Commission Implementing Regulation (EU) 2016/1240 of 18 May 2016 laying down rules for the application of Regulation (EU) No 1308/2013 of the European Parliament and of the Council with regard to public intervention and aid for private storage (Text with EEA relevance)

ANNEX I

CEREALS

[[F1]A1.

This Annex does not apply in relation to public intervention schemes in England, otherwise than in connection with exceptional market conditions which are the subject of a declaration under section 20 of the Agriculture Act 2020.**]**

[[F2]A2. This Annex ceases to apply in relation to public intervention schemes in Scotland, otherwise than in connection with public intervention measures which the appropriate authority takes under Article 219(1) of Regulation (EU) No 1308/2013, for a period of five years beginning on 1 July 2023.**]**

Textual Amendments

F2 Annex 1 para. A2 inserted (S.) (1.7.2023) by The Public Intervention and Private Storage Aid (Amendment and Suspension) (Scotland) Regulations 2023 (S.S.I. 2023/150), regs. 1, **9(15)** (with reg. 10)

PART I 1.DEFINITION OF MATTER OTHER THAN BASIC CEREALS OF UNIMPAIRED QUALITY1.1.Broken grains

For durum wheat, common wheat and barley, the definition of ‘broken grains’ is that contained in standard EN 15587.

For maize, the definition of ‘broken grains’ is that contained in standard EN 16378.
1.2.Grain impurities(a)Shrivelled grains

For durum wheat, common wheat and barley, the definition of ‘shrivelled grains’ is that contained in standard EN 15587. [F3]...

Textual Amendments

F3 Words in Annex 1 Pt. 1 para. 1.2(a) omitted (31.12.2020) by virtue of The Market Measures Payment Schemes (Amendment) (EU Exit) Regulations 2019 (S.I. 2019/823), regs. 1, **6(44)**; 2020 c. 1, **Sch. 5 para. 1(1)**

‘Shrivelled grains’ does not apply to maize.
(b)Other cereals

For durum wheat, common wheat and barley, the definition of ‘other cereals’ is that contained in standard EN 15587.

For maize, the definition of ‘other cereals’ is that contained in standard EN 16378.
(c)Grains damaged by pests

For durum wheat, common wheat and barley, the definition of ‘grains damaged by pests’ is that contained in standard EN 15587.

For maize, the definition of ‘grains damaged by pests’ is that contained in standard EN 16378.
(d)Grains in which the germ is discoloured

For durum wheat and common wheat, the definition is that contained in standard EN 15587.

‘Grains in which the germ is discoloured’ does not apply to barley or maize.
(e)Grains overheated during drying

For durum wheat, common wheat and barley, the definition of ‘grains overheated during drying’ is that contained in standard EN 15587.

For maize, the definition of ‘grains overheated during drying’ is that contained in standard EN 16378..
(f)Mottled grains

For durum wheat, the definition of ‘mottled grains’ is that contained in standard EN 15587.

‘Mottled grains’ does not apply to common wheat, barley or maize.
1.3.Sprouted grains

For durum wheat, common wheat and barley, the definition of ‘sprouted grains’ is that contained in standard EN 15587.

For maize, the definition of ‘sprouted grains’ is that contained in standard EN 16378.
1.4.Miscellaneous impurities(a)Extraneous seeds

For durum wheat, common wheat and barley, the definition of ‘extraneous seeds’ is that contained in standard EN 15587.

For maize, the definition of ‘extraneous seeds’ is that contained in standard EN 16378.

‘Noxious seeds’ means seeds which are toxic to humans and animals, seeds hampering or complicating the cleaning and milling of cereals and seeds affecting the quality of products processed from cereals.
(b)Damaged grains

For durum wheat, common wheat and barley, the definition of ‘damaged grains’ is that contained in standard EN 15587.

For maize, the definition of ‘damaged grains’ is that contained in standard EN 16378.

In standard EN 15587, for durum wheat, common wheat and barley, the definition of ‘grains affected by fusariosis’ is included in that of ‘damaged grains’.
(c)Extraneous matter

For durum wheat, common wheat and barley, the definition of ‘extraneous matter’ is that contained in standard EN 15587.

For maize, the definition of ‘extraneous matter’ is that contained in standard EN 16378.
(d)Husks (cob fragments in the case of maize)(e)Ergots(f)Decayed grains

For durum wheat and common wheat, the definition of ‘decayed grains’ is that contained in standard EN 15587.

‘Decayed grains’ does not apply to barley or maize.
(g)Impurities of animal origin.1.5.Live pests1.6.Mitadiné grains

Mitadiné grains of durum wheat are grains whose kernels cannot be regarded as entirely vitreous. They are defined in standard EN 15585.

2. SPECIFIC FACTORS TO TAKE INTO CONSIDERATION FOR EACH TYPE OF CEREAL FOR THE DEFINITION OF IMPURITIES

2.1. **Durum wheat**

‘Grain impurities’ means shrivelled grains, grains of other cereals, grains damaged by pests, grains in which the germ is discoloured, mottled grains and grains overheated during drying.

‘Miscellaneous impurities’ means extraneous seeds, damaged grains (including grains affected by fusariosis), extraneous matter, husks, ergot, decayed grains and impurities of animal origin.

2.2. **Common wheat**

‘Grain impurities’ means shrivelled grains, grains of other cereals, grains damaged by pests, grains in which the germ is discoloured (only where the content exceeds 8 %) and grains overheated during drying.

‘Miscellaneous impurities’ means extraneous seeds, damaged grains (including grains affected by fusariosis), extraneous matter, husks, ergot, decayed grains and impurities of animal origin.

2.3. **Barley**

‘Grain impurities’ means shrivelled grains, grains of other cereals, grains damaged by pests and grains overheated during drying.

‘Miscellaneous impurities’ means extraneous seeds, damaged grains (including grains affected by fusariosis), extraneous matter, husks and impurities of animal origin.

2.4. **Maize**

‘Grain impurities’ means grains of other cereals, grains damaged by pests and grains overheated during drying.

‘Miscellaneous impurities’ means extraneous seeds, damaged grains, extraneous matter, cob fragments and impurities of animal origin.

PART II Methods used for determining the quality of cereals offered or tendered for, or placed in, intervention

Pursuant to Article 4, the following methods are to be used to determine the quality of cereals offered or tendered for, or placed in, intervention:

(a) reference method for determining matter other than basic cereals of unimpaired quality:

  (i) for common wheat, durum wheat and barley: standard EN 15587,

  (ii) for maize: standard EN 16378;

(b) reference method for determining the moisture content:

  (i) for maize: standard EN ISO 6540,

  (ii) for cereals other than maize: standard EN ISO 712, or an infrared technology-based method complying with standard EN 15948.

In the event of a dispute, only the results resulting from applying standard EN ISO 6540 for maize and standard EN ISO 712 for cereals other than maize are to be considered valid;

(c) reference method for determining the non-stickiness and machinability of the dough obtained from common wheat: that set out in Part III of this Annex;

(d) reference method for determining the protein content in durum wheat and ground common wheat: that set out in:

(i) standard EN ISO 20483, or

(ii) standard CEN ISO/TS 16634-2.

In the event of a dispute, only the results obtained from applying standard EN ISO 20483 are to be considered valid;

(e) reference method for determining the Zeleny index of ground common wheat: that set out in standard EN ISO 5529;

(f) reference method for determining the Hagberg falling number (amylase activity test): that set out in standard EN ISO 3093;

(g) reference method for determining the rate of loss of the vitreous aspect of durum wheat: that set out in standard EN 15585;

(h) reference method for determining the specific weight: that set out in standard EN ISO 7971/3;

(i) sampling and analysis methods for establishing the rate of mycotoxins: those referred to in the Annex to Commission Regulation (EC) No 1881/2006[(1)] and set out in Annexes I and II to Commission Regulation (EC) No 401/2006[(2)].

PART III Method for determining the non-stickiness and machinability of the dough obtained from common wheat1.Title

Method for test baking of wheat flour.
2.Scope

The method is applicable to flour, experimentally milled from wheat for the production of yeast-raised bread.
3.Principle

Dough is made from flour, water, yeast, salt and sucrose, in a specified mixer. After dividing and rounding, the pieces are given 30 minutes' rest; they are moulded, placed on baking sheets and baked after a final proof of fixed duration. Dough-handling properties are noted. The loaves are judged by volume and height.
4.Ingredients4.1.Yeast

Active dry yeast of type Saccharomyces cerevisiae DHW-Hamburg-Wansbeck or a product having the same characteristics.
4.2.Tap water4.3.Sugar-salt-ascorbic acid solution

Dissolve 30 ± 0,5 g of sodium chloride (commercial grade), 30 ± 0,5 g of sucrose (commercial grade), and 0,040 ± 0,001 g ascorbic acid in 800 ± 5 g of water. Prepare fresh daily.

4.4.Sugar solution

Dissolve 5 ± 0,1 g sucrose (commercial grade) in 95 ± 1 g of water. Prepare fresh daily.
4.5.Enzyme active malt flour

Commercial grade.
5.Equipment and apparatus5.1.Baking room

Controlled to maintain a temperature of 22 to 25 °C.
5.2.Refrigerator

For maintaining a temperature of 4 ± 2 °C.
5.3.Balance

Maximum load 2 kg, accuracy 2 g.
5.4.Balance

Maximum load 0,5 kg, accuracy 0,1 g.
5.5.Analytical balance

Accuracy $0{,}1 \times 10^{-3}$ g.
5.6.Mixer

Stephan UMTA 10, with mixing arm model ‘Detmold’ (Stephan Soehne GmbH) or similar equipment having the same characteristics.
5.7.Proving cabinet

Controlled to maintain a temperature of 30 ± 1 °C.
5.8.Open plastic boxes

Made from polymethylmethacrylate (Plexiglas, Perspex). Inside dimensions: 25 × 25 × 15 cm height, wall thickness 0,5 ± 0,05 cm.
5.9.Square plastic sheets

Made from polymethylmethacrylate (Plexiglas, Perspex). At least 30 × 30 cm, thickness 0,5 ± 0,05 cm.
5.10.Moulder

Brabender ball homogeniser (Brabender OHG) or similar equipment having the same characteristics.
6.Sampling

According to standard EN ISO 24333.
7.Procedure7.1.Determination of water uptake

Determine the water absorption according to ICC Standard No 115/1.
7.2.Determination of malt flour addition

Determine the ‘falling number’ of the flour according to standard EN ISO 3093. If the ‘falling number’ is higher than 250, determine the malt flour addition required to bring it within the range 200 to 250, using a series of mixtures of the flour with increasing quantities of malt flour (point 4.5). If the ‘falling number’ is lower than 250, no malt flour is required.
7.3.Reactivation of active dry yeast

Adjust the temperature of the sugar solution (point 4.4) to 35 ± 1 °C. Pour one part by weight of the active dry yeast into four parts by weight of this tempered sugar solution. Do not stir. Swirl if necessary.

Allow to stand for 10 ± 1 minute, then stir until a homogeneous suspension is obtained. Use this suspension within 10 minutes.

7.4.Temperature adjustment of the flour and the dough liquid

The temperature of the flour and the water must be adjusted to give a dough temperature of 27 ± 1 °C after mixing.

7.5.Dough composition

Weigh, with a precision of 2 g, 10 y/3 g flour on as-is moisture basis (corresponding to 1 kg flour on a 14 % moisture basis), in which 'y' is the quantity of flour used in the farinograph test (see ICC Standard No 115/1).

