CECALAIT-S NEWSLETTER

ACTALIA
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ACTALIA Cecalait

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ARTICLE

EVALUATION OF THE CDR FOODLAB® INSTRUMENT

ACTALIA Cecalait was asked to carry out an evaluation of the performances of the CDR Foodlab® analyser on milk and some liquid dairy products. This instrument, produced by CDR, is a polyvalent photometric analyser for the determination of a large panel of chemical criteria in food products. The instrument is equipped with LED sources, reading and thermostated incubation at 37 °C cells, enabling the realisation of 16 determinations in parallel.

The characteristics of the instrument used for this study were:

- CDR Foodlab®
- Type: SLB 222
- Serial No: B-222003/1112
- Production year: 2019.

The instrument was installed in a temperature controlled room (2-23 °C – air-conditioning), without direct sunlight. The installation procedure was performed by CDR.

Lactose in lactose-reduced milk, urea in milk and ammonia in whey have been evaluated using respectively the following ready-to-use reagents kits: 300004, 300010 and 300054, which were packed in bag of 10 tests with a one year shelf-life. The analyses were carried out without prior preparation of the samples.

THE TESTS

The evaluation tests (instrumental and reference analyses according to ISO 14637 for urea and NF V 04-217 for ammonia) were carried out in ACTALIAT Cecalait physico-chemistry laboratory in July 2019. The reference analyses for lactose by HPLC were performed in ACTALIA Contrôle et Qualité at Villers Bocage.

The repeatability and the accuracy of the method and he stability of the instrument for each parameter, were evaluated.

As only the absorbance raw data of the CDR FoodLab® were available, they were transformed in rates using the reference values obtained within the context of the stability and accuracy evaluation for each parameter.

So, the evaluation of the accuracy of each parameter focused only on the residual standard deviation of regression Sy, x and the estimation accuracy ± 2.Sy,x. Indeed, because of this approach, the accuracy regression equation obtained on the basis of the transformed absorbances leads to a regression slope at 1.00 and an intercept at zero.

1. EVALUATION OF THE STABILITY (INTRA-LABORATORY REPRODUCIBILITY)

The stability of the instrument was evaluated by analysing:

• For determination of lactose content in milk:
2 milk samples with different lactose contents: 0.80 g/100 g (level 1) and 1.50 g/100 g (level 2). The milk samples are a mix of 2 UHT milks: one at lactose-reduced content 2.7 % and one without lactose < 0.1 g/100ml.

• For determination of urea content in milk:
3 whole milk samples with different urea contents: 180 mg/l (level 1), 500 mg/l (level 2) and 800 mg/l (level 3). The samples used were SRMs produced by ACTALIA Cecalait.

• For determination of ammonia content in whey:
3 whey samples with different ammonia contents: 20 ppm (level 1), 30 ppm (level 2) and 50 ppm (level 3). The samples used were whey samples from Franche-Comté region

Bronopol was added to the samples to give a final concentration at 0.02 % for each determination. The analyses were performed in duplicate, every 15 minutes to obtain at least 10 measurement cycles.
To evaluate the stability of the instrument, the repeatability and reproducibility were calculated for each level.

The results obtained are presented in the following tables:

Table 1: CRD FoodLab® stability lactose[1]

	Lactose (Abs)		Lactose (g/100g)	
	Level 1	Level 2	Level 1	Level 2
M	0.6180	1.0712	0.825	1.533
Sr	0.020	0.022	0.032	0.035
Sr (%)	3.29	2.07	3.85	2.27
SR	0.020	0.028	0.031	0.043
SR (%)	3.19	2.60	3.73	2.83
r	0.056	0.062	0.088	0.096
R	0.055	0.077	0.085	0.120

Table 2: CRD FoodLab® stability urea[2]

	Urea (Abs)			Urea (mg/l)		
	Level 1	Level 2	Level 3	Level 1	Level 2	Level 3
M	0.3979	0.9472	1.4942	181.69	500.95	818.80
Sr	0.011	0.016	0.036	6.204	9.163	20.74
Sr (%)	2.68	166	2.39	3.41	1.83	2.53
SR	0.012	0.016	0.033	7.028	9.561	19.057
SR (%)	3.04	1.74	2.19	3.87	1.91	2.33
r	0.030	0.044	0.099	17.186	25.382	57.439
R	0.034	0.046	0.091	19.468	26.485	52.787

