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ACTALIA Cecalait

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ARTICLE

EVALUATION OF THE INSTRUMENT « MOPLANT »

Moplant, manufactured by Hettich Benelux (Netherlands), is an instrument which allows the carrying out of some steps of the fat by extraction according to RG or SBR methods determination procedures in milk and dairy products.

This instrument combines a Mojonnier centrifuge (16 places), 2 compartments with heating plates for the evaporation of solvents, 2 drying chambers and 2 cooling chambers at room temperature. A vacuum system allows capture of vapours and removes them outside through an evacuation pipe put under a solvent fume chamber. A vacuum pump allows reduction of the pressure inside the chambers.

The objective of this study is to evaluate the possibility to replace the evaporation and drying (at constant weight) steps of the RG and SBR methods by the Moplant, becoming an alternative method.

The tests

The evaluation tests were carried out in ACTALIA Cecalait physico-chemistry laboratory (reference and instrumental analyses) in October and November 2016. The repeatability and the accuracy of the alternative method were evaluated for the determination of fat in milk (according to ISO 1211), in cream (ISO 2450), in dried milk (ISO 1736) and in cheese (ISO 1735).

1-MATERIALS AND METHODS USED

1.1- Material

The material used for the reference method is in accordance with the ISO standards. The laboratory uses glass tubes for the extraction and the tests were performed using 90 x 30 mm aluminium dishes with lids to collect the solvent.

Any specific consumable is necessary for the « Mo Plant » method. The extractions were performed using glass tubes, so the Mojonnier centrifuge was not used for these tests.

1.2- Procedure

General principle: Each sample was analysed in duplicate for each method (alternative and reference).
The 2 methods were performed in parallel for each sample: in the same set, 4 tubes for each sample; 2 tubes followed the end of the reference method procedure, and the 2 other one followed the alternative method procedure.
Please note, the beginning of the procedure (extraction and test portion) was the same for the both methods.

Evaporation process – drying of the alternative method using Mo-Plant:

- Place the empty dishes in the chamber at 110°C at reduced pressure (-0.8 bars) for 5 minutes.
- Place the dishes in the chamber at room temperature and let cool for 7 minutes.
- Weigh the dishes to the nearest 0.1mg.
- After transfer of the solvents in the dishes, place them on the heating plates at 110°C for the necessary time of complete evaporation of the solvent in the dish (about 10-15 mn). Proceed by progressive contact with the plate to avoid potential splashes. Proceed identically for the 3 extractions.
- Place the dishes in the chamber at 110°C at reduced pressure (-0.8 bars) for 5 minutes.
- Place the dishes in the chamber at temperature room and let cool for 7 minutes.
- Weigh the dishes to the nearest 0.1mg (1 blank sample per set was also performed according to this principle).

Calculate the fat content according to the following formula:

$$\text{Fat (\%)} = 100 \times [(M2_e - M0_e) - (M2_b - M0_b)]/M1$$

$M0_e$ and $M0_b$: mass of the empty dish after respectively drying of the sample and the blank sample (to the nearest 0.1 mg)
M1: mass of the test portion (to the nearest 0.1 mg)
$M2_e$ and $M2_b$: mass of the dish with the residue after respectively drying of the sample and the blank sample (to the nearest 0.1 mg).

