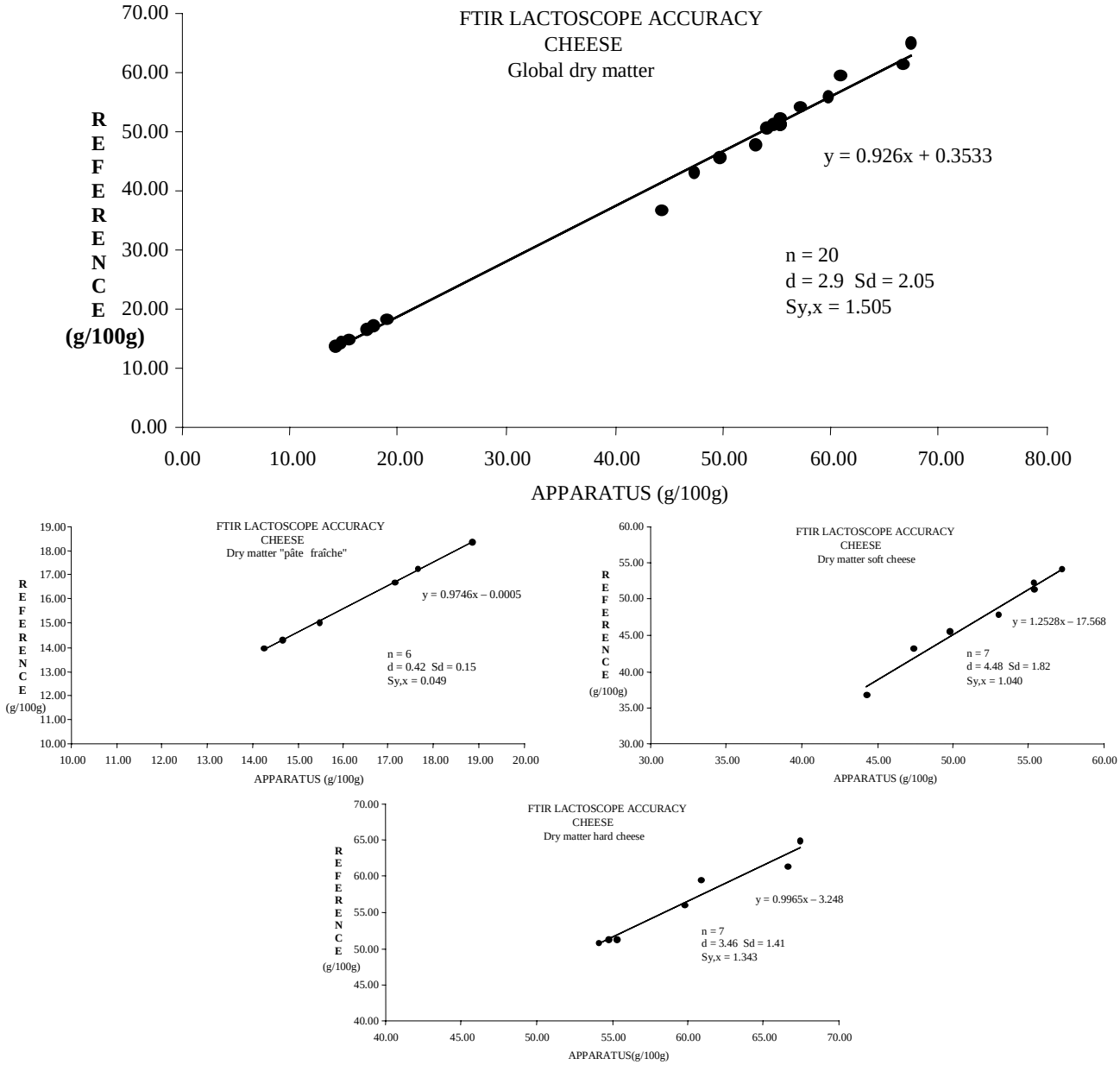
2.2.2- Dry matter

The results obtained are presented in the following tables and figures.

