



EUROPEAN COMMISSION
DIRECTORATE-GENERAL
TAXATION AND CUSTOMS UNION
Customs
Customs Tariff

Brussels,
TAXUD/A/4-
taxud.a.4(2018)1317454

LIMITED

MINUTES

CUSTOMS 2020

CLEN ACTION 2

DISCUSSION MEETING ON THE

CLEN RING TEST ON MILK PRODUCTS

25 January 2018 - Brussels - EC

Version 13 March 2018
Minutes reported by CLENTAS, under contract TAXUD/2017/DE/306
First version submitted 8 February
Approved by the coordinators and TAXUD-A4 representative,
the Action 2 Leader and the meeting participants,
Approved by the Head of DG TAXUD Unit A4

1. Approval of the agenda and of the minutes of previous meeting

The meeting took place in DG TAXUD premises in Brussels.

The coordinators of the test and the European Commission representative welcomed the participants. Practical information was given and the participants briefly introduced themselves.

The meeting agenda was adopted as follows:

- Welcome and introduction by DG TAXUD and by the ring test coordinators
- Discussion on **Permeates**: parameters for the differentiation between milk and whey permeates, in light of Commission Implementing Regulation (EU) No 2016/534
- Discussion on **Meursing**: milk proteins and milk fat determination, in light of Regulation (EU) No 2015/824
- Discussion on conclusions and proposals

Previous meeting minutes

The previous meeting was the preparatory meeting of the Customs Laboratories European Network (CLEN) ring test on milk products held on 5-6 October 2016 in Thessaloniki, Greece. The meeting draft minutes were first submitted on 19 October 2016 and the final approved minutes were released on 17 November 2016.

2. Nature of the meeting

This meeting was organised to discuss the results of the CLEN first ring test on milk products.

This meeting was not public; it was restricted to one representative from the Customs Laboratories per country having taken part in the test, the test coordinators (Greece), the CLEN Action 2 Leader, the contractor providing assistance to the CLEN and members of DG TAXUD Unit A4.

20 participants attended the meeting.

3. List of points discussed

3.1 Presentation of the CLEN ring test on milk products

The coordinators briefly showed the test design, slides 1 to 5 of the [Annex I](#).

The main characteristics are reported hereafter.

[Background and objectives](#)

The decision to perform this test was taken during the CLEN 18th Plenary Meeting, held on 19 February 2016 (report TAXUD/83610/2016).

A preparatory meeting to design the test was held on 5-6 October 2016 in Thessaloniki, Greece.

The test itself was performed in 2017.

“Permeates part”: the main objective of the test was to differentiate milk and whey permeates according to the new Additional Notes 3 and 4 to the Combined Nomenclature (Commission Implementing Regulation (EU) No 2016/534).

“Meursing part”: the second objective of the test was to estimate milk fat and quantify milk proteins using the methods of Regulation (EC) No 900/2008, as modified by Regulation (EU) No 2015/824.

The test focussed on parameters or products for which there was no commercial proficiency test available.

[Participants](#)

26 Customs Laboratories, belonging to 17 countries, took part in the ring test: 21 laboratories for the “Permeates” part and 25 for the “Meursing” part.

Samples

Permeates part, 6 commercial samples, all in powder:

- Sample 1: milk permeate
- Sample 2: sweet whey permeate
- Sample 4: acid whey permeate (from bacteria fermentation)
- Sample 5: unknown sample n°1 (milk permeate)
- Sample 6: unknown sample n°2 (sweet whey permeate)
- Sample 7: aged milk permeate

Note: a "Sample 3 as acid whey permeate from acidification" was initially foreseen but was abandoned in the test due to rarity on the market and difficulties in raw material supply.

Meursing part, 2 samples:

- Sample HPP: commercial sample, high protein preparation for beverage, in powder
- Sample MMS: reconstituted sample, modified milk shake, in powder

Parameters and performance of the test

The test samples for the "Meursing part" were shipped to the participants at the beginning of June 2017. The samples for the "Permeates part" were shipped at mid-July 2017.

In both cases, the laboratories had two months to perform the analyses and submit their results.

Analytical results were provided for the following parameters:

Permeates part:

- | | | |
|--------------------|---------------------|--------------|
| - pH | - CMP (facultative) | - Sulphates |
| - Moisture content | - Ash | - Calcium |
| - Organoleptic | - Lactose | - Phosphorus |
| - Lactates | - Nitrogen | |

In addition, the laboratories provided suggestions of CN codes

Meursing part:

- Milk fat
- Milk proteins

In addition, the laboratories provided suggestions of CN codes and additional codes.

Draft report

The first draft report of the test, including the test presentation, all the results, data treatment and statistics was sent to the participants on 15 January 2018, after validation by the coordinators.