Weigh, with a precision of 0,2 g, the quantity of malt flour necessary to bring the 'falling number' within the range 200 to 250 (point 7.2).

Weigh 430 ± 5 g sugar-salt-ascorbic acid solution (point 4.3) and add water to a total mass of (x – 9) 10 y/3 g, (see point 10.2) in which 'x' is the quantity of water used in the farinograph test (see ICC Standard No 115/1). This total mass (usually between 450 and 650 g) must be achieved with a precision of 1,5 g.

Weigh 90 ± 1 g yeast suspension (point 7.3).

Note the total mass of the dough (P), which is the sum of the masses of flour, sugar-salt-ascorbic acid solution plus water, yeast suspension and malt flour.

7.6.Mixing

Before starting, bring the mixer to a temperature of 27 ± 1 °C by use of a suitable quantity of tempered water.

Place the liquid dough ingredients in the mixer and place the flour plus malt flour on top.

Start the mixer (speed 1, 1 400 rev/min), and allow to run for 60 seconds. Twenty seconds after the start of mixing, turn the scraper attached to the lid of the mixing bowl two revolutions.

Measure the temperature of the dough. If it is outside the range 26 to 28 °C, discard the dough and mix a new one after adjustment of ingredient temperatures.

Note dough properties using one of the following terms:

— non-sticky and machinable, or

— sticky and non-machinable.

To be considered 'non-sticky and machinable' at the end of mixing, the dough should form a coherent mass which hardly adheres to the sides of the bowl and spindle of the mixer. It should be possible to collect the dough by hand and remove it from the mixing bowl in a single motion without noticeable loss.

7.7.Dividing and rounding

Weigh, with precision of 2 g, three pieces of dough according to the formula:

p = 0,25 P, where:
p = mass of scaled dough piece,
P = total mass of dough.

Immediately round the pieces for 15 seconds in the moulder (point 5.10) and place them for 30 ± 2 minutes on the square plastic sheets (point 5.9), covered by the inverted plastic boxes (point 5.8) in the proving cabinet (point 5.7).

Do not use dusting flour.

7.8.Moulding

Bring the pieces of dough on the plastic sheets, covered by the inverted boxes, to the moulder (point 5.10), and re-round each piece for 15 seconds. Do not remove cover from a piece of dough until immediately before rounding. Note dough properties again, using one of the following terms:

(a) non-sticky and machinable, or

(b) sticky and non-machinable.

To be considered as 'non-sticky and machinable' the dough should adhere hardly, or not at all, to the sides of the chamber so that it can freely rotate around itself and form a regular ball during the operation of the machine. At the end of the operation the dough should not stick to the sides of the dough-moulding chamber when the lid of the chamber is raised.

8.Test report

The test report shall mention:

(a) dough-handling properties at the end of mixing, and at moulding,

(b) the 'falling number' of the flour without addition of malt flour,

(c) any anomalies observed.

(d) the method used,

(e) all details required for the identification of the sample.

9.General remarks9.1.The formula for the calculation of the quantity of dough liquid is based on the following considerations:

Addition of x ml water to the equivalent of 300 g flour at 14 % moisture produces the required consistency. As in the baking test 1 kg of flour (14 % moisture basis) is used, whereas x is based on 300 g of flour, for the baking test x divided by three and multiplied by 10 g of water is needed, so 10 x/3 g.

The 430 g sugar-salt-ascorbic acid solution contains 15 g salt and 15 g sugar. This 430 g solution is included in the dough liquid. So to add 10 x/3 g water to the dough, (10 x/3 + 30) g dough liquid composed of the 430 g sugar-salt-ascorbic acid solution and an additional quantity of water must be added.

Although part of the water added with the yeast suspension is absorbed by the yeast, this suspension also contains 'free' water. It is arbitrarily supposed that 90 g yeast suspension contains 60 g 'free' water. The quantity of the dough liquid must be corrected for this 60 g of 'free' water in the yeast suspension, so 10 x/3 plus 30 minus 60 g must finally be added. This can be rearranged as follows: (10 x/3 + 30) – 60 = 10 x/3 – 30 = (x/3 – 3) 10 = (x – 9) 10/3, the formula given in point 7.5. If, for example, a water addition x in the farinograph test was found of 165 ml, this value must be substituted in this formula, so to the 430 g sugar-salt-ascorbic acid solution water must be added to a total mass of:

(165 – 9) 10/3 = 156 × 10/3 = 520 g.

9.2.The method is not directly applicable to wheat. The procedure to be followed for characterising the baking properties of wheat is as follows:

Clean the wheat sample, and determine the moisture content of the cleaned wheat. If the moisture content is within the range 15,0 % to 16,0 %, do not temper the wheat. If the moisture content is outside this range, adjust the moisture content to 15,5 ± 0,5 %, at least three hours prior to milling.

Mill the wheat into flour using a Buehler laboratory mill MLU 202 or a Brabender Quadrumat Senior mill or similar equipment having the same characteristics.

Choose a milling procedure that yields a flour of minimum 72 % extraction, with an ash content of 0,50 to 0,60 % on dry matter basis.

Determine the ash content of the flour according to Annex II to Commission Regulation (EU) No 234/2010[3] and the moisture content according to this Regulation. Calculate the extraction rate by the equation:

$E = (((100 – f) F)/(100 – w) W) \times 100\ \%$

where:

E	=	extraction rate,
f	=	moisture of the flour,
w	=	moisture content of the wheat,
F	=	mass of flour produced with moisture content f,
W	=	mass of wheat milled with moisture content w.

Note: Information concerning the ingredients and equipment to be used is published in Document T/77,300 of 31 March 1977 from the Instituut voor Graan, Meel en Brood, TNO — Postbus 15, Wageningen, Netherlands.

PART IV Methodology of sampling and analyses for cereals1.

For each lot of cereals, the quality characteristics shall be established on the basis of a representative sample of the lot offered, consisting of samples taken at the rate of once every delivery for at least every 60 tonnes.

2.

The reference methods to be used for determining the quality of cereals offered or tendered for, or placed in, intervention are those set out in Parts I, II and III of this Annex.

3.

In cases of dispute, the paying agency shall have the necessary tests on the cereals in question carried out again, the cost being met by the losing party.

PART V Price increases and reductionsTable IPrice increases for moisture content for cereals other than maizeMoisture content(%)Increases(EUR/tonne)Less than 12.5 to 120,5Less than 12 to 11,51Less than 11,51.5Price increases for moisture content for maizeMoisture content(%)Increases(EUR/tonne)Less than 12 to 11,50,5Less than 11,51Table IIPrice reductions for moisture content for cereals other than maizeMoisture content(%)Reduction(EUR/tonne)More than 13,0 to 13,50,5More than 13,5 to 14,01,0More than 14,0 to 14,51,5Price reductions for moisture content for maizeMoisture content(%)Reduction(EUR/tonne)More than 12,5 to 13,00,5More than 13,0 to 13,51,0

TABLE III

Price increases for protein content of common wheat

Protein content[a](N × 5,7)	Price increase(EUR/tonne)
More than 12,0	2,5

a As % of dry matter.

TABLE IV

Price reductions for protein content for common wheat

Protein content[a](N × 5,7)	Price reduction(EUR/tonne)
Less than 11,5 to 11,0	2,5

a As % of dry matter.

PART VI Calculation of prices increases and reductions

The price adjustments provided for in Article 26(1) shall be expressed in euro per tonne for offers or tenders for intervention by multiplying the price referred to in that Article by the sum of the established percentage increases or reductions, as follows:

(a) where the moisture content of cereals offered or tendered for intervention is less than 12,0 % for maize and 12,5 % for other cereals, the price increases to be applied shall be those listed in Table I of Part V of this Annex. Where the moisture content of these cereals offered or tendered for intervention is higher than 12,5 % for maize and 13,0 % for other cereals, the price reductions to be applied shall be those listed in Table II of Part V of this Annex;

(b) where the protein content of common wheat is higher than 12,0 %, the increases to be applied shall be those listed in Table III of Part V of this Annex. Where the protein content of common wheat is less than 11,5 %, the reductions to be applied shall be those listed in Table IV of Part V of this Annex.

ANNEX II

RICE

[[F4]A1.

This Annex does not apply in relation to public intervention schemes in England, otherwise than in connection with exceptional market conditions which are the subject of a declaration under section 20 of the Agriculture Act 2020.**]**

[[F5]A2. This Annex ceases to apply in relation to public intervention schemes in Scotland, otherwise than in connection with public intervention measures which the appropriate authority takes under Article 219(1) of Regulation (EU) No 1308/2013, for a period of five years beginning on 1 July 2023.**]**

Textual Amendments

F5 Annex 2 para. A2 inserted (S.) (1.7.2023) by The Public Intervention and Private Storage Aid (Amendment and Suspension) (Scotland) Regulations 2023 (S.S.I. 2023/150), regs. 1, **9(15)** (with reg. 10)

PART I

Methodology of sampling and analyses for paddy rice

1. With a view to verifying the quality requirements as laid down in Part I of Annex II to Delegated Regulation (EU) 2016/1238, samples shall be taken by the paying agency in the presence of the operator or his/her duly authorised agent.

Three representative samples, each weighing a minimum of one kilogram, shall be collected. One each shall go to:

(a) the operator;

(b) the storage place where takeover is to take place;

(c) the paying agency.

To make up the representative samples, the number of individual samples to be taken shall be obtained by dividing the quantity of the lot by 10 tonnes. Each individual sample shall weigh the same. The representative samples shall be made up of the sum of the individual samples, divided by three.

The quality requirements shall be verified using the representative sample intended for the store where takeover is to take place.

2. Representative samples shall be taken of each part-delivery (by lorry, barge, railway wagon) under the conditions laid down in point 1.

Before its entry into the intervention store the examination of each part-delivery can be restricted to a check of the moisture content and impurity level and verification that no live insects are present. However, if it later becomes apparent when the check is finalised that a part-delivery does not satisfy the minimum quality requirements, the concerned quantity shall be refused for takeover. If the paying agency is able to check all the minimum quality requirements for each part-delivery before it enters the store, it shall refuse takeover of any part-delivery that fails to satisfy these requirements.

3. The control of the radioactivity level is performed only if the situation so requires and for a limited period.

4. In cases of dispute, the paying agency shall have the necessary tests on the paddy rice in question carried out again, the cost being met by the losing party.

A new analysis is performed by a laboratory recognised by the paying agency on the basis of a new representative sample made up, in equal parts, by samples preserved by the operator and by the paying agency. In cases where there were part-deliveries of the lot tendered, the result is given by the weighted average of the results of analyses of new representative samples taken for each of the part-deliveries.

PART II

Prices increases and reductions

1. The price adjustments provided for in Article 26(1) shall be expressed in euro per tonne and apply to tenders for intervention by multiplying the price referred to in that Article by the sum of the established percentage increases in Tables I, II and III of this Part.

2. The price increases and decreases shall apply on the basis of the weighted average of the test results on the representative samples as defined in Part I of this Annex.

TABLE I

Price increases for moisture content

Moisture content(%)	Increases(EUR/tonne)
Less than 12,5 to 12	0,75
Less than 12 to 11,5	1,5

TABLE II

Price reductions for moisture content

Moisture content(%)	Reduction(EUR/tonne)
More than 13,5 to 14,0	0,75
More than 14,0 to 14,5	1,5

TABLE III

Price increases relating to milling yield

Yield of whole-grain milled paddy rice	Price increases per yield point[a]
Above the basic yield	0,75 % increase
Overall yield of milled paddy rice	Price increases per yield point
Above the basic yield	0,60 % increase

a To be applied where the milling yield of the rice differs from the basic milling yield for the variety concerned as set out in Part II of Annex II to Delegated Regulation (EU) 2016/1238.