	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x %	b	a
Global dry matter (g/100g)	20	13.94	64.96	41.08	17.98	2.90	2.05	1.505	3.7	0.926	0.35
Dry matter (g/100g) "Pâte fraîche"	6	13.94	18.37	15.92	1.77	0.42	0.15	0.049	0.3	0.975	0.00
Dry matter (g/100g) Soft cheese	7	36.76	54.20	47.31	6.05	4.48	1.82	1.040	2.2	1.253	-17.57
Dry matter (g/100g) Hard cheese	7	50.77	64.96	56.42	5.65	3.46	1.41	1.343	2.4	0.997	-3.25

Table 3: accuracy criteria of the FTIR Lactoscope for dry matter in cheese samples

n, min, max: number of results, minimum and maximum values; Y: mean results using the reference; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation; b, a: slope and intercept of the linear regression.



Figures 5, 6, 7 and 8: Relationship between the FTIR Lactoscope and the reference results for dry matter in "pâte fraîche", soft and hard cheese samples

It can be noted that:
- Overall, the mean and standard deviation of deviations are respectively 2.9 and 2.05 g/100g. The regression slope obtained is significantly different from 1 (P = 1%) and the intercept is significantly different from zero (P = 1%). The residual standard deviation is 1.505 g/100g.
- Concerning fat, the slope varies between 0.97 and 1.25 according to the type of cheese, which indicates the necessity for specific adjustments.