In addition, the complete summary tables of the results were sent to the participants on 22 January to prepare the meeting discussions.

This meeting aims at discussing and interpreting the test results.

3. II Discussion and interpretation of the results for the “Permeates” part

Discussion and interpretation of the analytical results for the **differentiation of milk permeates** (subheading 0404 90) and **whey permeates** (subheading 0404 10), according to Additional notes 3 and 4 of the Combined Nomenclature (Commission Implementing Regulation (EU) No 2016/534); differentiation required to check the compliance of the declarations for permeates.

Milk permeates have a higher duty rate than whey permeates.

As a result, there is a risk that importers might deliberately try to reduce customs duty liability by shifting to a product classification with a lower duty rate: in this case fraudulently declaring milk permeate product as a ‘whey permeate’.

The meeting discussions were based on the test 1st draft report, the results summary table (also reported in Annex I slide 6) and the coordinators presentation slides 6 to 12, in **Annex I**.

Regarding dairy permeate powders, the meeting participants quoted the recent standard adopted in 2017 and providing the dairy industry and interested actors with definitions and compositional characteristics: the Codex “Standard for dairy permeate powders – CXS 331-2017”.

3.II.1 Differentiating acid whey permeates from other permeates

pH determination is sufficient to distinguish between acid whey permeate and other permeates.

Lactates determination can be used for confirmation for whey permeates from fermentation.

Complementary note: the acid whey permeate product used in this test was obtained from lactic acid bacteria fermentation, and so the lactates content is high. Should an acid whey permeate initially obtained by chemical acidification be considered, then only the pH would probably be relevant. According to producers, acid whey (and consequently acid whey permeates) normally has a pH from 4.0 to 5.2.

3.II.2 Differentiation between milk permeates and sweet whey permeates

Overview of the parameters tested

Determinations of pH, moisture, sulphates, calcium, phosphorus, ash or organoleptic testing were not able to differentiate between milk permeates and sweet whey permeates.

Based on the test results, definite differentiation is not possible using a single parameter.

The three parameters that remain of interest are caseinomacropeptide, or glycomacropeptide (CMP), lactates and to a lesser extend nitrogen.

CMP

When defining the test, a low number of participants were expected for this parameter, as only very few Customs Laboratories currently have the ability to determine CMP. But the participation was indeed very low, with only 3 laboratories reporting “results” and in fact only one laboratory having performed the analysis with previous experience of this determination:

1. Laboratory 6109 reported ‘no’ in the result’s sheet, meaning ‘not performed’.
2. Laboratory 6213 performed this determination, but was not familiar with it and was uncertain about the results (the presence of CMP) and therefore reported ‘not present’ for all samples.
3. Laboratory 2591 was the only experienced laboratory.

From Laboratory 2591 results alone, CMP does not appear as a relevant parameter to distinguish between milk permeates (CMP presence in 1 of the 3 milk permeates) and sweet whey permeates (CMP presence in 1 of the 2 sweet whey permeates).

In order to confirm the results, Laboratory 2591 will be provided with a new set of 3 of the samples and will perform the CMP analysis again.

Lactates

Based on the available test results, lactates is a promising parameter because whey permeates show a higher concentration than milk permeates.

However, the results are not sufficient to reach a conclusion, in particular as the number of significant figures required for an adequate data treatment was underestimated when defining the test and taking into account the low amounts of lactates found in all samples of milk and sweet whey permeates. Specifically, in the test design, the lactates results were expressed with one decimal. Unfortunately, which is not sufficient for the interpretation of the results since available results show that milk permeates are below 0.1% lactates and the sweet whey permeates are exactly at 0.1%, with a Tolerance Value at 0.1%.

It is consequently unclear whether the lactate legal limit is a suitable classification criterion.

It was agreed to ask the laboratories immediately after the meeting for their limit of quantification, uncertainty and for their lactates results with three significant figures (if the results are still available in the laboratories).

If enough data is provided with 3 significant figures, a new data treatment will be performed and the results interpreted by the coordinators (before inclusion in the 2nd draft report and submission to the approval of the test participants).

The coordinators will re-evaluate based on new data the percentage of false negative and false positive results and will harmonise the results in this unit in the draft report (if needed, some laboratories will be contacted and asked for confirmation).

Analytical method by HPLC recommended:

In the test, lactates were analysed by the enzymatic method or by other method, specifically HPLC.

The alternative HPLC method gave similar results to the ISO 8069 method mentioned in the Regulation. Determination of lactates using HPLC method was recommended by the participants.

Such an HPLC method is indicated in the ILIADe record 340 “determination of organic acids by HPLC in fruit juices”, but the procedure is not linked to the record. To share good practices and harmonise procedure, the Czech Customs Technical Laboratory agreed to provide the CLEN community with their standard operating procedure for the determination of lactates by HPLC. Their procedure is provided as **Annex II**.