Table 3: CDR FoodLab® stability ammonia[3]

	Ammonia (Abs)			Ammonia (ppm)		
	Level 1	Level 2	Level 3	Level 1	Level 2	Level 3
M	0.3752	0.8086	1.8050	21.83	31.48	53.67
Sr	0.037	0.026	0.033	0.826	0.569	0.735
Sr (%)	9.88	3.16	1.83	3.78	1.81	1.37
SR	0.035	0.025	0.033	0.778	0.564	0.726
SR (%)	9.31	3.13	1.81	3.56	1.79	1.35
r	0.103	0.071	0.091	2.287	1.576	2.035
R	0.097	0.070	0.090	2.154	1.562	2.011

It can be noted that:

• For the determination of lactose content in milk:
The standard deviation of deviations are in the range of 2.3 to 3.9 % and the standard deviations of reproducibility are about 2.8 to 3.7 % according to the samples rates.
Concerning the standard deviation of reproducibility observed, with no standard criteria, it can be noted that they are very close to the observed repeatability deviations, reflecting a generally weak "instrumental stability" error.

[1] [2] [3] M: mean; Sr and SR (Sr% and SR%): absolute standard deviation of repeatability and reproducibility (and relative); r and R: maximal deviation of repeatability and reproducibility in 95 % of cases.

• For the determination of urea content in milk:
The standard deviations of repeatability are in the range of 1.8 to 3.4 % and the standard deviations of reproducibility are of 1.9 to 3.9 % according to the samples rates.
Concerning the standard deviation of reproducibility observed, with no standard criteria, it can be noted that they are very close to the observed repeatability deviations, reflecting a generally weak "instrumental stability" error.

• For the determination of ammonia content in whey:
The standard deviations of repeatability are in the range of 1.4 to 3.8 % and the standard deviations of reproducibility are of 1.4 to 3.6 % according to the samples rates.
The standard deviation of reproducibility observed is in the same range of the standard deviation of repeatability, indicating a good instrumental stability.

2. EVALUATION OF THE REPEATABILITY

The repeatability of the instrument was evaluated by analysing:

• For the determination of lactose in milk:
26 samples of milk (mix of 2 UHT milks) with lactose contents between 0.01 and 2 g/100 g.

• For the determination of urea in milk:
34 samples of raw milk with urea contents between 170 and 800 g/l: 5 samples of urea SRMs produced by ACTALIA Cecalait and 29 producer's milk from Franche-Comté region.

• For the determination of ammonia in whey:
33 samples of whey from Franche-Comté region with ammonia contents between 12 and 113 ppm.

The samples were analysed in duplicate and Bronopol was added to the samples to give a final concentration at 0.02 %.

The results obtained are presented in the following tables:

Table 4: CDR FoodLab® repeatability lactose urea and ammonia[4]

	n	min	max	M	Sx	Sr	Sr (%)	r
Lactose (Abs)	26	0.1985	1.1332	0.5354	0.254	0.011	2.03	0.030
Lactose (g/100g)		0.169	1.630	0.696	0.024	0.017	2.44	0.047
Urea (Abs)	34	0.3711	1.5231	0.6251	0.223	0.012	1.95	0.034
Urea (mg/l)		166.15	835.58	313.78	129.29	7.072	2.25	19.59
Ammonia (Abs)	33	0.2368	3.1301	0.8649	0.614	0.017	1.92	0.046
Ammonia (ppm)		12.93	112.66	34.58	0.808	0.572	1.66	1.585

It can be noted:

• For the determination of lactose in milk: a standard deviation of repeatability of 0.017 g/100 g on the measurement range of 0.16 to 1.63 g/100 g.
No standard criteria exist for lactose-reduced milk, but it can be compared to the existing standardised methods for the determination of lactose in milk: Sr = 0.022 g/100g (Sr% = 0.44) for the HPLC method according to ISO 22662 and Sr = 0.037 g/100g (Sr% = 0.74) for the differential pH-metric method according to ISO 26462.