ANNEX III

BEEF

[[F6]A1.

This Annex does not apply in relation to public intervention schemes in England, otherwise than in connection with exceptional market conditions which are the subject of a declaration under section 20 of the Agriculture Act 2020.**]**

[[F7]A2. This Annex ceases to apply in relation to public intervention schemes in Scotland, otherwise than in connection with public intervention measures which the appropriate

authority takes under Article 219(1) of Regulation (EU) No 1308/2013, for a period of five years beginning on 1 July 2023.**]**

Textual Amendments

F7 Annex 3 para. A2 inserted (S.) (1.7.2023) by The Public Intervention and Private Storage Aid (Amendment and Suspension) (Scotland) Regulations 2023 (S.S.I. 2023/150), regs. 1, **9(15)** (with reg. 10)

PART I

Conditions and controls for taking over

1. Products delivered shall be taken over subject to verification by the paying agency that they comply with the requirements laid down in Part I of Annex III to Delegated Regulation (EU) 2016/1238. In particular, a systematic check of the presentation, classification, weight and labelling of each carcass, half-carcass and quarter delivered shall be undertaken.

2. The failure to comply with the requirements laid down in Part I of Annex III to Delegated Regulation (EU) 2016/1238 shall result in rejection. Products rejected shall not be presented again for acceptance.

3. The outcome of the checks described in point 1 shall be systematically recorded by the paying agency.

PART II

Conversion coefficients

Conformation class/fat cover	Coefficient
U2	1,058
U3	1,044
U4	1,015
R2	1,015
R3	1,000
R4	0,971
O2	0,956
O3	0,942
O4	0,914

PART III

Deboning

I. General conditions governing deboning

1. Deboning may only be carried out in cutting plants approved and operating in accordance with the requirements of Regulation (EC) No 853/2004 of the European Parliament and of the Council[(4)].

2. For the purposes of this Regulation 'deboning operations' means the physical operations for beef/veal as referred to in Annex II to Delegated Regulation (EU) No 906/2014.

3. Boned cuts must meet the requirements laid down in Part IV of this Annex.

II. Contracts and specifications

1. Deboning shall be carried out under contract on terms laid down by the paying agencies, in accordance with their specifications and in compliance with the requirements of this Regulation.

2. The specifications of the paying agencies shall lay down the requirements to be met by cutting plants, shall specify the plant and equipment required and shall lay down detailed conditions covering deboning operations.

They shall in particular lay down detailed conditions specifying the method of preparation, trimming, packing, freezing and preservation of cuts with a view to their takeover by the paying agency.

III. Control and monitoring of deboning operations

The paying agencies shall take all the necessary measures to ensure that deboning operations are carried out in compliance with the requirements of this Regulation and with the contracts and specifications described in Section II of this Part.

In particular, the paying agencies shall put in place a system to ensure the continuous monitoring and verification of all deboning operations. The outcome of such monitoring and verification shall be recorded.

IV. Storage of cuts

Cuts shall be stored in cold stores located in the **[**[F8]United Kingdom.**]**

Textual Amendments

F8 Words in Annex 3 Pt. 3 para. 4 substituted (31.12.2020) by The Market Measures Payment Schemes (Amendment) (EU Exit) Regulations 2019 (S.I. 2019/823), regs. 1, **6(45)**; 2020 c. 1, **Sch. 5 para. 1(1)**

V. Costs of deboning operations

Contracts as referred to in Section II of this Part and payments made thereunder shall cover the costs of deboning operations as referred to in point 2 of Section I of this Part.

VI. Time limits for deboning operations

Deboning, trimming, weighing, packaging and rapid freezing must be completed within 10 calendar days of slaughter. However, the paying agency may set shorter time limits.

VII. **Checks and rejection of products**

1. When as a result of the checks provided for in Section III of this Part, products are found not to be in compliance with the requirements as laid down in this Regulation and with the contracts and specifications described in Section II of this Part they shall be rejected.

2. Without prejudice to the application of penalties, the paying agencies shall recover payments from the responsible parties for an amount equal to the price shown in Part V of this Annex for the cuts that have been rejected.

PART IV

Specifications for intervention deboning

1. HINDQUARTER CUTS

1.1. Description of cuts

1.1.1. Intervention shank (code INT 11)

Cutting and deboning: remove by a cut passing through the stifle joint and separating from the topside and the silverside by following the natural seam, leaving the heel muscle attached to the shank. Remove shank bones (tibia and hock).

Trimming: trim sinew tips back to the meat.

Wrapping and packing: these cuts must be individually wrapped before packing in cartons.

1.1.2. Intervention thick flank (code INT 12)

Cutting and deboning: separate from the topside by a straight cut down to and along the line of the femur and from the silverside by continuing the cut down in the line of the natural seam; the cap must be left naturally attached.

Trimming: remove the patella, the joint capsule and tendon; the external fat cover must not exceed one centimetre at any point.

1.1.3. Intervention topside (code INT 13)

Cutting and deboning: separate from the silverside and the shank by a cut following the line of the natural seam and detach from the femur; remove the aitchbone.

Trimming: remove the pizzle butt, the adjacent gristle and the scrotal (superficial inguinal) gland; remove the cartilage and connective tissues associated with the pelvic bone; the external fat cover must not exceed one centimetre at any point.

1.1.4. Intervention silverside (code INT 14)

Cutting and deboning: separate from the topside and the shank by a cut following the line of the natural seam; remove the femur.

Trimming: remove the heavy cartilage adjacent to the bone joint, the popiteal lymph node, attached fat and tendon; the external fat cover must not exceed one centimetre at any point.

1.1.5. Intervention fillet (code INT 15)

Cutting: remove entire length of fillet by freeing the head (butt end) from the hip bone (ilium) and by tracing along the fillet adjacent to the vertebrae, thereby freeing the fillet from the loin.

Trimming: remove gland and de-fat. Leave the silverskin and chain muscle intact and fully attached.

1.1.6. Intervention rump (code INT 16)

Cutting and deboning: separate from the silverside/thick flank by a straight cut from a point approximately five centimetres from the posterior edge of the fifth sacral vertebra, passing approximately five centimetres from the anterior edge of the aitchbone, taking care not to cut through the thick flank.

Separate from the loin by a cut between the last lumbar and first sacral vertebrae, clearing the anterior edge of the pelvic bone. Remove bones and cartilage.

Trimming: remove the pocket of fat on the internal surface below the eye muscle. The external fat cover must not exceed one centimetre at any point.

1.1.7. Intervention striploin (code INT 17)

Cutting and deboning: separate from the rump by a straight cut between the last lumbar and the first sacral vertebrae. Separate from the fore-rib (five bone) by a straight cut between the eleventh and tenth ribs. Remove the backbones cleanly. Remove the ribs and feather bones by sheeting out.

Trimming: remove any species of cartilage left after deboning. The tendon must be removed. The external fat cover must not exceed one centimetre at any point.

1.1.8. Intervention flank (code INT 18)

Cutting and deboning: remove the full flank from the eight-rib straight-cut hindquarter by a cut from the point where the flank has been laid back, following the natural seam down around the surface of the hind muscles to a point which is horizontal to the middle of the last lumbar vertebra.

Continue the cut downwards in a straight line parallel to the fillet, through the thirteenth to the sixth rib inclusive along a line running parallel to the dorsal edge of the vertebral column, so that the entire downward cut is no more than five centimetres from the lateral tip of the eye muscle.

Remove all bones and cartilage by sheeting out. The whole flank must remain in one piece.

Trimming: remove the coarse connective tissue sheath covering the goose skirt, leaving the goose skirt intact. Trim fat so that the overall percentage of visible (external and interstitial) fat does not exceed 30 %.

1.1.9. Intervention fore-rib (five bone) (code INT 19)

Cutting and deboning: this cut must be separated from the striploin by a straight cut between the eleventh and tenth ribs and must include the sixth to tenth ribs inclusive. Remove the intercostal muscles and pleura in a thin sheet with rib bones. Remove backbone and cartilage, including the tip of the scapula.

Trimming: remove the backstrap (ligamentum nuchae). The external fat cover must not exceed one centimetre at any point. The cap must be left attached.

2. FOREQUARTER CUTS

2.1. Description of cuts

2.1.1. Intervention shin (code INT 21)

Cutting and deboning: remove by a cut around the joint separating the shinbone (radius) and clod-bone (humerus). Remove the shinbone (radius).

Trimming: trim sinew tips back to the meat.

Shins must not be packed with shanks.

2.1.2. Intervention shoulder (code INT 22)

Cutting and deboning: separate the shoulder from the forequarter by cutting in a line following the natural seam around the edge of the shoulder and the cartilage at the tip of the scapula, continuing around the seam so that the shoulder is lifted from its natural pocket. Remove the scapula. The blade muscle under the scapula must be laid back but left attached so as to allow clean removal of the bone. Remove the clod-bone (humerus).

Trimming: remove cartilage, tendons and joint capsules; trim fat so that the overall percentage of visible (external and interstitial) fat does not exceed 10 %.

2.1.3. Intervention brisket (code INT 23)

Cutting and deboning: separate from the forequarter by cutting in a straight line perpendicular to the middle of the first rib. Remove intercostal muscles and pleura by ‘sheeting out’, with ribs, breastbone and cartilage. Deckle to be left attached to the brisket. Fat underlying the deckle and the sternum must be removed.

Trimming: trim fat so that the overall percentage of visible (external and interstitial) fat does not exceed 30 %.

2.1.4. Intervention forequarter (code INT 24)

Cutting and deboning: the cut remaining after removal of the brisket, shoulder and shin is classed as forequarter.

Remove rib bones by sheeting out. Neck bones must be removed cleanly.

The chain muscle must be left attached to this cut.

Trimming: tendons, joint capsules and cartilage to be removed. Trim fat so that the overall percentage of visible (external and interstitial) fat does not exceed 10 %.

PART V

Individual prices of rejected intervention cuts

(EUR/tonne)	
Intervention fillet	22 000
Intervention striploin	14 000
Intervention topside, Intervention rump	10 000

Intervention silverside, Intervention thick flank, Intervention forerib (with five ribs)	8 000
Intervention shoulder, Intervention forequarter	6 000
Intervention brisket, Intervention shank, Intervention shin	5 000
Intervention flank	4 000

ANNEX IV

BUTTER

[[F9]A1.

This Annex does not apply in relation to public intervention schemes in England, otherwise than in connection with exceptional market conditions which are the subject of a declaration under section 20 of the Agriculture Act 2020.**]**

[[F10]A2. This Annex ceases to apply in relation to public intervention schemes in Scotland, otherwise than in connection with public intervention measures which the appropriate authority takes under Article 219(1) of Regulation (EU) No 1308/2013, for a period of five years beginning on 1 July 2023.**]**

Textual Amendments

F10 Annex 4 para. A2 inserted (S.) (1.7.2023) by The Public Intervention and Private Storage Aid (Amendment and Suspension) (Scotland) Regulations 2023 (S.S.I. 2023/150), regs. 1, **9(15)** (with reg. 10)

PART I

Sampling for chemical and microbiological analysis and sensory evaluation

1. Chemical and microbiological analysis

Quantity of butter(kg)	Minimum number of samples(> 100 g)
≤ 1 000	2
> 1 000 ≤ 5 000	3
> 5 000 ≤ 10 000	4
> 10 000 ≤ 15 000	5
> 15 000 ≤ 20 000	6
> 20 000 ≤ 25 000	7
> 25 000	7 + 1 per 25 000 kg or part thereof

Sampling for microbiological analysis must be carried out aseptically.