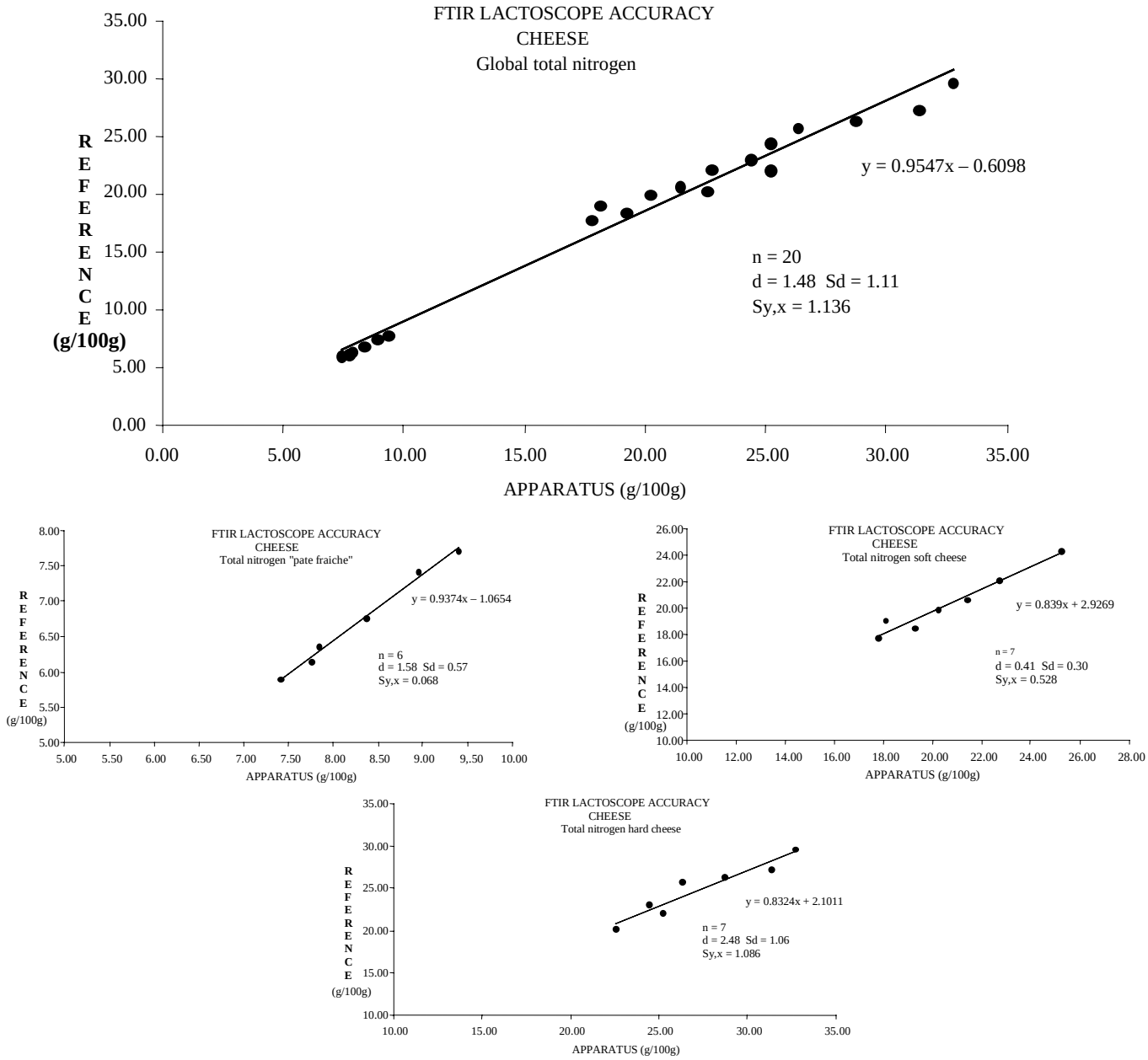
2.2.3- Total nitrogen

The results obtained are presented in the following tables and figures.

	n	min	max	Y	Sy	d	Sd	Sy,x	Sy,x%	b	a
Global total nitrogen (g/100g)	20	5.89	29.54	17.81	8.04	1.48	1.11	1.136	6.4	0.955	-0.61
Total nitrogen (g/100g) "Pâte fraîche"	6	5.89	7.70	6.71	0.72	1.58	0.57	0.068	1.0	0.937	-1.07
Total nitrogen (g/100g) soft cheese	7	17.69	24.30	20.29	2.28	0.41	0.30	0.528	2.6	0.839	2.93
Total nitrogen (g/100g) hard cheese	7	20.16	29.54	24.86	3.27	2.48	1.06	1.086	4.4	0.832	2.10

Table 4: accuracy criteria of the FTIR Lactoscope for total nitrogen in cheese samples

n, min, max: number of results, minimum and maximum values; Y: mean results using the reference; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x: residual standard deviation; b, a: slope and intercept of the linear regression.



Figures 9, 10, 11 and 12: Relationship between the FTIR Lactoscope and the reference results for total nitrogen in "pâte fraîche", soft and hard cheese samples.

It can be noted that:
- Overall, the mean and standard deviation of deviations are respectively 1.48 and 1.11 g/100g. The regression slope obtained is significantly different from 1 (P = 1%) and the intercept is significantly different from zero (P = 1%). The residual standard deviation of regression is 1.136 g/100g.
- Concerning fat and dry matter, the slope varies between 0.83 and 0.94 according to the type of cheese, which indicates the necessity for specific adjustments. However, the slopes are similar between soft and hard cheese.

2.3- Conclusion

Concerning fat, dry matter and protein, with no standard criteria or recommendations from the manufacturer, the results obtained should enable an accuracy of the overall estimation (in 95% of cases) of approximately +/- 1.1; 3.0 and 2.3 g/100g respectively after adjustment using representative samples. Specific adjustments according to the type of cheese improve the accuracy of the results.

From the evaluation report of the infrared Advanced LactoScope FTIR™ analyser - X. QUERVEL, Ph. TROSSAT – Actilait / Cecalait – November 2009