• For the determination of urea in milk: a repeatability r obtained (19.6 mg/l) slightly higher than the reference method (ISO 14637): r = 15 mg/l ; Sr = 5.42 mg/l.

• For the determination of ammonia in whey: on the range of the considered rates, a repeatability r obtained using CDR FoodLab® equal to 1.59 ppm against 2.46 ppm using the reference method (NF V 04-217).

3. EVALUATION OF THE ACCURACY

The accuracy was evaluated by analysing 26 samples for the determination of lactose content in milk, 34 samples for the determination of urea in milk and 33 samples for the determination of ammonia in whey. The samples were the same samples analysed for the repeatability evaluation.
Samples with aberrant reference values were eliminated on the basis of the regression residuals greater than 2 x standard deviation of regression residuals: 5% threshold.

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

The results obtained are presented in the following table and figures:

Table 5: CDR FoodLab® accuracy criteria lactose, urea and ammonia[5]

	n	Min	Max	Y	X	Sy	Sx	Sd	Sy,x
Lactose (g/100g)	26	0.179	1.621	0.692	0.692	0.405	0.408	0.044	0.045
Urea (mg/l)	34	173.79	834.04	313.78	313.78	130.52	131.26	13.944	14.160
Ammonia (ppm)	33	13.03	111.12	34.58	34.58	21.38	22.21	6.020	6.116

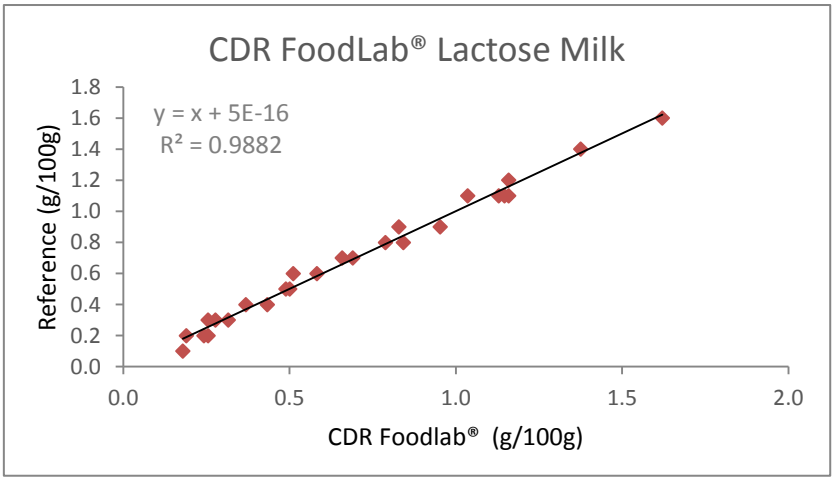

Figure 1: Relation between instrumental and reference results in g/100g of lactose