Up to five samples of 100 g may be combined into one sample for analysis after thorough mixing.

The samples must be taken randomly from different parts of each lot before or at the time of entry into the cold store designated by the paying agency.

Preparation of composite butter sample (chemical analysis):

(a) using a clean, dry butter trier or similar suitable instrument, extract a core of butter of at least 30 g and place in a sample container. The composite sample must then be sealed and forwarded to the laboratory for analysis;

(b) at the laboratory the composite sample is to be warmed in the original unopened container to 30 °C and shaken frequently until a homogeneous fluid emulsion free of unsoftened pieces is obtained. The container should be one half to two thirds full.

Two samples per year per producer offering butter for intervention must be analysed for non-milk fat.

2. **Sensory evaluation**

Quantity of butter(kg)	**Minimum number of samples**
1 000 ≤ 5 000	2
> 5 000 ≤ 25 000	3
> 25 000	3 + 1 per 25 000 kg or part thereof

Following a trial storage period of 30 days, samples are to be taken randomly from different parts of each lot between the 30th and the 45th day following delivery of the butter and graded.

[[F11]Each sample shall be assessed individually. No resampling or re-evaluation is allowed.**]**

Textual Amendments

F11 Substituted by Commission Implementing Regulation (EU) 2018/150 of 30 January 2018 amending Implementing Regulation (EU) 2016/1240 as regards methods for the analysis and quality evaluation of milk and milk products eligible for public intervention and aid for private storage.

3. **Guidelines to be followed where samples show defects**

(a) chemical and microbiological analysis:

(i) where individual samples are analysed, one sample showing a single defect out of five to 10 samples or two samples each showing a single defect out of 11 to 15 samples may be allowed. Where a sample shows a defect, two new samples must be taken from either side of the sample showing the defect and checked for the parameter in question. Where neither sample meets the specification, the quantity of butter between the original two samples on either side of the sample showing the defect must be rejected from the quantity offered.

Quantity to be rejected where the new sample shows a defect:

(ii) where composite samples are analysed and found to show defects in respect of one parameter, the quantity represented by the composite sample concerned is to be

rejected from the quantity offered. The quantity represented by one composite sample may be determined by subdividing the quantity before samples are taken randomly from each part thereof;

(b) sensory evaluation: where a sample fails the sensory evaluation, the quantity of butter between two neighbouring samples on either side of the sample failing is to be rejected from the quantity of the lot,

(c) where samples show a sensory defect and either a chemical or a microbiological defect, the whole quantity is to be rejected.

[[F12]PART IA

Methods of analysis of unsalted butter for public intervention

Parameter	Method
Fat [a]	ISO 17189 or ISO 3727 part 3
Water	ISO 3727 part 1
Non-fat solids	ISO 3727 part 2
Fat acidity	ISO 1740
Peroxide value	ISO 3976
Non-milk fat	ISO 17678
Sensory characteristics	ISO 22935 parts 2 and 3 and scoring table hereafter.

a The method to be applied shall be approved by the paying agency.

Scoring table

Appearance		Consistency		Odour and Flavour	
Points	**Remarks**	**Points**	**Remarks**	**Points**	**Remarks**
5	*Very good* Ideal type Highest quality (equal dry)	5	*Very good* Ideal type Highest quality (equal spreadable)	5	*Very good* Ideal type Highest quality (absolutely pure finest odour)
4	*Good* (no evident defects)	4	*Good* (no evident defects)	4	*Good* (no evident defects)
1, 2 or 3	Any defect	1, 2 or 3	Any defect	1, 2 or 3	Any defect**]**

Textual Amendments

F12 Inserted by Commission Implementing Regulation (EU) 2018/150 of 30 January 2018 amending Implementing Regulation (EU) 2016/1240 as regards methods for the analysis and quality evaluation of milk and milk products eligible for public intervention and aid for private storage.

PART II

Delivery and packaging of butter

1. Butter shall be delivered in blocks and packed in new, strong material in such a way as to ensure it is protected throughout transportation, entry into storage, storage and removal from storage.

2. The packing shall show at least the following particulars, where appropriate in code:

(a) the approval number identifying the factory and **[**F13indicating that the butter was produced in the United Kingdom**]**;

(b) the date of production;

(c) the production batch number and the package number; the package number may be replaced by a pallet number marked on the pallet;

(d) the words ‘sweet cream’ if the butter has a pH of 6,2 or higher.

Textual Amendments

F13 Words in Annex 4 Pt. 2 para. 2(a) substituted (31.12.2020) by The Market Measures Payment Schemes (Amendment) (EU Exit) Regulations 2019 (S.I. 2019/823), regs. 1, **6(46)**; 2020 c. 1, **Sch. 5 para. 1(1)**

3. The storekeeper shall keep a register in which the particulars referred to in point 2 are recorded on the date of entry into storage.

ANNEX V

SKIMMED MILK POWDER

[F14A1.

This Annex does not apply in relation to public intervention schemes in England, otherwise than in connection with exceptional market conditions which are the subject of a declaration under section 20 of the Agriculture Act 2020.**]**

[F15A2. This Annex ceases to apply in relation to public intervention schemes in Scotland, otherwise than in connection with public intervention measures which the appropriate authority takes under Article 219(1) of Regulation (EU) No 1308/2013, for a period of five years beginning on 1 July 2023.**]**

Textual Amendments

F15 Annex 5 para. A2 inserted (S.) (1.7.2023) by The Public Intervention and Private Storage Aid (Amendment and Suspension) (Scotland) Regulations 2023 (S.S.I. 2023/150), regs. 1, **9(15)** (with reg. 10)

PART I

Sampling and analysis of skimmed-milk powder offered for intervention

1. Samples per lot shall be taken in accordance with the procedure laid down in International Standard ISO 707. However, paying agencies may use another method of sampling provided that it complies with the principles of that standard.

2. Number of packages to be selected for taking samples for analysis:

(a) lots containing up to 800 25-kg bags: at least eight;

(b) lots containing more than 800 25-kg bags: at least eight, plus one for each additional 800 bags or fraction thereof.

3. Weight of sample: samples of at least 200 g are to be taken from each package.

4. Grouping of samples: no more than nine samples are to be combined in a global sample.

5. Analysis of samples: each global sample is to undergo an analysis to verify all the quality characteristics laid down in Part II of Annex V to Delegated Regulation (EU) 2016/1238.

6. Where samples show defects:

(a) where a composite sample shows a defect with regard to one parameter, the quantity from which the sample came is rejected;

(b) where a composite sample shows a defect with regard to more than one parameter, the quantity from which the sample came is rejected and samples are taken from the remaining quantities from the same plant; the analysis of those samples shall be decisive. In that case:

— the number of samples laid down in point 2 is doubled,

— where a composite sample shows a defect with regard to one or more parameters, the quantity from which the sample came is rejected.

[F12PART IA

Methods of analysis of skimmed milk powder for public intervention

Parameter	Method
Protein	ISO 8968 part 1
Fat	ISO 1736
Water	ISO 5537
Acidity	ISO 6091
Lactates	ISO 8069
Phosphatase test	ISO 11816 part 1
Insolubility index	ISO 8156
Scorched particles [a]	ADPI
Micro-organisms	ISO 4833-part 1
Buttermilk	Appendix I
Rennet whey [b]	Appendix II and III
Acid whey [c]	ISO 8069 or On-the-spot inspections
Sensory checks [d]	ISO 22935 part 2 and 3

a Scorched particles' analyses may be conducted systematically. However, such analyses shall always be conducted if no sensory checks are performed.

b The method to be applied shall be approved by the paying agency (one or both methods).

c The method to be applied shall be approved by the paying agency.

d Sensory checks shall be performed where deemed necessary after risk based analysis approved by the paying agency.

Appendix I

SKIMMED MILK POWDER: QUANTITATIVE DETERMINATION OF PHOSPHATIDYLSERINE AND PHOSPHATIDYLETHANOLAMINE

Method: reversed-phase HPLC

1. PURPOSE AND FIELD OF APPLICATION

The method describes a procedure for the quantitative determination of phosphatidylserine (PS) and phosphatidylethanolamine (PE) in skimmed milk powder (SMP) and is suitable for detecting buttermilk solids in SMP.

2. DEFINITION

PS + PE content : the mass fraction of substance determined using the procedure here specified. The result is expressed as milligrams of phosphatidylethanolamine dipalmitoyl (PEDP) per 100 g powder.

3. PRINCIPLE OF THE METHOD

Extraction of aminophospholipids by methanol from reconstituted milk powder. Determination of PS and PE as o-phthaldialdehyde (OPA) derivatives by reversed-phase (RP) HPLC and

fluorescence detection. Quantification of PS and PE content in the test sample by reference to a standard sample containing a known amount of PEDP.

4. REAGENTS

All reagents shall be of recognised analytical grade. Water shall be distilled or water of at least equivalent purity, unless otherwise specified.

4.1. **Standard material: PEDP, at least 99 % pure**

Note: Standard material shall be stored at – 18 °C.

4.2. **Reagents for standard sample and test sample preparation**

4.2.1. *HPLC-grade methanol*

4.2.2. *HPLC-grade chloroform*

4.2.3. *Tryptamine-monohydrochloride*

4.3. **Reagents for o-phthaldialdehyde derivatisation**

4.3.1. *Sodium hydroxide, 12 M water solution*

4.3.2. *Boric acid, 0,4 M water solution adjusted to pH 10,0 with sodium hydroxide (4.3.1)*

4.3.3. *2-mercaptoethanol*

4.3.4. *o-phthaldialdehyde (OPA)*

4.4. **HPLC elution solvents**

4.4.1. *Elution solvents shall be prepared using HPLC-grade reagents.*

4.4.2. *HPLC-grade water*

4.4.3. *Methanol of tested fluorimetric purity*

4.4.4. *Tetrahydrofuran*

4.4.5. *Sodium dihydrogen phosphate*

4.4.6. *Sodium acetate*

4.4.7. *Acetic acid.*

5. APPARATUS

5.1. **Analytical balance, capable of weighing to the nearest 1 mg, with a readability of 0,1 mg**

5.2. **Beakers, 25 and 100 ml capacity**

5.3. **Pipettes, capable of delivering 1 and 10 ml**

5.4. **Magnetic stirrer**

5.5. **Graduated pipettes, capable of delivering 0,2, 0,5 and 5 ml**

5.6. **Volumetric flasks, 10, 50 and 100 ml capacity**

5.7. **Syringes, 20 and 100 µl capacity**

5.8. **Ultrasonic bath**

5.9. **Centrifuge, capable of operating at 27 000 × g**

5.10. **Glass vials, about 5 ml capacity**

5.11. **Graduated cylinder, 25 ml capacity**

5.12. **pH-meter, accurate to 0,1 pH units**

5.13. **HPLC equipment**

5.13.1. *Gradient pumping system, capable of operating at 1,0 ml/min at 200 bar*

5.13.2. *Autosampler with derivatisation capability*

5.13.3. *Column heater, capable of maintaining the column at 30 °C ± 1 °C*

5.13.4. *Fluorescence detector, capable of operating at 330 nm excitation wavelength and 440 nm emission wavelength*

5.13.5. *Integrator or data processing software capable of peak area measurement*

5.13.6. *A LiChrospher® — 100 column (250 × 4,6 mm) or an equivalent column packed with octadecylsilane (C 18), 5 µm particle size.*

6. SAMPLING

Sampling shall be carried out in accordance with ISO Standard 707.