Attempt using chemometrics: lactates and nitrogen as parameters of interest

The test coordinators tried to discriminate between milk and whey permeates using linear discriminant analysis (LDA). Only results with satisfactory z-scores were kept and only the parameters with a sufficient number of participants were analysed. Some parameters such as lactates, CMP, smell and sulphates could not be used. After removing them, only **complete result sets**, i.e. only results from laboratories that responded to all the other variables were utilised (63 of the original 126 sets).

According to this multivariate analysis (slide 11 in Annex I), pH is obviously the relevant parameter for acid whey permeates, and lactates combined with nitrogen are the only other influencing parameters (see also slide 12 in Annex I). This observation is to be taken with care, as it is based on a small sample set size and so the model is currently insufficient and cannot be generalised.

In the future more authentic milk and sweet whey permeates should be analysed for lactates and nitrogen, to complete the data set of results and confirm the hypothesis.

3. III Discussion and interpretation of the results for the “Meursing” part

The discussion was based on the test 1st draft report, the results summary table and the coordinators presentation, in **Annex I**, slides 13 to 17.

Determination of the **additional code** following **milk fat content determination and milk proteins quantification** according to the methods of Regulation (EC) No 900/2008, as modified by Regulation (EU) No 2015/824 was analysed in depth.

3.III.1. Proteins

The laboratories performances were satisfactory regarding the Nitrogen determination.

Non-milk nitrogen estimation was mainly done according to the declared ingredients.

For the total protein calculation, most laboratories used the factor 6.38, appropriate for products relevant of the Meursing table, on the assumption that the products contain mostly milk proteins. Only few laboratories applied the factor 6.25 normally applied to products with “mixed proteins”. Nevertheless, as this parameter is not required for classification, the result is not significant. Regarding the milk protein calculation almost all laboratories used the correct factor showing a marked improvement in the harmonisation of the approach.

On the other hand, a precipitation method was used as an alternative method but resulted in a lower content of milk proteins and in a classification under a different additional code.

3.III.2. Milk fat

Total fat: a group of results for sample HPP showed a lower total fat content, resulting in a very-high coefficient of variation and lowering the assigned value below the one expected from the product's ingredients. Using the SBR (Schmid Bodzynski Ratzlaff) method is linked to higher results.

Butyric acid methyl ester (BAME) showed two apparent groups of results, most certainly due to differences in the reporting unit. Some laboratories reported BAME in % by weight of the total sample and others in % by weight of the fat content. Both possibilities are correct and relevant, but harmonisation is required, and to evaluate the laboratories performances only the BAME analysis expressed as % by weight of total fat is convenient.

The coordinator will harmonise the results in this unit in the draft report (if needed, some laboratories will be contacted and asked for confirmation).

Milk fat: The total fat and BAME results and calculations have led to a wide dispersion of milk fat values. Some laboratories even reported a milk fat content higher than the total fat.

The factor used for milk fat calculation is 25 (by default) or 50 (if the importer declared milk proteins >30% and milk fat content <6%).

For samples with milk proteins content near 30%, which was the case for sample MMS, and considering the measurement uncertainty, both factors could be accepted for calculation. For such products, the importer's declaration is essential for the laboratories to perform the control and to make an assessment on the compliance of the product.

3.III.3. Classification

Sample MMS

Sample MMS was a milk shake powder modified by adding fructose to the commercial product.

Two headings were suggested for the sample MMS: 2106 or 1901 depending on whether it is a food preparation based on products of headings 0401 to 0404.

Looking at the declared composition of the product, heading 1901 was suggested by a majority of laboratories considering that the characteristics of the product are based on milk products (note: a product made of a modified milk proteins alone would not be considered under heading 1901).

The two additional codes proposed, 7066 (suggested at 90%) and 7086, are considered as correct.

Sample HPP

Similarly, sample HPP (high protein preparation) was proposed in headings 2106 or 1901.

Most of the laboratories (more than 70%) classified HPP under heading 2106. "Whey protein concentrate" is not considered as belonging to Chapter 04 as it is a product of Chapter 35. Then if more than 80% of the total proteins in the sample belong to products of Chapter 35, the sample cannot be classified under Chapter 19, but falls into Chapter 21.

A quite similar case has been identified: a product, consisting of caseinate has been classified under heading 1901 as "not only containing dairy products belonging to 0401 to 0404". (Reg. No 440/91 amended by Reg. No 936/1999).

The participant laboratories came to two different classification suggestions because they did not know the proportion of proteins coming from the whey protein concentrate in the final product.