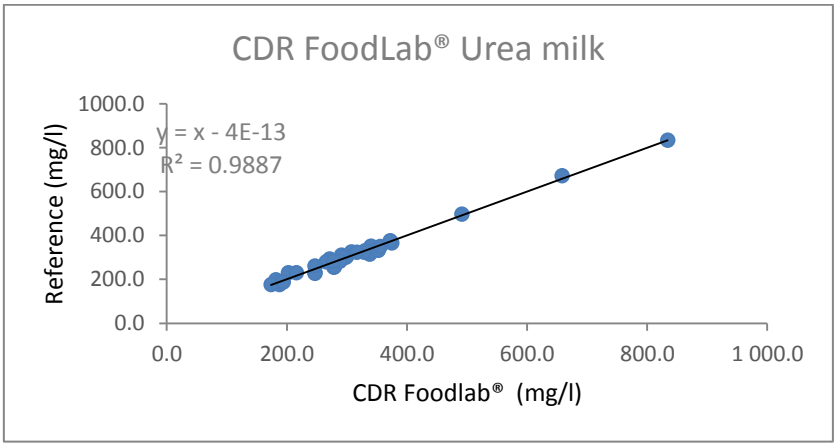

Figure 2: Relation between instrumental and reference results in mg/l of urea

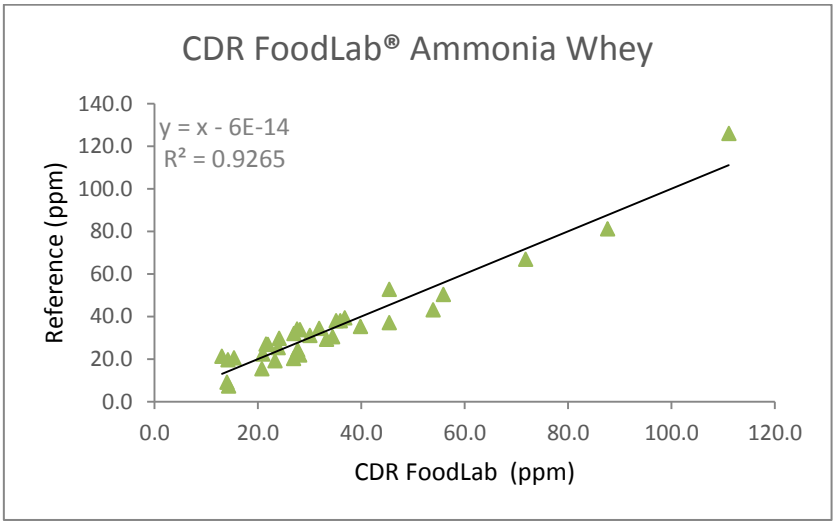

Figure 3: Relation between instrumental and reference results in ppm of ammonia

[5] n, min, max: number of results, minimum and maximum value; Y,X: mean of the results using the reference and instrumental method; Sy, Sx: standard deviation of the results from the reference and instrumental method; Sd: standard deviation of deviations; Sy,x: residual standard deviation

Concerning the relation between the results obtained using CDR FoodLab® method (calculated with the regression equation) and the reference method, it can be noted:

- For the determination of lactose in milk, a residual regression standard deviation (Sy,x) of 0.045 g/100 g, so a precision of estimation of ± 0,09 g/100g.
- For the determination of urea in milk, a residual regression standard deviation (Sy,x) of 14.2 mg/l, so a precision of estimation of ± 28 mg/l.
- For the determination of ammonia in whey, a residual regression standard deviation (Sy,x) of 4.2 ppm, so a precision of estimation of about ± 8 ppm.

4. CONCLUSION

The evaluation of the lactose content in milk (range of 0.01 – 2 g/100 g), the urea content in milk and the ammonia content in whey allows the following conclusions:

- The CDR FoodLab® instrument is easy to use thanks to the use procedures incorporated in the methods.
- No recurring problems were noted during tests with the CDR FoodLab® instrument. It can nevertheless be noted the importance of sampling as well as the addition of the reagents made with a pipette. The pipette must be used with precision so as not to introduce performance problems.
- Concerning the performance of the instrument, it can be noted that:
  - For the determination of the lactose content in lactose-reduced milk, the repeatability is superior to the standard methods (Sr% = 2.44 vs 0.44 for ISO 22662 and 0.74 for ISO 26462) and the accuracy enables to obtain a precision of estimation < 0.1 g/100 g (0.09 g/100 g).
  - For the determination of the urea content in milk; the repeatability is close to the reference method and the precision of estimation is of ± 28 mg/l.
  - For the determination of the ammonia in whey, the repeatability is significantly better than the NF V04-217 reference method (1.59 ppm vs 2.5 ppm) and the precision of estimation is about ± 8 ppm for this type of product (for a range of about 12 to 113 ppm).

According to the evaluation report of the CDR Foodlab® analyser – A. OUDOTTE and P. TROSSAT – July-August 2019

STANDARDS, DRAFT STANDARDS

Classification in alphabetical order by theme

ISO standards under development

MICROBIOLOGY OF THE FOOD CHAIN	
ISO/DIS 6579/A1 September 2019	MICROBIOLOGY OF THE FOOD CHAIN Horizontal method for the detection, enumeration and serotyping of *Salmonell*a – Part 1: Detection of *Salmonella* spp. – Amendment 1
SENSORY ANALYSIS	
ISO/DIS 11036 September 2019	SENSORY ANALYSIS Methodology – Texture profile