7. PROCEDURE

7.1. **Preparation of the internal standard solution**

7.1.1. *Weigh 30,0 ± 0,1 mg of tryptamine-monohydrochloride (4.2.3) into a 100 ml volumetric flask (5.6) and make up to the mark with methanol (4.2.1)*

7.1.2. *Pipette 1 ml (5.3) of this solution into a 10 ml volumetric flask (5.6) and make up to the mark with methanol (4.2.1) in order to obtain a 0,15 mM tryptamine concentration*

7.2. **Preparation of the test sample solution**

7.2.1. *Weigh 1,000 ± 0,001 g of the SMP sample into a 25 ml beaker (5.2). Add 10 ml of distilled water at 40 °C ± 1 °C by a pipette (5.3) and stir with a magnetic stirrer (5.4) for 30 minutes in order to dissolve any lumps*

7.2.2. *Pipette 0,2 ml (5.5) of the reconstituted milk into a 10 ml volumetric flask (5.6), add 100 µl of the 0,15 mM tryptamine solution (7.1) using a syringe (5.7) and make up to the volume with methanol (4.2.1). Mix carefully by inversion and sonicate (5.8) for 15 min*

7.2.3. *Centrifuge (5.9) at 27 000 g × g for 10 minutes and collect the supernatant in a glass vial (5.10)*

Note: Test sample solution should be stored at 4 °C until the HPLC analysis is performed.

7.3. **Preparation of the external standard solution**

7.3.1. *Weigh 55,4 mg PEDP (4.1) into a 50 ml volumetric flask (5.6) and add about 25 ml of chloroform (4.2.2) using a graduated cylinder (5.11). Heat the stoppered flask to*

50 °C ± 1 °C and mix carefully till the PEDP dissolves. Cool the flask to 20 °C, make up to the volume with methanol (4.2.1) and mix by inversion

7.3.2. *Pipette 1 ml (5.3) of this solution into a 100 ml volumetric flask (5.6) and make up to the volume with methanol (4.2.1). Pipette 1 ml (5.3) of this solution into a 10 ml volumetric flask (5.6), add 100 µl (5.7) of 0,15 mM tryptamine solution (7.1) and make up to the volume with methanol (4.2.1). Mix by inversion*

Note: Reference sample solution should be stored at 4 °C until the HPLC analysis is performed.

7.4. **Preparation of the derivatising reagent**

Weigh 25,0 ± 0,1 mg of OPA (4.3.4) into a 10 ml volumetric flask (5.6), add 0,5 ml (5.5) of methanol (4.2.1) and mix carefully to dissolve the OPA. Make up to the mark with boric acid solution (4.3.2) and add 20 µl of 2-mercaptoethanol (4.3.3) by syringe (5.7).

Note: The derivatising reagent should be stored at 4 °C in a brown glass vial and is stable for one week.

7.5. **Determination by HPLC**

7.5.1. *Elution solvents (4.4)*

Solvent A: Solution of 0,3 mM sodium dihydrogen phosphate and 3 mM sodium acetate solution (adjusted to pH 6,5 ± 0,1 with acetic acid): methanol: tetrahydrofuran = 558:440:2 (v/v/v)

Solvent B: methanol

7.5.2. *Suggested eluting gradient:*

Time (min)	Solvent A (%)	Solvent B (%)	Flow rate (ml/min)
Initial	40	60	0
0,1	40	60	0,1
5,0	40	60	0,1
6,0	40	60	1,0
6,5	40	60	1,0
9,0	36	64	1,0
10,0	20	80	1,0
11,5	16	84	1,0
12,0	16	84	1,0
16,0	10	90	1,0
19,0	0	100	1,0
20,0	0	100	1,0
21,0	40	60	1,0
29,0	40	60	1,0
30,0	40	60	0

Note: The eluting gradient may require slight modification in order to achieve the resolution shown in figure 1.

Column temperature: 30 °C.

7.5.3. *Injection volume: 50 µl derivatising reagent and 50 µl sample solution*

7.5.4. *Column equilibration*

Starting up the system on a daily basis, flush the column with 100 % solvent B for 15 minutes, then set at A:B = 40:60 and equilibrate at 1 ml/min for 15 minutes. Perform a blank run by injecting methanol (4.2.1).

Note: Before long-term storage flush the column with methanol: chloroform = 80:20 (v/v) for 30 minutes.

7.5.5. *Determine the PS + PE content in the test sample*

7.5.6. *Perform the sequence of the chromatographic analyses keeping constant the run-to-run time in order to obtain constant retention times. Inject the external standard solution (7.3) every 5-10 test sample solutions in order to calculate the response factor*

Note: The column shall be cleaned by flushing with 100 % solvent B (7.5.1) for at least 30 minutes every 20-25 runs.

7.6. **Integration mode**

7.6.1. *PEDP peak*

PEDP is eluted as a single peak. Determine the peak area by valley-to- valley integration.

7.6.2. *Tryptamine peak*

Tryptamine is eluted as a single peak (Figure 1). Determine the peak area by valley-to-valley integration.

7.6.3. *PS and PE peaks groups*

Under the described conditions (Figure 1), PS elutes as two main partially unresolved peaks preceded by a minor peak. PE elutes as three main partially unresolved peaks. Determine the whole area of each peak cluster setting the baseline as reported in Figure 1.

8. CALCULATION AND EXPRESSION OF RESULTS

PS and PE content in the test sample shall be calculated as follows:

$C = 55{,}36 \times ((A_2)/(A_1)) \times ((T_1)/(T_2))$

where:

C	=	PS or PE content (mg/100 g powder) in the test sample
A_1	=	PEDP peak area of the standard sample solution (7.3)
A_2	=	PS or PE peak area of the test sample solution (7.2)
T_1	=	Tryptamine peak area of the standard sample solution (7.3)
T_2	=	Tryptamine peak area of the test sample solution (7.2).

9. ACCURACY OF THE METHOD

Note: The values for repeatability were calculated according to the IDF International Standard (*).

9.1. **Repeatability**

The relative standard deviation of the repeatability, which expresses the variability of independent analytical results obtained by the same operator using the same apparatus under the same conditions on the same test sample and in a short interval of time, should not exceed 2 % relative. If two determinations are obtained under these conditions, the relative difference between the two results should not be greater than 6 % of the arithmetic mean of the results.

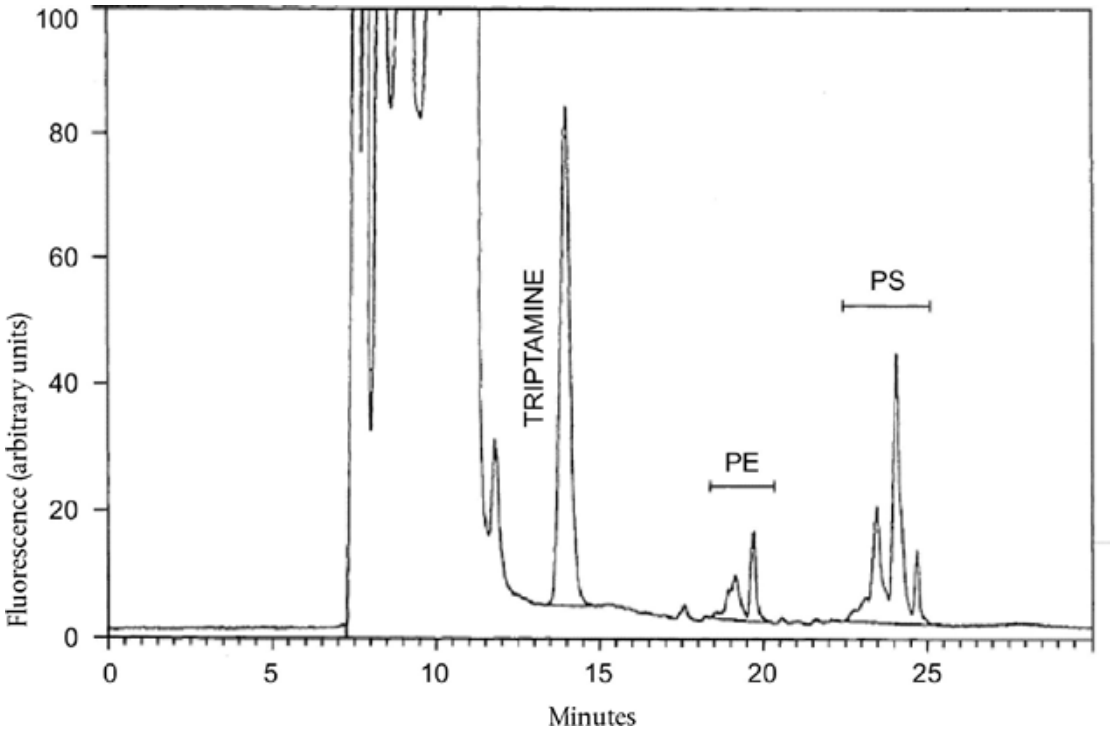
9.2. **Reproducibility**

If two determinations are obtained by operators in different laboratories using different apparatus under different conditions for the analysis on the same test sample, the relative difference between the two results should not be greater than 11 % of the arithmetic mean of the results.

10. REFERENCES

10.1. *Resmini P., Pellegrino L., Hogenboom J.A., Sadini V., Rampilli M., ' Detection of buttermilk solids in skimmilk powder by HPLC quantification of aminophospholipids ' . Sci. Tecn. Latt.-Cas., 39,395 (1988).*

Figure 1 **HPLC pattern of OPA-derivatives of phosphatidylserine (PS) and phosphatidylethanolamine (PE) in methanol extract of reconstituted skim-milk powder. Integration mode for the peaks of PS, PE and tryptamine (internal standard) is reported**



Appendix II

DETECTION OF RENNET WHEY IN SKIMMED MILK POWDER FOR PUBLIC STORAGE BY DETERMINATION OF CASEINOMACROPEPTIDES HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

1. SCOPE AND FIELD OF APPLICATION

This method allows detection of rennet whey in skimmed milk powder intended for public storage by determination of the caseinomacropeptides.

2. REFERENCE

International Standard ISO 707 - Milk and Milk Products - Guidance on sampling.

3. DEFINITION

The content of rennet whey solids is defined as the percentage by mass as determined by the caseinomacropeptide content by the procedure described.

4. PRINCIPLE

— Reconstitution of the skimmed milk powder, removal of fat and proteins with trichloroacetic acid, followed by centrifugation or filtration;

— Determination of the quantity of caseinomacropeptides (CMP) in the supernatant by high-performance liquid chromatography (HPLC);

— Evaluation of the result obtained for the samples by reference to standard samples consisting of skimmed milk powder with or without the addition of a known percentage of whey powder.

5. REAGENTS

All reagents shall be of recognised analytical grade. The water used shall be distilled water or water of at least equivalent purity.

5.1. **Trichloroacetic acid solution**

Dissolve 240 g of trichloroacetic acid (CCl_3COOH) in water and make up to 1 000 ml. The solution should be clear and colourless.