Both headings are acceptable according to the lack of information.

As regards the additional codes, the suggestions were more dispersed, the most prevalent additional codes suggested being 7190 and 7090, depending on the milk proteins content reported.

Compliance decision would be done taking into consideration both the results and their uncertainty.

Once again, the participants insisted on the fact that the situation is quite different when a declaration with a suggestion of code and additional code by the applicant is provided.

In the present case, if the product had been declared as heading 2016 or 1901, both would have been accepted (stated as compliant to the declaration) from the analytical determinations. This is also the case with the declaration of additional codes. The declarant's suggestion can be challenged only in those situations when analytical results (taking into account measurement uncertainty) prove it to be false.

4. Conclusions/recommendations/opinions

Differentiation of permeates

Acid whey permeates can easily be distinguished from other permeates thanks to their low pH.

The Laboratories currently have no analytical solution to differentiate sweet whey permeates from milk permeates; at best a suggestion can be made based on lactates, or lactates and nitrogen.

Several parameters clearly do not allow the differentiation (pH, organoleptic test, sulphates, calcium, phosphorus, ash, etc.).

CMP determination is only performed in one of the EU Customs Laboratories; even then the results obtained – to be confirmed – do not at present allow differentiation. The CMP analysis will be repeated by one laboratory, to reach a final conclusion on the method's suitability. If it is proven to be suitable, all labs might have to rely on them for CMP analysis.

An immunochemical method has been suggested in the meeting and will be further explored by the European Commission.

Lactates concentration, possibly in combination with nitrogen, is probably the only parameter which could differ between milk and sweet whey permeates. This hypothesis needs to be confirmed.

In particular, it is still uncertain whether the lactates legal limit is a suitable classification criterion. All test samples were below or near the 0.1% lactates limit value. The results will be re-evaluated as detailed in part 3.II.2 and part 5.).

The participants recommended using an HPLC method for the quantification of lactates in permeates.

Measuring part of the test

Improvements are observed in the laboratories practices. Most of the laboratories followed similar or equivalent approaches for milk fat and milk proteins determinations/calculations.

Nevertheless, there is still room for improvement: performing the analytical determination with the same recommended methods, applying the same and appropriate calculation factors.

The suggested classifications and additional codes (from the Meursing table) showed apparent dispersion mainly because some analytical results were close to the legal limit corresponding to different additional codes (i.e. one sample had a milk protein content of 30%). Most of the CN codes and additional codes suggestions were relevant, and would have been accepted in declarations, taking into account measurement uncertainties.

In such cases, the producer/importer's declaration is required before arriving at a statement regarding product compliance. The upcoming CLEN Project Group on compliance assessment is expected to give guidelines on situations such as these.

Recommendations would be appreciated by the CLEN to correctly classify goods containing milk products in Chapter 19 or Chapter 21 depending on their declared composition.

Miscellaneous – closing of the discussion meeting

The test coordinator thanked the new Action 2 Leader for his presence at the meeting and all the participants for their contributions to the discussions and interpretation of the test results.

5. Next steps

As detailed in part 3:

- The only laboratory experienced in CMP analysis will re-perform the determinations to confirm the results;
- The participants will be requested to re-send their results for lactates in permeates with 3 significant figures (together with information on their quantification limit and uncertainty); and the statistics on lactates will be performed again, interpreted and proposed to the participants.
- The coordinator will work on the table of the butyric acid methyl ester content to harmonise the units; the results will be re-evaluated and the interpretation proposed to the participants.

In addition, minor improvements will be done in the test report:

- The tables with the CN codes and additional codes, in the report part 3.4 (two first tables with the detailed results) will be corrected, as in the first draft report one was the copy of the other.
- In the description of the samples, the ranges of values provided for the two samples MMS and HPP for sucrose/invert sugar/isoglucose and starch/glucose as well as the fat content range for the permeates, will be added.

Taking into account the above tasks, the 2nd draft report will presumably be available only by the second half of March 2018.

This second draft report of the test will be provided to the test participant laboratories (and the meeting attendees) for comments or approval.

The contractor will prepare this 2nd draft report, except for the conclusions which will be drafted by the test coordinators.

Once the second draft report has been validated by the coordinators and participants, it will be submitted for final validation to DG TAXUD Head of Unit A4, before issuing the final report.

The final report of the CLEN ring test on milk products is expected to be available in May-June 2018.

6. Next meeting

There is no need for another meeting.