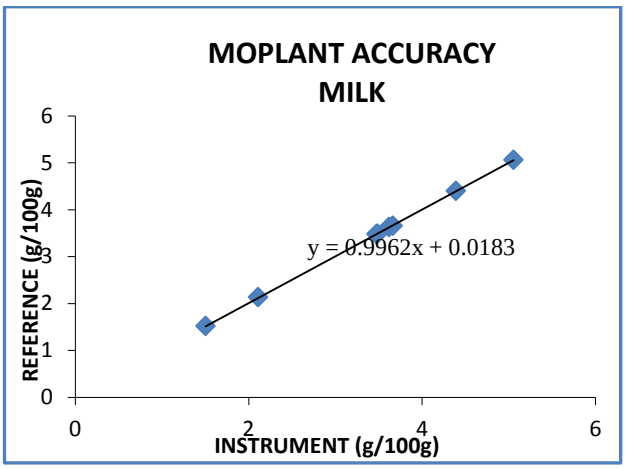
2-RESULTS

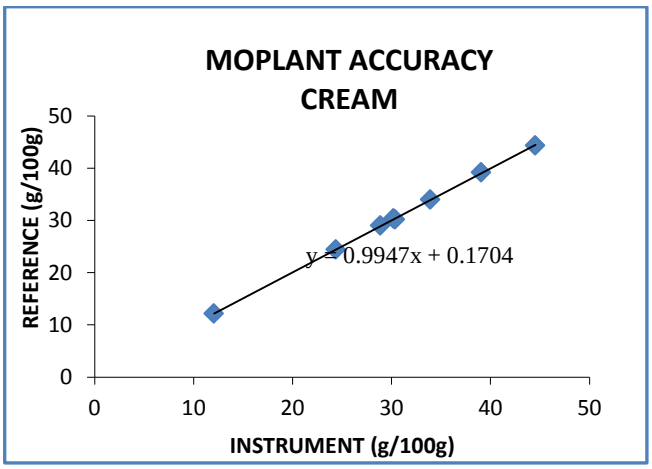
The tests were performed on 8 samples of raw and UHT milk, 8 samples of raw and UHT cream, 9 samples of dried milk and 9 samples of cheese

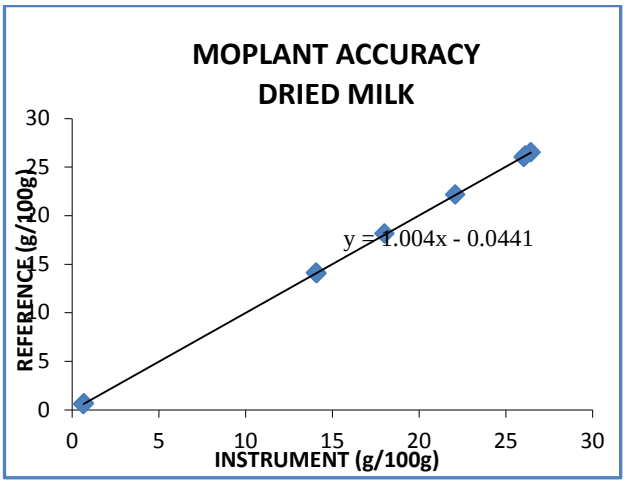
The following table presents the results obtained:

	MILK		CREAM		DRIED MILK		CHEESE	
Criterion	Instrument	Reference	Instrument	Reference	Instrument	Reference	Instrument	Reference
n	8		8		9		9	
M (g/100g)	3.41	3.42	30.43	30.44	16.47	16.49	22.34	22.14
Sx (g/100g)	1.14	1.14	9.73	9.68	10.18	10.22	11.35	11.35
Sr (g/100g)	0.007	0.009	0.23	0.23	0.05	0.08	0.08	0.06
d (g/100g)	-0.005*		-0.01*		-0.02*		0.20*	
Sd (g/100g)	0.009		0.15		0.07		0.13	

Table 1: Moplant repeatability and accuracy criteria in milk, cream, dried milk and cheese samples
n: number of results, M and Sx: mean and standard deviation of the results, Sr: standard deviation of repeatability, d and Sd: mean and standard deviation of deviations (instrumental vs reference).
*Non significant difference within 5%







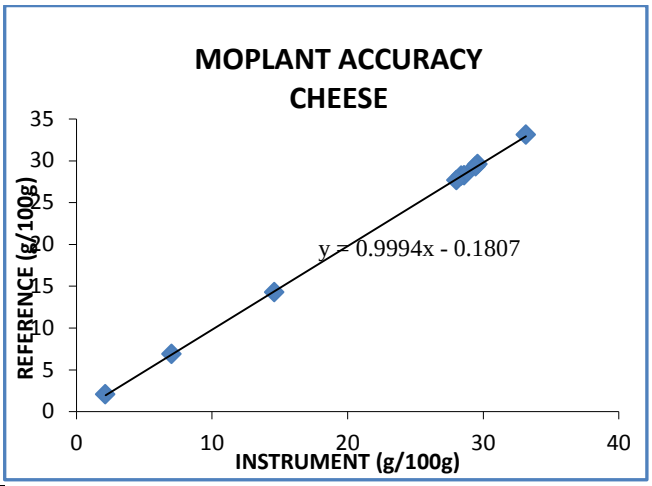


Figure 1: Relation between the « Moplant » and « reference » results on milk, cream, dried milk and cheese samples

For milk, the standard deviation of repeatability observed for the alternative method is lower than the ISO 1211 method limit value (Sr ≤ 0.015 g/100g). The mean deviation between the both sets of results is very weak (-0.005 g/100g) and non significant.