ISO published standards

MICROBIOLOGY OF THE FOOD CHAIN	
ISO 15216-2 July 2019	MICROBIOLOGY OF THE FOOD CHAIN Horizontal method for determination of hepatitis A virus and norovirus using real-time RT-PCR – Part 2: Method for detection *Replace ISO 15216-2:2013*
ISO 17410 July 2019	MICROBIOLOGY OF THE FOOD CHAIN Horizontal method for the enumeration of psychrotrophic microorganisms *Replace ISO 17410:2001*

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

NOVEL FOOD

O.J.E.U. L 205, 5th August 2019 – Commission Implementing Regulation (EU) 2019/1314 of 2 August 2019 authorising the change of the specifications of the novel food Lacto-N-neotetraose produced with Escherichia coli K-12 under Regulation (EU) 2015/2283 of the European Parliament and of the Council and amending Commission Implementing Regulation (EU) 2017/2470

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.205.01.0004.01.ENG

O.J.E.U. L 258, 9th October 2019 – Commission Implementing Regulation (EU) 2019/1686 of 8 October 2019 authorising the extension of use of bovine milk basic whey protein isolate as a novel food under Regulation (EU) 2015/2283 of the European Parliament and of the Council and amending Commission Implementing Regulation (EU) 2017/2470

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.258.01.0013.01.ENG

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

O.J.E.U. C 251, 26th July 2019 – Corrigendum to publication of an application for approval of a minor amendment in accordance with the second subparagraph of Article 53(2) of Regulation (EU) No 1151/2012 of the European Parliament and of the Council on quality schemes for agricultural products and foodstuffs [Parmigiano Reggiano (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2019.251.01.0014.01.ENG

O.J.E.U. C 257, 31st July 2019 – Publication of an application for registration of a name 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 [Provola dei Nebrodi (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2019.257.01.0018.01.ENG

O.J.E.U. L 203, 1st August 2019 – Commission Implementing Regulation (EU) 2019/1288 of 25 July 2019 entering a name in the register of traditional specialities guaranteed [Rögös turo (TSG) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.203.01.0001.01.ENG

STANDARDS - REGULATIONS

O.J.E.U. L 214, 16th August 2019 – Commission Implementing Regulation (EU) 2019/1347 of 8 August 2019 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Roquefort (PDO) (cheese)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.214.01.0001.01.ENG

O.J.E.U. C 283, 21st August 2019 – Publication of an application for registration of a name 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 [Krasotiri Ko / Tiri tis Possias (PGI) (cheese)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2019.283.01.0001.01.ENG

O.J.E.U. C 320, 24th September 2019 – Publication of an application for registration of a name 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 [Bjelovarski Kvargl (PGI) (cheese)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2019.320.01.0009.01.ENG

O.J.E.U. C 333, 4th October 2019 – Publication of an application for registration of a name 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 [Pecorino del Monte Poro (PDO) (cheese)]
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2019.333.01.0019.01.ENG

O.J.E.U. L 256, 7th October 2019 – Commission Implementing Regulation (EU) 2019/1670 of 1 October 2019 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications “Grana Padano” (PDO) (cheese)
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.256.01.0006.01.ENG

O.J.E.U. L 257, 8th October 2019 – Commission Implementing Regulation (EU) 2019/1674 of 27 September 2019 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications “Beurre d’Isigny” (PDO) (butter)
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.257.01.0004.01.ENG

O.J.E.U. L 258, 9th October 2019 – Commission Implementing Regulation (EU) 2019/1683 of 2 October 2019 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications “Crème d’Isigny” (PDO) (cream)
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.258.01.0008.01.ENG

O.J.E.U. L 258, 9th October 2019 – Commission Implementing Regulation (EU) 2019/1684 of 2 October 2019 approving non-minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications “Banon” (PDO) (cheese)
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.258.01.0010.01.ENG

PESTICIDES

O.J.E.U. L 185, 11th July 2019 – Commission Regulation (EU) 2019/1176 of 10 July 2019 amending Annexes II, III and V to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for 2,5-dichlorobenzoic acid methylester, mandipropamid and profoxydim in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.185.01.0001.01.ENG