ANNEX I

Presentation of the CLEN ring test on milk products, by the test coordinators, during the 25 January 2018 discussion meeting



Customs Laboratories
European Network

Ring Test on Milk Products CLEN Action 2



ΑΑΔΕ

Ανεξάρτητη Αρχή
Δημοσίων Εσόδων

Dr Petroula Tarantili
Chemical Service of Central Macedonia
General Chemical State Laboratory of Greece
Dr Dimitra Triantafyllidou
DG TAXUD A.4

Ring Test on Milk Products



Customs Laboratories
European Network

Subject	<i>Permeates</i> - Differentiation of milk and whey permeates according to the new Additional Notes 3 and 4 to the Combined Nomenclature (Commission Implementing Regulation (EU) No 2016/534) <i>'Meursing' determinations</i> - Milk fat estimation from the method of Regulation (EC) No 900/2008 and milk protein quantification
Coordination	Ms D. Triantafyllidou, Ms P. Tarantili, Mr D. Pappas, Ms K. Grigoriadou General Chemical State Laboratory, Thessaloniki, Greece
Samples	<i>Permeates</i> - 6 commercial samples: milk permeate sweet whey permeate acid whey permeate (from bacteria fermentation) aged milk permeate 2 "unknown" permeates <i>Meursing</i> - 1 reconstituted sample (modified milk shake - MMS) 1 commercial sample (high protein preparation for beverage - HPP)
Parameters	<i>Permeates</i> - 11 parameters: pH, moisture content, organoleptic analysis, lactates, CMP, ash, lactose, nitrogen, sulphates, calcium, phosphorus <i>Meursing</i> - 2 main parameters: milk fat, milk proteins
Participants	26 Customs Laboratories 25 Customs Laboratories for the "Meursing" part, 21 Customs Laboratories for the "Permeates" part.

Test Targets-Part I & II

- The purpose of **Part I** of this ring test was the determination of the Meursing table code following the analysis of milk fat content and the quantification of milk protein using the methods of Regulation (EC) No 900/2008, as modified by Regulation (EU) 2015/824.
- The purpose of **Part II** was to differentiate milk (0404 90) and whey (0404 10) permeates according to the new additional notes 3 and 4 to the Combined Nomenclature (Commission Implementing Regulation (EU) No 2016/534), the duty being much lower for whey permeates.

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Ring Test on Milk Products

Part I- Parameters

Parameters	Methods	Samples
Milk fat	Method described in Regulation (EC) No. 900/2008 modified by Regulation (EU) No. 2015/824	Sample MMS - Modified Milk Shake
Milk proteins	Method described in Regulation (EC) No. 900/2008 modified by Regulation (EU) No. 2015/824 (Kjeldahl method and calculation)	Sample HPP - High Proteins Preparation for beverage
"Conclusion"	8-digit CN code and additional code	

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Ring Test on Milk Products



Part II- Parameters

	Parameters	Methods to be used	Milk permeates	Whey permeates
Sample Sample 1 Milk permeate Sample 2 Sweet whey permeate Sample 4 Acid whey permeate from bacteria activity Sample 5 Unknown sample No.1 Sample 6 Unknown sample No.2 Sample 7 Aged milk permeate	pH			
	Moisture content	Regulation (EC) No 273/2008 - Annex XVIII. Drying at 102°C until stable weight		
	Organoleptic	Regulation (EU) 2016/534	slightly sour smell	milky smell
	Lactates	ISO 8069, Regulation (EC) No 273/2008 and (EU) 2016/534	-	max. 0,1 %
	CMP (facultative)	Regulation (EC) No 273/2008 (Annex XII, Annex XIII)	Absence	Presence
	Ash	Ash at 550°C		
	Lactose	Regulation (EC) No 273/2008		
	Nitrogen	Regulation (EC) No 900/2008		
	Sulphates	Detection of sulphates by the addition of Barium Chloride		
	Calcium	Laboratory routine method	-	+
	Phosphorus	Laboratory routine method	-	+
	CN code	8-digit CN code	0404 10	0404 90

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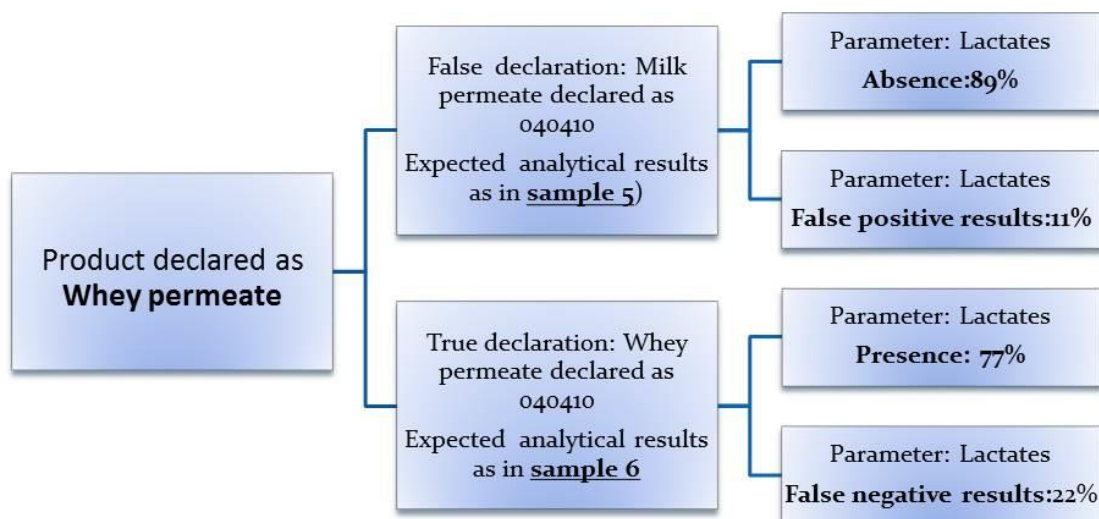
Summary table of the results on permeates:

		Sample 1		Sample 5		Sample 7		Sample 2		Sample 6		Sample 4	
		Milk permeate		Unknown permeate 1 Milk permeate		Aged milk permeate		Sweet whey permeate		Unknown permeate 2 Sweet whey permeate		Acid whey permeate by bacteria activity	
	Unit	X	VT	X	VT	X	VT	X	VT	X	VT	X	VT
pH	%	6,3	0,1	6,3	0,2	6,3	0,2	6,3	0,1	6,2	0,1	4,4	0,1
Moisture content	%	1,4	0,4	1,4	0,6	1,5	0,6	1,3	0,4	1,5	0,4	5,9	4,0
Lactates : Enzymatic method	%	-	-	-	-	-	-	0,1	0,1	0,1	0,1	10,0	0,6
Other method	%	-	-	-	-	-	-	-	-	-	-	-	-
Ash	%	7,35	0,48	7,41	0,36	7,34	0,38	7,45	0,66	7,34	0,56	11,99	0,54
Lactose : HPLC method	%	83,9	6,0	83,2	6,6	83,9	6,8	83,8	7,0	84,4	7,2	58,2	5,2
Other method	%	-	-	-	-	-	-	-	-	-	-	-	-
Nitrogen	%	0,533	0,038	0,522	0,046	0,543	0,056	0,486	0,048	0,439	0,046	0,627	0,066
Calcium	mg.kg ⁻¹	2927,4	1392,0	3414,1	1785,0	3332,1	1189,8	2630,0	1114,6	2794,3	1079,4	18097,7	4463,4
Phosphorus	mg.kg ⁻¹	5402,6	805,4	5617,8	1033,2	5212,3	681,6	5112,4	1165,0	5425,0	900,2	11392,7	1728,8
Sulphates	Yes/no	Presence (69%)		Presence (69%)		Presence (69%)		Presence (62%)		Presence (69%)		Absence (62%)	
CMP		No (3/3)		No (2/3)		No (4/4)		No (2/3)		No (3/3)		No (3/3)	
Organoleptic		Milky (77%)		Milky (62%)		Sour (87%)		Milky (72%)		Milky (64%)		Milky (73%)	
CN codes		0404 90 (93%) 04 90 21 (80%)		0404 90 (70%) 0404 90 21 (60%)		0404 90 (86%) 0404 90 21 (73%)		0404 10 (86%) 0404 10 02 (73%)		0404 90 (60%) 0404 90 21 (50%)		0404 10 (100%) 0404 10 02 (73%)	

Correlation between Sample Category and lactates analysis results

Identification	1.Milk permeate	5.Milk permeate	7. Aged milk permeate	2.Whey permeate	6.Whey Permeate	4.Whey permeate
Correct	100% Absence	89% Absence	100% Absence	77,8% Presence	77,8% Presence	100% Presence
False positive	0	11%	0			
False negative				22,2%	22,2%	0
Classification using all parameters	0404 90 (93%)	0404 90 (70%)	0404 90 (86%)	0404 10 (86%)	0404 10 (40%) 0404 90 (60%)	0404 10 (100%)

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Part II: Chemometric analysis of results

Chemometric tool used:

Linear Discriminant Analysis (LDA)

Pattern Recognition and Machine Learning classification and discrimination multivariate methodology

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LINEAR DISCRIMINANT ANALYSIS Discrimination as Milk/Whey permeate Critical parameters pH, nitrogen

Classification Results					
		SampleType	Predicted Group Membership		Total
			Milk permeate	Whey permeate	
Cross-validated ^b	Count	Milk permeate	59	4	63
		Whey permeate	15	48	63
	%	Milk permeate	93.7	6.3	100.0
		Whey permeate	23.8	76.2	100.0