STANDARDS, DRAFT STANDARDS

Classification in alphabetic order by theme

ISO published standards

CHEESE		
NISIN A CONTENT	ISO/TS 27106:2009 December 2009	CHEESE Determination of nisin A content by LC-MS and LC-MS-MS
DRIED SKIMMED MILK		
VITAMIN A CONTENT	ISO 12080-1-2:2009 (IDF 142-1-2) September 2009	DRIED SKIMMED MILK Determination of vitamin A content Part 1: Colorimetric method Part 2: Method using high-performance liquid chromatography
INSTANT WHOLE MILK POWDER		
WHITE FLECKS	ISO 11865:2009 (IDF 174) September 2009	INSTANT WHOLE MILK POWDER Determination of white flecks number
MILK		
ALTERNATIVE METHODS	ISO 8196-1-2-3:2009 (IDF 128-1-2-3) October 2009	MILK Definition and evaluation of the overall accuracy of alternative methods of milk analysis Part 1: Analytical attributes of alternative methods Part 2: Calibration and quality control Part 3: Protocol for the evaluation and validation of alternative quantitative methods of milk analysis
MILK AND DRIED MILK		
IODIDE CONTENT	ISO 14378:2009 (IDF 167) September 2009	MILK AND DRIED MILK Determination of iodide content – Method using high-performance liquid chromatography
MILK AND MILK PRODUCTS		
FAT CONTENT	ISO 11870:2009 (IDF 152) September 2009	MILK AND MILK PRODUCTS Determination of fat content – General guidance on the use of butyrometric methods
HEN'S EGG WHITE LYSOZYME	ISO/TS 27105:2009 (December 2009	MILK AND MILK PRODUCTS Determination do hen's egg white lysozyme by HPLC
PESTICIDES	ISO 3890-1-2:2009 (IDF 75-1-2) October 2009	MILK AND MILK PRODUCTS Determination of residues of organochlorine compounds (pesticides) Part 1: General considerations and extraction methods Part 2: Test methods for crude extract purification and confirmation
MILKFAT PRODUCTS		
WATER CONTENT	ISO 5536:2009 (IDF 23) November 2009	MILKFAT PRODUCTS Determination of water content – Karl Fischer method
QUALITY		
REFERENCE MATERIAL	ISO GUIDE 34:2009 November 2009	General requirements for the competence of reference material producers

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

HEALTH CLAIMS

O.J.E.U. L 277, 22nd October 2009 – Commission Regulation (EC) n° 983/2009 of 21 October 2009 on the autorisation and refusal of autorisation of certain health claims made on food and refering to the reduction of disease risk and to children's development and health
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:277:0003:0012:EN:PDF

O.J.E.U. L 314, 1st December 2009 – Commission Regulation (EC) n° 1167/2009 of 30 November 2009 refusing to authorise certain health claims made on foods and referring to the reduction of disease risk and to children's development and health
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:314:0029:0031:EN:PDF

HEALTH RULES

O.J.E.U. L 300, 14th November 2009 – Regulation (EC) n° 1069/2009 of the European Parliament and of the Council of 21 October 2009 laying down health rules as regards animal by-products and derived products not intended for human consumption and repealing Regulation (EC) n° 1774/2002 (Animal by-products Regulation)
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:300:0001:0033:EN:PDF

HYGIENE

O.J.E.U. L 314, 1st December 2009 – Commission Regulation (EC) n° 1162/2009 of 30 November 2009 laying down transitional measures for the implementation of Regulations (EC) n° 853/2004, (EC) n° 854/2004 and (EC) n° 882/2004 of the European Parliament and of the Council
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:314:0010:0012:EN:PDF

DESIGNATION OF ORIGIN / TRADITIONAL SPECIALITIES GUARANTEED

O.J.E.U. C 238, 3rd October 2009 – Publication of an amendment application pursuant to Article 6(2) of Council Regulation (EC) n° 510/2006 on the protection of geographical indications and designations of origin for agricultural products and foodstuffs [Spressa delle Giudicarie (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2009:238:0014:0017:EN:PDF

O.J.E.U. L 274, 20th October 2009 – Commission Regulation (EC) n° 977/2009 of 19 October 2009 approving minor amendments to the specification of a name registered in the register of traditional specialities guaranteed [Boerenkaas (TSG) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:274:0019:0024:EN:PDF

O.J.E.U. L 282, 29th October 2009 – Commission Regulation (EC) n° 1019/2009 of 28 October 2009 approving minor amendments to the specification of a name registered in the register of protected designations of origin and protected geographical indications [Pouligny-Saint-Pierre (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:282:0003:0006:EN:PDF