5.2. **Eluent solution, pH 6,0**

Dissolve 1,74 g of dipotassium hydrogen phosphate (K_2HPO_4), 12,37 g of potassium dihydrogen phosphate (KH_2PO_4) and 21,41 g of sodium sulphate (Na_2SO_4) in about 700 ml of water. Adjust, if necessary, to pH 6,0, using a solution of phosphoric acid or potassium hydroxide.

Make up to 1 000 ml with water and homogenise.

Note: The composition of the eluent can be updated to comply with the certificate of the standards or the recommendations of the manufacturer of the column packing material.

Filter the eluent solution, prior to use, through a membrane filter with a 0,45 μm pore diameter.

5.3. **Flushing solvent**

Mix one volume acetonitrile (CH_3CN) with nine volumes water. Filter the mixture prior to use through a membrane filter with a 0,45 μm pore diameter.

Note: Any other flushing solvent with a bactericidal effect which does not impair the columns' resolution efficiency may be used.

5.4. **Standard samples**

5.4.1. *Skimmed milk powder meeting the requirements of this Regulation (i.e. [0])*

5.4.2. *The same skimmed milk powder adulterated with 5 % (m/m) rennet-type whey powder of standard composition (i.e. [5])*

6. APPARATUS

6.1. **Analytical balance**

6.2. **Optional centrifuge capable of attaining a centrifugal force of 2 200 g, fitted with stoppered or capped centrifuge tubes of about 50 ml capacity**

6.3. **Mechanical shaker**

6.4. **Magnetic stirrer**

6.5. **Glass funnels, diameter about 7 cm**

6.6. **Filter papers, medium filtration, diameter about 12,5 cm**

6.7. **Glass filtration equipment with 0,45 µm pore diameter membrane filter**

6.8. **Graduated pipettes allowing delivery of 10 ml (ISO 648, Class A, or ISO/R 835) or a dispensing system capable of delivering 10,0 ml in two minutes**

6.9. **Dispensing system capable of delivering 20,0 ml water at ca. 50 °C**

6.10. **Thermostatic water bath, set at 25 ± 0,5 °C**

6.11. **HPLC equipment, consisting of:**

6.11.1. *Pump*

6.11.2. *Injector, hand or automatic, with a 15 to 30 µl capacity*

6.11.3. *Two TSK 2 000 -SW columns in series (length 30 cm, internal diameter 0,75 cm) or equivalent columns (e.g. single TSK 2 000 -SWxl, single Agilent Technologies Zorbax GF 250) and a precolumn (3 cm × 0,3 cm) packed with I 125 or material of equivalent effectiveness*

6.11.4. *Thermostatic column oven, set at 35 ± 1 °C*

6.11.5. *Variable wavelength UV detector, permitting measurements at 205 nm with a sensitivity of 0,008 Å*

6.11.6. *Integrator capable of valley-to-valley integration*

Note: Working with columns kept at room temperature is possible, but their power of resolution is slightly lower. In that case, the temperature should vary by less than ± 5 °C in any one range of analyses.

7. SAMPLING

7.1. Samples shall be taken in accordance with the procedure laid down in International Standard ISO 707. However, **[**F16the appropriate authority**]** may use another method of sampling provided that it complies with the principles of the abovementioned standard

Textual Amendments

F16 Words in Annex 5 Pt. 1A Appendix 2 para. 7.1 substituted (31.12.2020) by The Market Measures Payment Schemes (Amendment) (EU Exit) Regulations 2019 (S.I. 2019/823), regs. 1, **6(47)(a)(i)**; 2020 c. 1, **Sch. 5 para. 1(1)**

7.2. Store the sample in conditions which preclude any deterioration or change in composition

8. PROCEDURE

8.1. **Preparation of the test sample**

Transfer the milk powder into a container with a capacity of about twice the volume of the powder, fitted with an airtight lid. Close the container immediately. Mix the milk powder well by means of repeated inversion of the container.

8.2. **Test portion**

Weight 2,000 ± 0,001 g of test sample into a centrifuge tube (6.2) or a suitable stoppered flask (50 ml).

8.3. **Removal of fat and proteins**

8.3.1. *Add 20,0 ml of warm water (50 °C) to the test portion. Dissolve the powder by shaking for five minutes using a mechanical shaker (6.3). Place the tube into the water bath (6.10) and allow to equilibrate to 25 °C*

8.3.2. *Add 10,0 ml of the trichloroacetic acid solution (5.1) of ca. 25 °C in two minutes, while stirring vigorously with the aid of the magnetic stirrer (6.4). Place the tube in a water bath (6.10) and leave for 60 minutes*

8.3.3. *Centrifuge (6.2) for 10 minutes at 2 200 g, or filter through paper (6.6), discarding the first 5 ml of filtrate*

8.4. **Chromatographic determination**

8.4.1. *Inject 15 to 30 µl of accurately measured supernatant or filtrate (8.3.3) into the HPLC apparatus (6.11) operating at a flow rate of 1,0 ml of eluent solution (5.2) per minute*

Note 1. Another flow rate may be used, dependent of the internal diameter of the columns used or the instructions of the manufacturer of the column.

Note 2. Rinse the columns with water during each interruption. Never leave the eluent solution in them (5.2).

Prior to any interruption of more than 24 hours, rinse the columns with water then wash them with solution (5.3) for at least three hours at a flow rate of 0,2 ml per minute.

8.4.2. *The results of chromatographic analysis of the test sample [E] are obtained in the form of chromatogram in which each peak is identified by its retention time RT as follows:*

Peak II:	The second peak of the chromatogram having an RT of about 12,5 minutes.
Peak III:	The third peak of the chromatogram, corresponding to the CMP, having an RT of 15,5 minutes.

The choice of the column(s) can affect the retention times of the individual peaks considerably.

The integrator (6.11.6) automatically calculates the area A of each peak:

A_{II} :	area of peak II,
A_{III} :	area of peak III,

It is essential to examine the appearance of each chromatogram prior to quantitative interpretation, in order to detect any abnormalities due either to malfunctioning of the apparatus or the columns, or to the origin and nature of the sample analysed.

If in doubt, repeat the analysis.

8.5. **Calibration**

8.5.1. *Apply exactly the procedure described from point 8.2 to point 8.4.2 to the standard samples (5.4)*

Use freshly prepared solutions, because CMP degrade in an 8 % trichloroacetic environment. The loss is estimated at 0,2 % per hour at 30 °C.

8.5.2. *Prior to chromatographic determination of the samples, condition the columns by repeatedly injecting the standard sample (5.4.2) in solution (8.5.1) until the area and retention time of the peak corresponding to the CMP are constant*

8.5.3. *Determine the response factors R by injecting the same volume of filtrates (8.5.1) as used for the samples*

9. EXPRESSION OF RESULTS

9.1. **Method of calculation and formulae**

9.1.1. *Calculation of the response factors R:*

Peak II:	$R_{II} = 100/(A_{II}\ [0])$

where:

R_{II} = the response factors of peaks II,
$A_{II}\ [0]$ = the areas of peaks II of the standard sample [0] obtained in 8.5.3.

Peak III:	$R_{III} = W/(A_{III}\ [5] - A_{III}\ [0])$

where:

R_{III} = the response factor of peak III,
$A_{III}\ [0]$ and $A_{III}\ [5]$ = the areas of peak III in standard samples [0] and [5] respectively obtained in 8.5.3,
W = the quantity of whey in standard sample [5], i.e. 5.

9.1.2. *Calculation of the relative area of the peaks in the sample [E]*

$S_{II}\,[E] = R_{II} \times A_{II}\,[E]$

$S_{III}\,[E] = R_{III} \times A_{III}\,[E]$

$S_{IV}\,[E] = R_{IV} \times A_{IV}\,[E]$

where:

S_{II} [E], S_{III} [E], S_{IV} [E] = the relative areas of peaks II, III and IV respectively in the sample [E],

A_{II} [E], A_{III} [E] = the areas of peaks II and III respectively in the sample [E] obtained in 8.4.2,

R_{II} , R_{III} = the response factors calculated in 9.1.1.

9.1.3. *Calculation of the relative retention time of peak III in sample [E]:*

$$RRT_{III}[E] = (RT_{III}[E])/(RT_{III}[5])$$

where:

RRT_{III} [E] = the relative retention time of peak III in sample [E],

RT_{III} [E] = the retention time of peak III in sample [E] obtained in 8.4.2,

RT_{III} [5] = the retention time of peak III in control sample [5] obtained in 8.5.3.

9.1.4. *Experiments have shown that there is a linear relation between the relative retention time of peak III, i.e. RRT_{III} [E] and the percentage of whey powder added up to 10 %*

— The RRT_{III} [E] is < 1,000 when the whey content is > 5 %;

— The RRT_{III} [E] is ≥ 1,000 when the whey content is ≤ 5 %.

The uncertainty allowed for the values of RRT_{III} is ± 0,002.

Normally the value of RRT_{III} [0] deviates little from 1,034. Depending on the condition of the columns, the value may approach 1,000, but it shall always be greater.

9.2. **Calculation of the percentage of rennet whey powder in the sample:**

$$W = S_{III}[E] - [1,3 + (S_{III}[0] - 0,9)]$$

where:

W = the percentage m/m of rennet whey in the sample [E];

S_{III} [E] = the relative area of peak III of test sample [E] obtained as in 9.1.2;

1,3 = represents the relative average area of peak III expressed in grams of rennet whey per 100 g determined in non-adulterated skimmed milk powder of various origins. This figure was obtained experimentally;

S_{III} [0] = represents the relative area of peak III which is equal to $R_{III} \times A_{III}$ [0]. These values are obtained in 9.1.1 and 8.5.3 respectively;

(S_{III} [0] – 0,9) = represents the correction to be made to the relative average area 1,3 when S_{III} [0] is not equal to 0,9. Experimentally the relative average area of peak III of the control sample [0] is 0,9.

9.3. **Accuracy of the procedure**

9.3.1. *Repeatability*

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst using the same apparatus on identical test material shall not exceed 0,2 % m/m.

9.3.2. *Reproducibility*

The difference between two single and independent results, obtained in two different laboratories on identical test material shall not exceed 0,4 % m/m.

9.4. **Interpretation**

9.4.1. *Assume the absence of whey if the relative area of peak III, S_{III} [E] expressed in grams of rennet whey per 100 g of the product is ≤ 2,0 + (S_{III} [0] – 0,9)*

where

2,0	is the maximum value allowed for the relative area of peak III taking into account the relative average area of peak III, i.e. 1,3, the uncertainty due to variations in the composition of skimmed milk powder and the reproducibility of the method (9.3.2),
(S_{III} [0] – 0,9)	is the correction to be made when the area S_{III} [0] is different from 0,9 (see point 9.2)

9.4.2. *If the relative area of peak III, S_{III} [E] is > 2,0 + (S_{III} [0] – 0,9) and the relative area of peak II, S_{II} [E] ≤ 160, determine the rennet whey content as indicated in point 9.2.*

9.4.3. *If the relative area of peak III, S_{III} [E] is > 2,0 + (S_{III} [0] – 0,9) and the relative area of peak II, S_{II} [E] ≤ 160, determine the total protein content (P %); then examine graphs 1 and 2.*

9.4.3.1. *The data obtained after analysis of samples of unadulterated skimmed milk powders with a high total protein content have been assembled in graphs 1 and 2.*

The continuous line represents the linear regression, the coefficients of which are calculated by the least squares method.

The dashed straight line fixes the upper limit of the relative area of peak III with a probability of not being exceeded in 90 % of cases.