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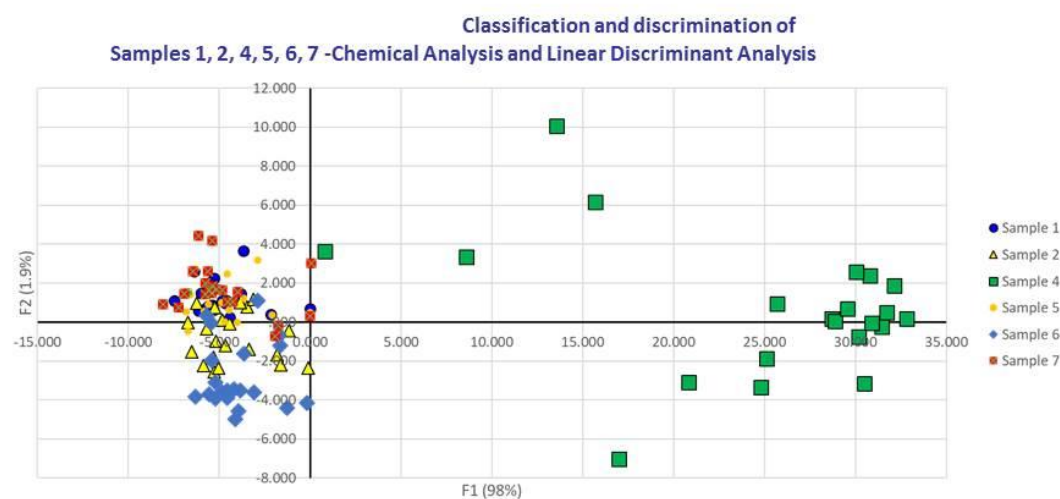
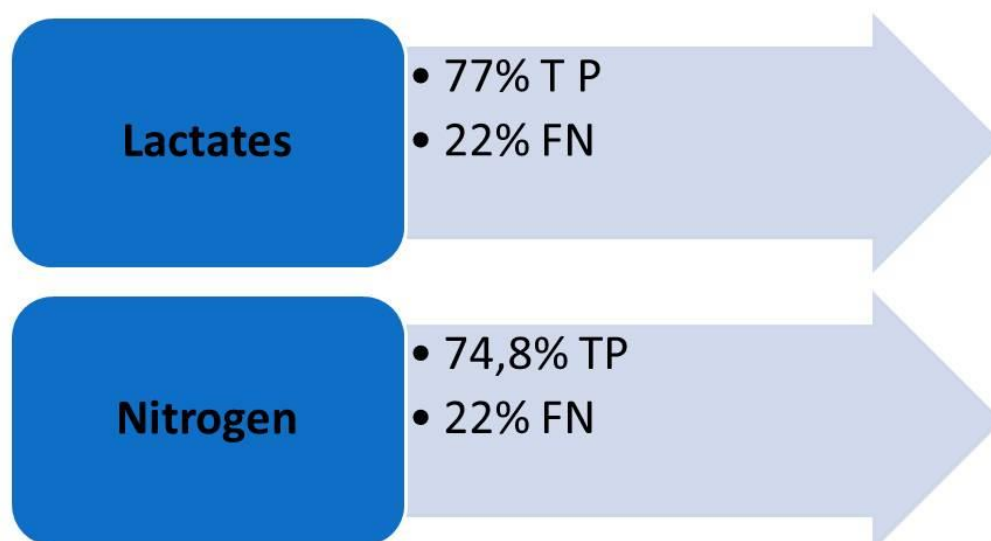


Figure 1 :Discriminant scatterplot for Samples 1, 2, 4, 5, 6, 7

Sample 1 - Milk permeate
 Sample 2 - Sweet whey permeate
 Sample 4 - Acid whey permeate
 Sample 5 - Unknown permeate (milk permeate)
 Sample 6 - Unknown permeate (sweet whey permeate)
 Sample 7 - Aged milk permeate

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Analysis of Lactates and Nitrogen – “Selectivity” for correct identification of whey permeate



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Meursing Part: Methods used

Parameter	Method used
Total fat	Reg 900/2008: method based on extraction with light petroleum, preceded by hydrolysis with hydrochloric acid –SOXHLET (Method indicated in the ring test) Automated methodologies Schmid-Bondzynski-Ratzlaff
Milk Proteins	Reg 900/2008: Kjeldahl for total Nitrogen, estimation and subtraction of non-milk nitrogen, multiplication by 6.38 Other methods: Precipitation and Kjeldahl process.