O.J.E.U. C 260, 30th October 2009 – Publication of an application pursuant to Article 6(2) of Council Regulation (EC) n° 510/2006 on the protection of geographical indications and designations of origin for agricultural products and foodstuffs [Ricotta di Bufala Campana (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2009:260:0043:0046:EN:PDF

O.J.E.U. L 283, 30th October 2009 – Commission Regulation (EC) n° 1027/2009 of 29 October 2009 approving minor amendments to the specification of a name registered in the register of protected designations of origin and protected geographical indications [Morbier (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:283:0034:0037:EN:PDF

O.J.E.U. L 283, 30th October 2009 – Commission Regulation (EC) n° 1030/2009 of 29 October 2009 approving minor amendments to the specification of a name registered in the register of protected designations of origin and protected geographical indications [Pecorino Romano (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:283:0043:0046:EN:PDF

O.J.E.U. L 311, 26th November 2009 – Commission Regulation (EC) n° 1138/2009 of 25 November 2009 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Bitto (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:311:0023:0024:EN:PDF

STANDARDS - REGULATIONS

O.J.E.U. L 312, 27th November 2009 – Commission Regulation (EC) n° 1143/2009 of 26 November 2009 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Picodon de l'Ardèche or Picodon de la Drôme (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:312:0014:0015:EN:PDF

O.J.E.U. L 314, 1st December 2009 – Commission Regulation (EC) n° 1176/2009 of 30 November 2009 entering a name in the register of protected designations of origin and protected geographical indications [Redykolka (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:314:0062:0063:EN:PDF

O.J.E.U. L 317, 3rd December 2009 – Commission Regulation (EC) n° 1183/2009 of 30 November 2009 entering a name in the register of protected designations of origin and protected geographical indications [Formaggio di Fossa di Sogliano (PDO) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:317:0034:0035:EN:PDF

O.J.E.U. C 305, 16th December 2009 – Publication of an application for registration pursuant to Article 8(2) of Council Regulation (EC) n° 509/2006 on agricultural products and foodstuffs as traditional specialities guaranteed [Ovci Salasnicky Udeny Syr (STG) (cheese)]
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:C:2009:305:0027:0031:EN:PDF

PESTICIDES

O.J.E.U. L 290, 6th November 2009 – Commission Regulation (EC) n° 1050/2009 of 28 October 2009 amending Annexes II and III to Regulation (EC) n° 396/2005 of the European Parliament and of the Council as regards maximum residue levels for azoxystrobin, acetamipid, clomazone, cyflufenamid, emamectin benzoate, famoxadone, fenbutatin oxide, flufenoxuron, fluopicolide, indoxacarb, ioxynil, mepanipyrim, prothioconazole, pyridalyl, thiacloprid and trifloxystrobin in or on certain products
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:290:0007:0055:EN:PDF

O.J.E.U. L 301, 17th November 2009 – Commission Regulation (EC) n° 1097/2009 of 16 November 2009 amending Annex II to Regulation (EC) n° 396/2005 of the European Parliament and of the Council as regards maximum residue levels for dimethoate, ethephon, fenamiphos, fenarimol, methamidophos, methomyl, omethoate, oxydemeton-methyl, procymidone, thiodicarb and vinclozolin in or on certain products
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:301:0006:0022:EN:PDF

O.J.E.U. L 307, 21st November 2009 – Corrigendum to Commission Regulation (EC) n° 1097/2009 of 16 November 2009 amending Annex II to Regulation (EC) n° 396/2005 of the European Parliament and of the Council as regards maximum residue levels for dimethoate, ethephon, fenamiphos, fenarimol, methamidophos, methomyl, omethoate, oxydemeton-methyl, procymidone, thiodicarb and vinclozolin in or on certain products
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:307:0008:0008:EN:PDF