For cream, the relative standard deviation of repeatability observed for the alternative method is lower than the ISO 2450 method limit value (Sr ≤ 0.5 %). The mean deviation between the both sets of results is very weak (-0.01 g/100g) and non significant.

For dried milk, the standard deviation of repeatability observed for the alternative method is lower than the ISO 1736 method limit value (Sr ≤ 0.20 g/100g). The mean deviation between the both sets of results is very weak (-0.02 g/100g) and non significant.

For cheese, the standard deviation of repeatability observed for the alternative method is lower than ISO 1735 method limit value (Sr ≤ 0.30 g/100g). The mean deviation between the both sets of results is of 0.20 g/100g (no significant difference at 5 % threshold).

Overall treatment

The following table presents the results obtained for all matrices dropped:

Criterion	Instrument	Reference
n	34	
M (g/100g)	18.24	18.19
Sx (g/100g)	13.09	13.08
d (g/100g)	0.04	
Sd (g/100g)	0.14	
Sy,x (g/100g)	0.14	
Sy,x (%)	0.75	

Table 2: Moplant accuracy criteria in milk and dairy products

n: number of results, M and Sx: mean and standard deviation of the results, Sr: standard deviation of repeatability, d and Sd: mean and standard deviation of deviations (instrumental-reference), Sy,x and Sy,x(%): absolute and relative residual standard deviation of the reference linear regression = f(instrument).

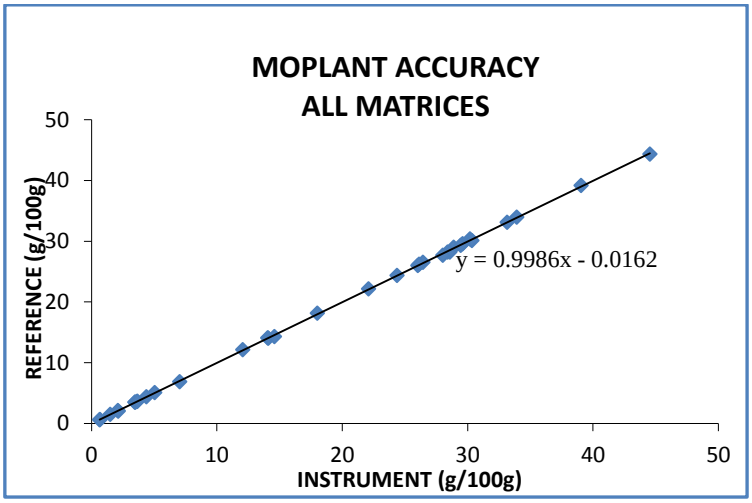


Figure 2: Relation between the « Moplant » and « reference » results in milk and dairy products samples.

It can be noted that the mean and the standard deviation of deviations are respectively 0.04 and 0.14 g/100g. The slope (0.999) and the intercept (-0.02) of regression are respectively not significantly different from 1 and 0 (P=5%). The residual standard deviation is 0.14 g/100g.

CONCLUSION

We can conclude that the results obtained for the tests performed in milk, cream, dried milk and cheese are not significantly different from the reference values obtained using the standardised methods.
This alternative method gives equivalent results compared to reference methods on milk and dairy products.

The use of Moplant can be an interesting possibility to gain time thanks to the dishes drying and the solvent evaporation steps.

For safety reason, the Moplants exhaust pipe must be placed either in a fume cupboard or through an external window.

STANDARDS, DRAFT STANDARDS

Classification in alphabetical order by theme

ISO standards under development

INFANT FORMULA AND ADULT NUTRITIONALS	
ISO/DIS 20635 March 2017	INFANT FORMULA AND ADULT NUTRITIONALS Determination of vitamin C by (ultra) high performance liquid chromatography with ultraviolet detection ((U)HPLC-UV)
QUALITY ASSURANCE	
ISO/IEC DIS 17025 March 2017	General requirements for the competence of testing and calibration laboratories