The equations for the dashed straight lines of graphs 1 and 2 are:

S_{III} = 0,376 P % – 10,7	(graph 1),
S_{III} = 0,0123 S_{II} [E] + 0,93	(graph 2),

respectively where:

S_{III}	is the relative area of peak III calculated either according to total protein content or according to the relative area of peak S_{II} [E],
P %	is the total protein content expressed as a percentage, by weight,
S_{II} [E]	is the relative area of sample calculated in point 9.1.2.

These equations are equivalent to the figure of 1,3 mentioned in point 9.2.

The discrepancy (T_1 and T_2) between the relative area S_{III} [E] found and the relative area S_{III} is given by means of the following: $T_1 = S_{III}[E] – [(0,376\ P\% – 10,7) + (S_{III}[0] – 0,9)]$ $T_2 = S_{III}[E] – [(0,0123\ S_{II}[E] + 0,93) + (S_{III}[0] – 0,9)]$

9.4.3.2.

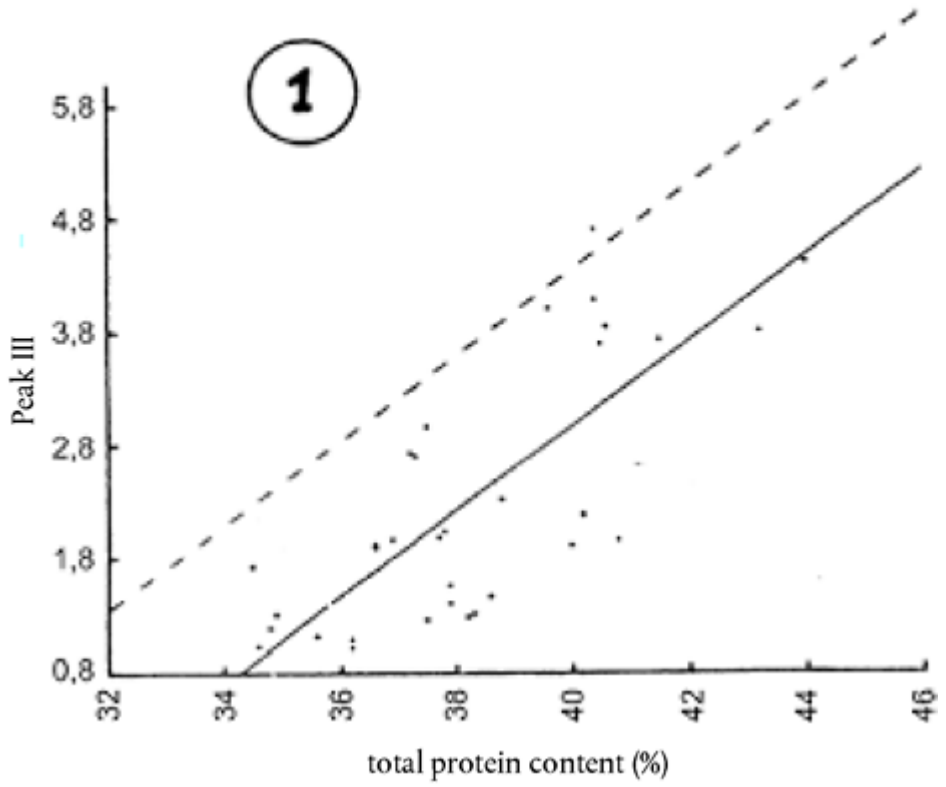
If T_1 and/or T_2	are zero or less, the presence of rennet whey cannot be determined.
If T_1 and T_2	exceed zero, rennet whey is present.

The rennet whey content is calculated according to the following formula: $W = T_2 + 0,91$

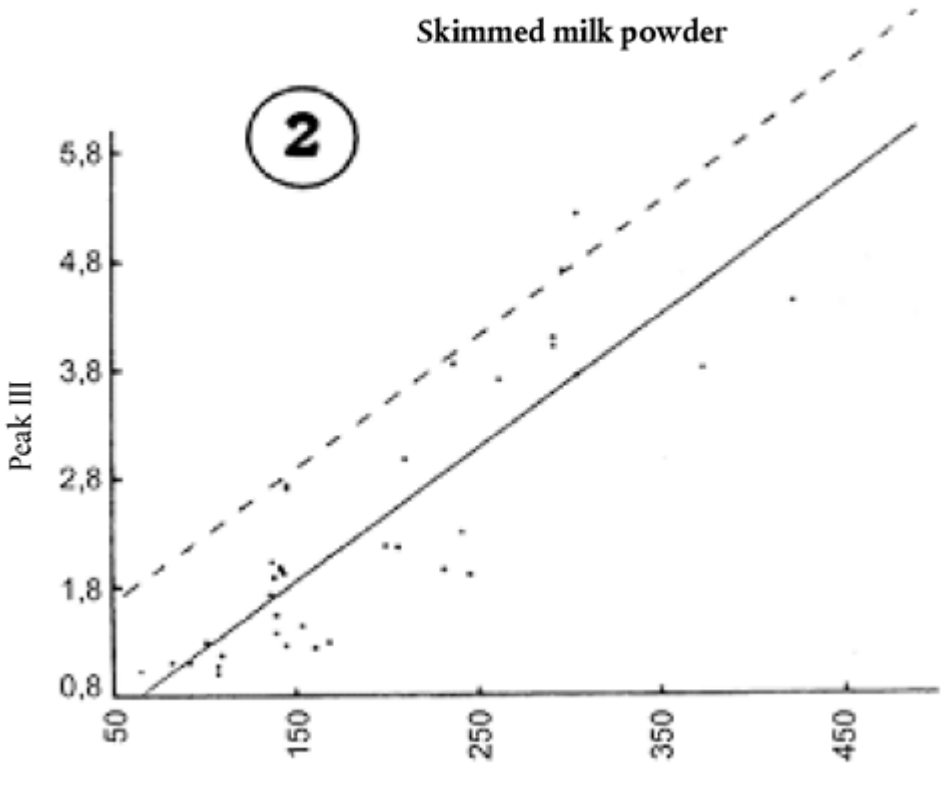
where:

0,91 is the distance on the vertical axis between the continuous and dotted straight lines.

Skimmed milk powder



Skimmed milk powder



Appendix III

DETERMINING RENNET WHEY SOLIDS IN SKIMMED MILK POWDER

1. PURPOSE: DETECTING THE ADDITION OF RENNET WHEY SOLIDS TO SKIMMED MILK POWDER

2. REFERENCES: INTERNATIONAL STANDARD ISO 707

3. DEFINITION

The content of rennet whey solids is defined as the percentage by mass as determined by caseinomacropeptide content by the procedure described.

4. PRINCIPLE

Samples are analysed for caseinomacropeptide A by a reversed-phase high-performance liquid chromatography procedure (HPLC procedure). Evaluation of the result is obtained by reference to standard samples consisting of skimmed milk powder with and without a known percentage of whey powder. Results higher than 1 % (m/m) show that rennet whey solids are present.

5. REAGENTS

All reagents shall be of recognised analytical grade. The water used shall be distilled water or water of at least equivalent purity. Acetonitrile should be of spectroscopic or HPLC quality.

5.1. **Trichloroacetic acid solution**

Dissolve 240 g of trichloroacetic acid (CCl_3COOH) in water and make up to 1 000 ml. The solution should be clear and colourless.

5.2. **Eluents A and B**

Eluent A: 150 ml of acetonitrile (CH_3CN), 20 ml of isopropanol ($CH_3CHOHCH_3$), and 1,00 ml of trifluoroacetic acid (TFA, CF_3COOH) are placed in a 1 000 ml volumetric flask. Make up to 1 000 ml with water.

Eluent B: 550 ml of acetonitrile, 20 ml of isopropanol and 1,00 ml of TFA are placed in a 1 000 ml volumetric flask. Make up to 1 000 ml with water. Filter the eluent solution, prior to use, through a membrane filter with a 0,45 μm pore diameter.

5.3. **Conservation of the column**

After the analyses the column is flushed with eluent B (via a gradient) and subsequently flushed with acetonitrile (via a gradient for 30 minutes). The column is stored in acetonitrile.

5.4. **Standard samples**

5.4.1. *Skimmed milk powder meeting the requirements for public storage (i.e. [0]).*

5.4.2. *The same skimmed milk powder adulterated with 5 % (m/m) rennet-type whey powder of standard composition (i.e. [5]).*

5.4.3. *The same skimmed milk powder adulterated with 50 % (m/m) rennet-type whey powder of standard composition (i.e. [50])*

6. APPARATUS

6.1. **Analytical balance**

6.2. **Optional centrifuge capable of attaining a centrifugal force of 2 200 g, fitted with stoppered or capped centrifuge tubes of about 50 ml capacity**

6.3. **Mechanical shaker**

6.4. **Magnetic stirrer**

6.5. **Glass funnels, diameter about 7 cm**

6.6. **Filter papers, medium filtration, diameter about 12,5 cm**

6.7. **Glass filtration equipment with 0,45 µm pore diameter membrane filter**

6.8. **Graduated pipettes, allowing delivery of 10 ml (ISO 648, Class A, or ISO/R 835), or a dispensing system capable of delivering 10,0 ml in two minutes**

6.9. **Dispensing system capable of delivering 20,0 ml water at ca. 50 °C**

6.10. **Thermostatic water bath, set at 25 ± 0,5 °C**

6.11. **HPLC equipment, consisting of:**

6.11.1. *Binary gradient pumping system*

6.11.2. *Injector, hand or automatic, with a 100 µl capacity*

6.11.3. *Agilent Technologies Zorbax 300 SB-C3 column (length 25 cm, 0,46 cm internal diameter) or an equivalent wide-pore silica based reversed-phase column*

6.11.4. *Thermostatic column oven, set at 35 ± 1 °C*

6.11.5. *Variable wavelength UV detector, permitting measurements at 210 nm (if necessary, a higher wavelength up to 220 nm may be used) with a sensitivity of 0,02 Å*

6.11.6. *Integrator capable of setting the integration to common baseline or valley-to-valley*

Note : Operation of the column at room temperature is possible, provided that the room temperature does not fluctuate more than 1 °C, otherwise too much variation in the retention time of CMP_A takes place.

7. SAMPLING

7.1. **Samples shall be taken in accordance with the procedure laid down in International Standard ISO 707. However, [F17the appropriate authority] may use another method of sampling provided that it complies with the principles of the abovementioned standard**

Textual Amendments

F17 Words in Annex 5 Pt. 1A Appendix 3 para. 7.1 substituted (31.12.2020) by The Market Measures Payment Schemes (Amendment) (EU Exit) Regulations 2019 (S.I. 2019/823), regs. 1, **6(47)(a)(ii)**; 2020 c. 1, **Sch. 5 para. 1(1)**

7.2. **Store the sample in conditions which preclude any deterioration or change in composition.**

8. PROCEDURE

8.1. **Preparation of the test sample**

Transfer the milk powder into a container with a capacity of about twice the volume of the powder, fitted with an airtight lid. Close the container immediately. Mix the milk powder well by means of repeated inversion of the container.

8.2. **Test portion**

Weigh 2,00 ± 0,001 g of test sample into a centrifuge tube (6.2) or suitable stoppered flask (50 ml).

Note: In the case of mixtures, weigh such an amount of the test sample that the defatted sample portion corresponds to 2,00 g.