VETERINARY MEDICINAL PRODUCTS

O.J.E.U. L 304, 19th November 2009 – Corrigendum to Commission Regulation (EC) n° 508/1999 of 4 March 1999 amending Annexes I to IV of Council Regulation (EEC) n° 2377/90 laying down a Community procedure for the establishment of maximum residue limits of veterinary medicinal products in foodstuffs of animal origin
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:304:0024:0024:EN:PDF

VITAMINS AND MINERALS

O.J.E.U. L 314, 1st December 2009 – Commission Regulation (EC) n° 1170/2009 of 30 November 2009 amending Directive 2002/46/EC of the European Parliament and of the Council and Regulation (EC) n° 1925/2006 of the European Parliament and of the Council as regards the lists of vitamins and minerals and their forms that can be added to ffods, including food supplements
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2009:314:0036:0042:EN:PDF

BOOKSHOP: LATEST PUBLICATIONS

The classification in alphabetic order of the first keyword allows you to consult the references according to your interests. The web site allows you to know more, or to order the book.

ANALYSES

NOLLET L.M.L.; TOLDRA F. **– Handbook of dairy foods analysis** –CRC Press Editions – 2009 – ISBN 9781420046311 – 918 pages

http://www/crcpress.com

This handbook compiles the most used dairy analytical techniques and methodologies in the world. It details these methods, covers a large range of products, presents the tools for analysing chemical and biochemical compounds, discusses current methods for the detection of microorganisms, allergens…, and proposes rheological methods.

PATHOGENES

LEVIN R.E. **– Rapid detection and characterization of foodborne pathogens by molecular techniques** –CRC Press Editions – 2009 – ISBN 9781420092424 – 608 pages

http://www/crcpress.com

This book describes molecular techniques for the detection and enumration of the major pathogens. Each chapter deals with a pathogen and the technique applied to it, with a particular focus on the PCR technique and its limits with certain pathogens.

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

ADULTERATION

Testing software aids food safety screening

http://www.laboratorytalk.com/news/fos/fos106.html

► Foss Analytical has developed the functionalities of the software supplied with Milkoscan analysers. This software helps now to identify a suspect raw milk sample quickly and to spot adulterated raw milk.

ANTIBIOTICS

Charm test detects chloramphenicol in raw milk

http://www.laboratorytalk.com/news/chk/chk104.html

Flow strip test detects sulfonamide drugs in milk

http://www.laboratorytalk.com/news/chk/chk103.html

► Charm Sciences has launched two news tests to detect chloramphenicol and sulfonamide drugs in milk: Charm Chloramphenicol Test and Charm Sulfa Test. These tests use the ROSA (Rapid One Step Assay) technology.

HYGIENE

Code of hygienic practice for milk and milk products (CAC/RCP 57-2004)

http://www.codexalimentarius.net/download/standards/10087/CXP_057e.pdf

► The Codex Alimentarius commission has amended this code, which is to apply the recommendations of the *Recommended Code of Practice: General Principles of Food Hygiene* to the particular case of milk and milk producs. It also provides guidance on how to achieve the general requirements contained in the hygiene sections of the Codex commodity standards for milk products.

MELAMINE

Metrohm develops melamine-analysis method

http://www.laboratorytalk.com/news/jmq/jmq146.html

► Methrohm has announced its 844 Compact UV-VIS IC can be used to determine melamine in food products in less than 20 minutes.

PASTEURISATION

F-AP Test verifies pasteurisation in 45 seconds

http://www.laboratorytalk.com/news/chk/chk105.html

► Charm Sciences' Fast Alkaline Phosphatase (F-AP) Test has been accepted as an official pasteurisation test for milk and dairy products.

FORTHCOMING EVENTS

Classified in chronological order

ADDITIVES

15-19 March 2010 Beijing, China	42nd session of the Codex Committee on Food Additives	http://www.codexalimentarius.net

ANALYSIS METHODS AND SAMPLING

8-12 March 2010 Hungary	31st session of the Codex Committee on Methods of Analysis and Sampling	http://www.codexalimentarius.net

MILK AND DAIRY PRODUCTS

1-5 February 2010 Auckland, New-Zealand	9th session of the Codex Committee on Milk and Milk Products	http://www.codexalimentarius.net

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