ISO published standards

MICROBIOLOGY OF THE FOOD CHAIN	
ISO 18465 January 2017	MICROBIOLOGY OF THE FOOD CHAIN Quantitative determination of emetic toxin (Cereulide) using LC-MS/MS
SENSORY ANALYSIS	
ISO 5492/Amd 1 December 2016	SENSORY ANALYSIS Vocabulary - Amendment 1

NEW EU REGULATIONS

Classification is established in alphabetical order of the first keyword

CONTAMINANTS

O.J.E.U. L 327, December 2nd, 2016 – Commission Recommendation (EU) 2016/2115 of 1 December 2016 on the monitoring of the presence of Δ^9-tetrahydrocannabinol, its percursors and other cannabinoids in food
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.327.01.0103.01.ENG

HYGIENE RULES

O.J.E.U. L 29, February 3rd, 2017 – Commission Regulation (EU) 2017/185 of 2 February 2017 laying down transitional measures for the application of certain provisions of Regulation (EC) No 853/2004 and (EC) No 854/2004 of the European Parliament and of the Council
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2017.029.01.0021.01.ENG

PESTICIDES

O.J.E.U. L 30, February 3rd, 2017 – Commission Regulation (EU) 2017/170 of 30 January 2017 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 bifenthrin, carbetamide, cinifon ethyl, fenpropimorph and triflusulfuron in or on certain products
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2017.030.01.0001.01.ENG

O.J.E.U. L 30, February 3rd, 2017 – Commission Regulation (EU) 2017/171 of 30 January 2017 amending Annexes II, III and IV to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for aminopyralid, azoxystrobin, cyantraniliprole, cyflufenamid, cyproconazole, diethofencarb, dithiocarbamates, fluazifop-P, fluopyram, haloxyfop, isofetamid, metalaxyl, prohexadione, propaquizafop, pyrimethanil, *Trichoderma atroviride* strain SC1 and zoxamide in or on certain produtcs
http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2017.030.01.0045.01.ENG

STANDARDS - REGULATIONS

P.D.O. / P.G.I.

O.J.E.U. L 327, December 2nd, 2016 – Commission Implementing Regulation (EU) 2016/2103 of 21 November 2016 entering a name in the register of protected designations of origin and protected geographical indications [Burrata di Andria (PGI) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2016.327.01.0016.01.ENG

O.J.E.U. L 15, January 19th, 2017 – Commission Implementing Regulation (EU) 2016/2103 of 21 November 2016 entering a name in the register of protected designations of origin and protected geographical indications [Brillat-Savarin (PGI) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2017.015.01.0036.01.ENG

O.J.E.U. C 25, January 25th,2017 – Publication of an amendment application 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 [Picodon (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.C_.2017.025.01.0005.01.ENG

O.J.E.U. L 22, January 27th, 2017 – Commission Implementing Regulation (EU) 2017/136 of 16 January 2017 approving non- minor amendments to the specification for a name entered in the register of protected designations of origin and protected geographical indications [Ossau-Iraty (PDO) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2017.022.01.0004.01.ENG

O.J.E.U. L 22, January 27th, 2017 – Commission Implementing Regulation (EU) 2017/138 of 16 January 2017 entering a name in the register of protected designations of origin and protected geographical indications [Raclette de Savoie (PGI) (cheese)]

http://eur-lex.europa.eu/legal-content/FR/TXT/?uri=uriserv:OJ.L_.2017.022.01.0006.01.ENG

AFNOR VALIDATIONS

During its November 2016 and January 2017 meetings
the Technical Committee of NF VALIDATION approved by vote:

Commercial name	Date	Certificate	Description
NEW VALIDATIONS			
3M™ MOLECULAR DETECTION ASSAY 2- *SALMONELLA*	Validation date: 25 Nov 2016 **End of validity: 25 Nov 2020**	**3M-01/16-11/16**	**Detection of *Salmonella* spp.** All human food products and production environmental samples
3M™ PETRIFILM™ RAPID AEROBIC COUNT PLATE	Validation date: 25 Nov 2016 **End of validity: 25 Nov 2020**	**3M-01/17-11/16**	**Enumeration of mesophilic aerobic flora** Milk powders and dairy products
GENE-UP *LISTERIA MONOCYTOGENES*	Validation date: 25 Nov 2016 **End of validity: 25 Nov 2020**	**BIO-12/40-11/16**	**Detection of *Listeria monocytogenes*** Meat products and dairy products
BACGENE *LISTERIA MONOCYTOGENES* + BACGENE *LISTERIA* MULTIPLEX	Validation date: 25 Jan 2017 **End of validity: 26 Jan 2021**	**EGS-38/03-01/17**	**Detection of *Listeria monocytogenes*** All human food products and production environment samples
BACSPEC *LISTERIA*	Validation date: 25 Jan 2017 **End of validity: 26 Jan 2021**	**EGS-38/04-01/17**	**Detection of *Listeria* spp.** All human food products and production environment samples
BACGENE *LISTERIA* SPP. + BACGENE *LISTERIA* MULTIPLEX	Validation date: 25 Jan 2017 **End of validity: 26 Jan 2021**	**EGS-38/02-01/17**	**Detection of *Listeria* spp.** All human food products and production environment samples
RENEWALS OF VALIDATIONS			
GENEDISC *E. COLI* O157:H7	Validation date: 28 Nov 2008 Extension: 27 Jan 2009, 4 Feb .2010, 20 Mar 2014 and 14 Oct 2015 Renewal: 23 May 2013 and 24 Nov 2016 **End of validity: 28 Nov 2020**	**GEN-25/06-11/08**	**Detection of *E. coli* O157:H7** Meats, dairy products and vegetables
TEMPO EC	Validation date: 4 Feb 2005 Renewal: 26 Jan 2009, 30 Nov 2012 & 27 Jan 2017 **End of validity: 4 Feb 2021**	**BIO-12/13-02/05**	**Enumeration of *E. coli*** All human food and pet food products (except beverages and cattle food)

AFNOR VALIDATIONS

GENEDISC *SALMONELLA* SPP.	Validation date: 28 Nov 2008 Extension: 27 Jan 2009, 4 Feb 2010, 20 Mar 2014 and 14 Oct 2015 Renewal: 23.05.2013 and 24.11.2016 **End of validity: 28 Nov 2020**	**GEN-25/05-11/08**	**Detection of *Salmonella* spp.** All human food products and animal feed
TRANSIA PLATE *SALMONELLA* GOLD	Validation date: 23 Mar 2001 Extension: 12 May 2011 and 18 Mar 2016 Renewal: 3 Feb 2005, 2 Jul 2009, 29 Nov 2012 and 25 Jan 2017 **End of validity: 3 Feb 2021**	**TRA-02/08-03/01**	**Detection of *Salmonella* spp.** All human and animal food products and production environment samples (except primary production stage environment)
IQ-CHECK *CRONOBACTER* SPP.	Validation date: 31 Jan 2013 Extension: 3 Oct 2013 Renewal: 27 Jan 2017 **End of validity: 31 Jan 2021**	**BRD-07/23-01/13**	**Detection of *Cronobacter* spp.** Powdered infant formula and production environment samples
EXTENSIONS OF VALIDATIONS			
GENE-UP *LISTERIA* SPP.	Validation date: 29 Sep 2016 Extension: 24 Nov 2016 **End of validity: 29 Sep 2020**	**BIO-12/39-09/16**	**Detection of *Listeria* spp. (except *Listeria grayi*)** All human food products and production environment samples
GENE-UP *LISTERIA MONOCYTOGENES*	Validation date: 24 Nov 2016 Extension: 27 Jan 2017 **End of validity: 24 Nov 2020**	**BIO-12/40-11/16**	**Detection of *Listeria monocytogenes*** All human food products and production environment samples
VALIDITY EXTENSIONS OF VALIDATIONS			
RAPID' *E. COLI* 2	Validation date: 19 Nov 1997 Renewal: 7 Mar 2002, 2 Dec 2004, 28 Nov 2008 and 29 Nov 2012 **End of validity: 2 Dec .2016** **Validity extented till: 2 Jun 2017**	**BRD-07/01-07/93**	**Enumeration of *E. coli* at 44 °C** All human food products