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Meursing Part: Protein

Comments on analytical results	
Accuracy and precision data	The nitrogen analysis results were satisfactory.
Total protein estimation	Most labs used the 6.38 based on the assumption that the sample contained mostly milk protein. In previous ring tests a factor of 6.25 has been used for mixed proteins but it does influence classification.
Non-milk nitrogen estimation	Non-milk protein nitrogen were estimated in most cases based on the ingredients list. Most labs agree that their contribution to total nitrogen was minimal. In both cases, factors other than 6.38 that is defined in the Regulation have been used.
Milk protein nitrogen determination	For both cases, factors other than 6.38 that is defined in the Regulation have been used. The use of precipitation methods as a alternative to the Regulation method resulted in an underestimation of milk protein content.

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Meursing Part: Milk fat

	the statistical processing of the results be re-introduced and the result with "BIAS " omitted.
Total fat	There is a group of results in sample HPP with low total fat content e.g. resulting in a very high CV value and lowering the assigned value below that expected from the product's ingredients. The use of the Schmid Bodzynski Ratzlaff method is linked to higher results.
Butyric acid methyl ester	Sample MMS has a very low content, close to the labs' LOQ resulting in a high CV. This is however not important for classification. For sample MMS there is a robust group of values close to the average. However there is an extremely wide range of values resulting in a high CV. There are values that exceed the 4% reference value of BAME in milk. There are also low values of BAME not linked to low milk fat values pointing to inconsistencies in reporting.
Milk fat	The total fat and BAME results have led to a wide dispersion of milk fat values. In some cases milk fat values exceeded the total fat.
Factor used for milk fat calculation	25 (default) or 50 (if importer declared MP>30% AND MF<6%) There might be an issue for samples with milk protein content close to 30% (e.g. sample MMS). If the analytical result is $\geq 30\%$ the factor to be used is 50. If the analytical result is $< 30\%$ account should be taken of the importer's declaration, as well as the uncertainty of the method.

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Classification: MMS

MMS

CN code: 2106 (57%), 1901 (43%) depending on whether it is a food preparation based on products of 0401 to 0404.

Additional code

Additional code	Frequency	Analytical ranges	Duty
7066	70%	Milk fat<1.5% MP< 30%	103,32 €
7086	13%	Milk fat<1.5% MP> 30%	187,67 €

The importer's declaration and the uncertainty need to be taken into consideration

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Classification: HPP

CN code: 2106 (71%), 1901 (43%) depending on whether it is a food preparation based on products of 0401 to 0404. Product based on WPC (0404 since MP<80%). A similar product was classified under 1901 in the previous ring-test. A classification opinion places a similar product (albite one where milk proteins were less than 50%).

Additional code	Frequency	Analytical ranges	Duty
7066 7090 7180 7185 7190	70%	Milk protein >30%	177,61-188,02 €
7270	13%	Milk protein <30%	87,73 €

The importer's declaration and the uncertainty need to be taken into consideration before arriving at a conclusion.

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Possible further development

Permeates

Use of alternate methods for lactates.

Further investigation of nitrogen, lactates & pH parameters for permeate classification via a feasibility study or ring test on additional milk and whey permeate samples.

Possible introduction of novel statistical methods for difficult classification decisions.

Meursing

Re-evaluation of submitted lab results and repeat of statistical treatment

General

Emphasis and further information needed on procedures to be followed for:

- analysis and result calculation (e.g. non-protein nitrogen estimation)
- utilization of analytical results for classification decision (use of uncertainty vis-à-vis the declarant's statements).

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Acknowledgments

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Ms Stella Synouri &
Ms Christina Vlachou

ANNEX II

Standard Operating Procedure “Determination of organic acid by HPLC” provided by the Czech Republic Customs Technical Laboratory.

Recommended for the determination of lactates during the 25 January 2018 discussion meeting of the CLEN test on milk products

Column: IEX WATREX 250x8 mm (or 300x8 mm), 8 µm particles in H⁺ form (similar column produce for example Bio-Rad, Shodex, PolymerLab, Merck)

Detectors: UV (210 nm), DAD (210 nm), CD

Mobil phase: for UV or DAD - 0,005 mol/L H₂SO₄

Mobil phase: for CD - 0,001 mol/L H₂SO₄

Temperature: 30 – 80 °C, for lactic acid 80 °C

Flow rate: 0,8 ml/min

Standard injection: 10 µl

LOD lactic acid: 50 mg/L injected sample

LOQ lactic acid: 100 mg/L injected sample

Analysis of milk permeate

10 g permeate is dissolved in 100 ml flask in 70 ml of water and sonicated for 30 min in 40 °C bath.

After cooling to laboratory temperature is flask filled to 100 ml by water, mixed and sample is filtered through 0,045 µm filter.

20 µl of sample is injected on the column.

By this condition we have this LOD and LOQ:

LOD for lactic acid: 0,025 %

LOQ for lactic acid: 0,050 %