O.J.E.U. L 239, 17th September 2019 – Commission Regulation (EU) 2019/1559 of 16 September 2019 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 cyflufenamid, fenbuconazole, fluquinconazole and tembotrione in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.239.01.0001.01.ENG

O.J.E.U. L 246, 26th September 2019 – Commission Regulation (EU) 2019/1582 of 25 September 2019 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 imazalil in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2019.246.01.0001.01.ENG

AFNOR VALIDATIONS

During its May and July meetings, the Technical Committee of NF VALIDATION approved by vote:

Commercial name	Date	Certificate	Description
NEW VALIDATIONS			
RAPID'*B.CEREUS*	Validation date: 22 Mar 2019 **End of validity: 22 Mar 2023**	**BRD-07/26-03/19**	**Enumeration of *Bacillus cereus*** Dairy products, ready-to-eat and ready-to-reheat and vegetables products
SALMONELLA DNA TEST KIT	Validation date: 3 Jul 2019 **End of validity: 3 Jul 2023**	**BCK-40/01-07/19**	**Detection of *Salmonella* spp.** Ready-to-eat and ready-to-reheat products, meats products, ingredient and specific food, human and animal feed products, industrial production environ-mental samples and primary production samples
RENEWALS OF VALIDATIONS			
RAPID'*L. MONO* DETECTION	Validation date: 7 Sep 1998 Renewal: 28 Nov 2002, 28 Sep 2006, 1 Jul 2010, 3 Jul 2014 and 3 Jul 2019 Extension: 25 Sep 2008 **End of validity: 15 Sep 2023**	**BRD-07/04-09/98**	**Detection of *Listeria monocytogenes* and *Listeria* spp.** All human food products and production environmental samples
RAPID'*LISTERIA* SPP.	Validation date: 15 Dec 2006 Renewal: 1 Jul 2010, 27 Nov 2014 and 3 Jul 2019 Extension: 29 Mar 2007 **End of validity: 15 Dec 2023**	**BRD-07/12-12/06**	**Detection of *Listeria* spp.** All human food products and production environmental samples
COMPASS *LISTERIA* AGAR DETECTION	Validation date: 28 Nov 2002 Renewal: 25 May 2007, 24 Sep 2010, 27 Nov 2014 and 4 Jul 2019 Extension: 27 Sep 2007, 12 May 2011 and 29 Mar 2013 **End of validity: 28 Nov 2023**	**BKR-23/02-11/02**	**Detection of *Listeria monocytogenes* and *Listeria* spp.** All human food products and production environmental samples
RHAPSODY AGAR	Validation date: 5 Jun 2015 Renewal: 16 May 2019 **End of validity: 5 Jun 2023**	**BKR-23/09-05/15B**	**Enumeration of *Pseudomonas* spp.** Dairy products
EXTENSIONS OF VALIDATIONS			
THERMO SCIENTIFIC SURETECT *SALMONELLA* SPECIES PCR ASSAY	Validation date: 4 Nov 2013 Extension: 30 Jan 2014, 21 Mar 2014, 30 Jun 2016, 24 Mar 2017, 3 Dec 2018 and 16 May 2019 Renewal: 22 Mar 2018 **End of validity: 4 Nov 2021**	**UNI-03/07-11/13**	**Detection of *Salmonella* spp.** All human food products, pet food and production environmental samples (ex-cept primary production environment)

The validation certificates and the recapitulative list are available at the following website address:
http://www.afnor-validation.com/afnor-validation-validated-methods/validated-methods.html

IN THE PRESS – ON THE WEB

Classification in alphabetical order of keywords

FOOD ADDITIVES

Re-evaluation of hydrochloric acid (E 507), potassium chloride (E 508), calcium chloride (E 509) and magnesium chloride (E 511) as food additives

https://efsa.onlinelibrary.wiley.com/doi/10.2903/j.efsa.2019.5751

▸ The Panel on Food Additives and Flavourings added to Food provided a scientific opinion re-evaluating the safety of chlorides (E 507-509, E 511) as food additives, in particular in milk and dairy products. The Panel concluded that the exposure to chloride from hydrochloric acid and its potassium, calcium and magnesium salts (E 507, E 508, E 509 and E 511) does not raise a safety concern at the reported use and use levels.

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