AFNOR VALIDATIONS

RAPID' *E. COLI* 2	Validation date: 2 Dec 2004 Renewal: 28 Nov 2008 and 29 Nov 2012 **End of validity:** **2 Dec 2016** **Validity extented till:** **2 Jun 2017**	**BRD-07/07-12/04**	**Enumeration of *E. coli* at 37 °C** All human food products
RAPID' *E. COLI* 2	Validation date: 2 Dec 2004 Renewal: 28 Nov 2008 and 29 Nov 2012 **End of validity:** **2 Dec 2016** **Validity extented till:** **2 Jun 2017**	**BRD-07/08-12/04**	**Enumeration of coliforms at 37 °C** All human food products
LUMIPROBE 24 ***SALMONELLA***	Validation date: 29 Nov 2000 Extension: 7 Mar 2002 Renewal: 8 Apr 2005, 18 May 2009 & 29 Nov 2012 **End of validity:** **29 Nov 2016** **Validity extented till:** **29 May 2017**	**EUR-15/02-11/00**	**Detection of *Salmonella* spp.** All human and animal food products
ASSURANCE GDS ***SALMONELLA***	Validation date: 26 Jan 2009 Renewal: 29 Nov 2012 **End of validity:** **26 Jan 2017** **Validity extented till:** **26 May 2017**	**TRA-02/12-01/09**	**Detection of *Salmonella* spp.** All human and animal food products and production environment samples (excluding primary production samples environment)
RAPID' *STAPH*	Validation date: 4 Feb 2005 Renewal: 27 Jan 2009 and 31 Jan 2013 **End of validity:** **4 Feb 2017** **Validity extented till:** **30 Sep 2017**	**BRD-07/09-02/05**	**Enumeration of coagulase positive *Staphylococcus*** All human food products and production environment samples
AL/GELOSE DETECTION	Validation date: 26 Jan 2009 Renewal: 29 Nov 2012 Extension: 2 Feb 2012 **End of validity:** **26 Jan 2017** **Validity extented till:** **26 May 2017**	**BRD-07/16-01/09**	**Detection of *Listeria monocytogenes* and *Listeria* spp.** All human food products and production environment samples

AFNOR VALIDATIONS

AL/GELOSE ENUMERATION	Validation date: 26 Jan 2009 Renewal: 29 Nov 2012 **End of validity:** **26 Jan 2017** **Validity extented till:** **26 May 2017**	**BRD-07/17-01/09**	**Enumeration of *Listeria monocytogenes*** All human food products and production environment samples
QIAGEN MERICON *SALMONELLA* SPP.	Validation date: 1 Jan 2013 **End of validity:** **1 Feb 2017** **Validity extented till:** **24 Mar 2017**	**QIA-36/01-02/13**	**Detection of *Salmonella* spp.** All human food and animal feeding stuffs and environmental samples (except primary production stage 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

ADDITIVES

Re-evaluation of karaya gum (E 416) as a food additive

http://www.efsa.europa.eu/en/efsajournal/pub/4598

► Following a request from the European Commission, the EFSA panel on food additives was asked to deliver a scientific opinion on the re-evaluation of karaya gum (E 416) as a food additive, in particular in fermented dairy products. The Panel concluded that there is no safety concern at the refined exposure assessment for the use of this food additive.

Re-evaluation of agar (E 406) as a food additive

http://www.efsa.europa.eu/en/efsajournal/pub/4645

► Following a request from the European Commission, the EFSA panel on food additives was asked to deliver a scientific opinion on the re-evaluation of agar (E 406) as a food additive. The Panel concluded that there is no need for a numerical ADI for agar and that there is no safety concern for the general population at the refined exposure assessment for the reported uses of agar as a food additive.

Re-evaluation of locust bean gum (E 410) as a food additive

http://www.efsa.europa.eu/en/efsajournal/pub/4646

► Following a request from the European Commission, the EFSA panel on food additives was asked to deliver a scientific opinion on the re-evaluation of locust bean gum (E 410) as a food additive. The Panel concluded that there is no need for a numerical ADI for locust bean gum and that there is no safety concern for the general population at the refined exposure assessment for the reported uses of agar as a food additive. However, the available data do not allow an adequate assessment of the safety of locust bean gum (E 410) in the food for infants and young children.

PESTICIDES

Review of the existing maximum residue levels for lufenuron according to Article 12 of Regulation (EC) No 396/2005

http://www.efsa.europa.eu/en/efsajournal/pub/4652

► According to Article 12 of Regulation (EC) No 396/2005, EFSA has reviewed the maximum residue levels (MRLs) currently established at the European level for the pesticide active substance lufenuron. No apparent risk to consumers was identified but some information required by the regulatory framework was missing. Hence, the consumer risk assessment is considered indicative only and some MRL proposals derived by EFSA still require consideration by risk managers.

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