8.3. **Removal of fat and proteins**

8.3.1. *Add 20,0 ml of warm water (50 °C) to the test portion. Dissolve the powder by shaking for five minutes using a mechanical shaker (6.3). Place the tube into the water bath (6.10) and allow to equilibrate to 25 °C*

8.3.2. *Add 10,0 ml of the trichloroacetic acid solution of ca. 25 °C (5.1) constantly over two minutes, while stirring vigorously with the aid of the magnetic stirrer (6.4). Place the tube in a water bath (6.10) and leave for 60 minutes*

8.3.3. *Centrifuge (6.2) 2 200 g for 10 minutes, or filter through paper (6.6), discarding the first 5 ml of filtrate*

8.4. **Chromatographic determination**

8.4.1. *The reversed-phase HPLC method excludes the possibility false-positive results due to the presence of acid buttermilk powder.*

8.4.2. *Before the reversed phase HPLC-analysis is carried out, the gradient conditions should be optimised. A retention time of 26 ± 2 minutes for CMP $_A$ is optimal for gradient systems having a dead volume of about 6 ml (volume from the point where the solvents come together to the volume of the injector loop, inclusive). Gradient systems having a lower dead volume (e.g. 2 ml) should use 22 minutes as an optimal retention time*

Take solutions of the standard samples (5.4) without and with 50 % rennet whey.

Inject 100 µl of supernatant or filtrate (8.3.3) into the HPLC apparatus operating at the scouting gradient conditions given in Table 1.

TABLE 1

Scouting gradient conditions for optimisation of the chromatography

Time (min)	Flow (ml/min)	% A	% B	Curve
Initial	1,0	90	10	*
27	1,0	60	40	linear
32	1,0	10	90	linear
37	1,0	10	90	linear
42	1,0	90	10	linear

Comparison of the two chromatograms should reveal the location of the peak of CMP_A.

Using the formula given below, the initial solvent composition to be used for the normal gradient (see 8.4.3) can be calculated % B = 10 – 2,5 + (13,5 + (RT_{cmpA} – 26) / 6) * 30 / 27 % B = 7,5 + (13,5 + (RT_{cmpA} – 26) / 6) * 1,11

Where:

RT_{cmpA}	:	retention time of CMP_A in the scouting gradient
10	:	the initial % B of the scouting gradient
2,5	:	% B at midpoint minus % B at initial in the normal gradient
13,5	:	midpoint time of the scouting gradient
26	:	required retention time of CMP_A
6	:	ratio of slopes of the scouting and normal gradient
30	:	% B at initial minus % B at 27 minutes in the scouting gradient
27	:	run-time of the scouting gradient.

8.4.3. *Take solutions of the test samples*

Inject 100 µl of accurately measured supernatant or filtrate (8.3.3) into the HPLC apparatus operating at a flow rate of 1,0 ml of eluent solution (5.2) per minute.

The composition of the eluent of the start of the analysis is obtained from 8.4.2. It is normally close to A:B = 76:24 (5.2). Immediately after the injection a linear gradient is started, which results in a 5 % higher percentage of B after 27 minutes. Subsequently a linear gradient is started, which brings the eluent composition to 90 % B in five minutes. This composition is maintained for five minutes, after which the composition is changed, via a linear gradient in five minutes to the initial composition. Depending on the internal volume of the pumping system, the next injection can be made 15 minutes after reaching the initial conditions.

Note 1. The retention time of the CMP_A should be 26 ± 2 minutes. This can be achieved by varying the initial and end conditions of the first gradient. However, the difference in the % B for the initial and end conditions of the first gradient shall remain 5 % B.

Note 2. The eluents should be degassed sufficiently and should also remain degassed. This is essential for proper functioning of the gradient pumping system. The standard deviation for the retention time of the CMP_A peak should be smaller than 0,1 minutes (n = 10).

Note 3. Every five samples the reference sample [5] should be injected and used to calculate a new response factor R. (9.1.1).

8.4.4. *The results of the chromatographic analysis of the test sample (E) are obtained in the form of a chromatogram in which the CMP_A peak is identified by its retention time of about 26 minutes*

The integrator (6.11.6) automatically calculates the peak height H of the CMP_A peak. The baseline location should be checked in every chromatogram. The analysis or the integration should be repeated if the baseline was incorrectly located.

Note: If the CMP_A peak is sufficiently separated from other peaks valley-to-valley baseline allocation should be used, otherwise use dropping perpendiculars to a common baseline, which should have starting point close to the CMP_A peak (thus not at t = 0 min!).Use for the standard and the samples the same type integration type and check in case of common baseline its consistency for the samples and the standard.

It is essential to examine the appearance of each chromatogram prior to quantitative interpretation, in order to detect any abnormalities due either to malfunctioning of the apparatus or the column, or to the origin and nature of the sample analysed. If in doubt, repeat the analysis.

8.5. **Calibration**

8.5.1. *Apply exactly the procedure described from point 8.2 to point 8.4.4 to the standard samples (5.4.1 to 5.4.2). Use freshly prepared solutions, because CMP degrades in an 8 % trichloroacetic acid environment at room temperature. At 4 °C the solution remains stable for 24 hours. In the case of long series of analyses the use of a cooled sample tray in the automatic injector is desirable*

Note: 8.4.2. may be omitted if the % B at initial conditions is known from previous analyses.

The chromatogram of the reference sample [5] should be analogous to Figure. 1. In this figure the CMP_A peak is preceded by two small peaks. It is essential to obtain a similar separation.

8.5.2. *Prior to chromatographic determination of the samples inject 100 µl of the standard sample without rennet whey [0] (5.4.1)*

The chromatogram should not show a peak at the retention time of the CMP_A peak.

8.5.3. *Determine the response factors R by injecting the same volume of filtrate (8.5.1) as used for the samples.*

9. EXPRESSION OF RESULTS

9.1. **Method of calculation and formulae**

9.1.1. *Calculation of the response factor R:*

CMP_A peak: $R = W/H$

Where:

R	=	the response factor of the CMP_A peak
H	=	the height of the CMP_A peak
W	=	the quantity of whey in the standard sample [5].

9.2. **Calculation of the percentage of rennet whey powder in the sample**

$W(E) = R \times H(E)$

Where:

W(E)	=	the percentage (m/m) of rennet whey in the sample (E).
R	=	the response factor of the CMP_A peak (9.1.1)
H(E)	=	the height of the CMP_A peak of the sample (E)

If W(E) is greater than 1 % and the difference between the retention time and that of the standard sample [5] is smaller than 0,2 minutes then rennet whey solids are present.

9.3. **Accuracy of the procedure**

9.3.1. *Repeatability*

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst using the same apparatus on identical test material shall not exceed 0,2 % m/m.

9.3.2. *Reproducibility*

Not determined.

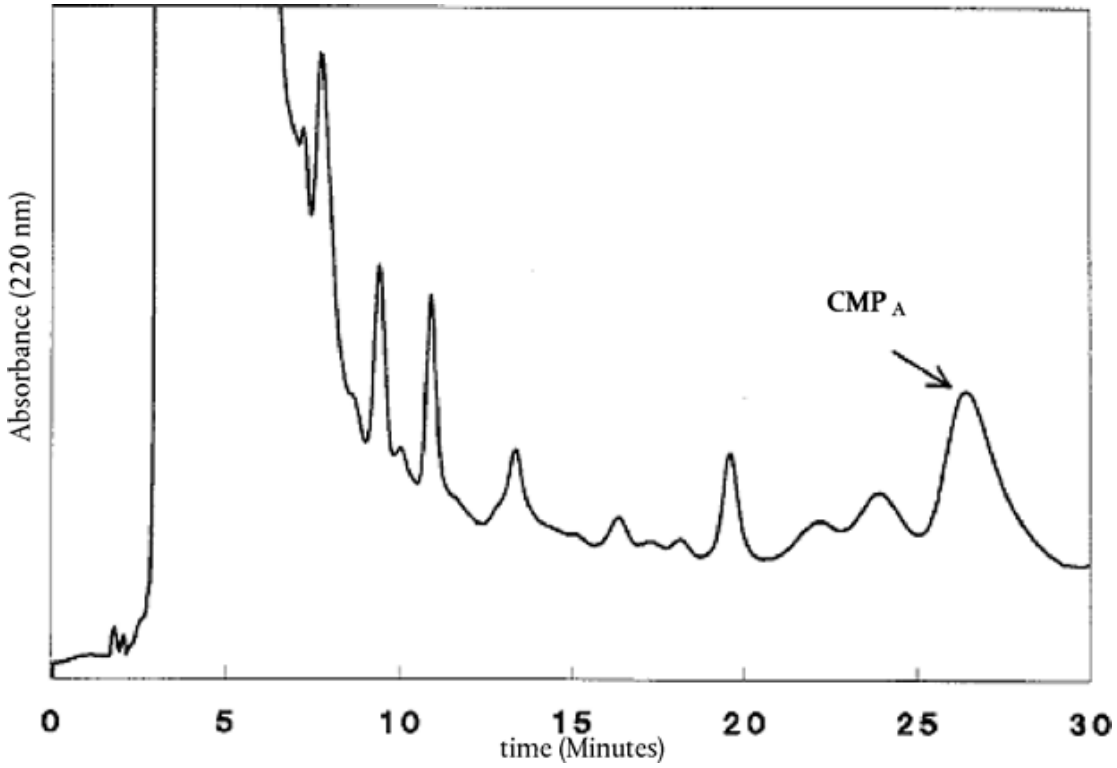
9.3.3. *Linearity*

From 0 to 16 % of rennet whey a linear relationship should be obtained with a coefficient of correlation > 0,99.

9.4. **Interpretation**

The 1 % limit includes the uncertainty due to reproducibility.

Figure 1 **Ni—4.6 standard**



(*) International IDF Standard 135B/1991. Milk and milk products. Precision characteristics of analytical methods. Outline of collaborative study procedure.**]**

PART II

Delivery and packaging of skimmed milk powder

1. Skimmed milk powder shall be packed in new, clean, dry and intact bags meeting the following requirements:

(a) the bags shall have at least three layers, which together correspond to at least 420 J/m^2 TEA average;

(b) the second layer shall be covered with a layer of polyethylene of at least 15 g/m^2;

(c) inside the paper layers, a polyethylene bag at least 0,08 mm thick shall be fused to the bottom;

(d) bags shall conform to standard EN 770;

(e) when filling, the powder should be well pressed down. Loose powder must on no account be allowed to penetrate between the various layers.

2. The bags shall show the following particulars, where appropriate in code:

(a) the approval number identifying the factory and **[**[F18]indicating that the skimmed milk powder was produced in the United Kingdom**]**;

(b) the date or, where appropriate, the week of production;

(c) the number of the production batch;

(d) the description ‘spray skimmed-milk powder’.

Textual Amendments

F18 Words in Annex 5 Pt. 2 para. 2(a) substituted (31.12.2020) by The Market Measures Payment Schemes (Amendment) (EU Exit) Regulations 2019 (S.I. 2019/823), regs. 1, **6(47)(b)**; 2020 c. 1, **Sch. 5 para. 1(1)**

3. The storekeeper shall keep a register in which the particulars referred to in point 2 are recorded on the date of entry into storage.

[[F12]ANNEX VI

Methods of analysis of butter under private storage

Parameter	Method
Fat [a]	ISO 17189 or ISO 3727 part 3
Water	ISO 3727 part 1
Non Fat Solids (excluding salt)	ISO 3727 part 2
Salt	ISO 15648

a The method to be applied shall be approved by the paying agency.

ANNEX VII

Methods of analysis of skimmed milk powder under private storage

Parameter	Method
Fat	ISO 1736
Protein	ISO 8968 part 1

Water	ISO 5537

ANNEX VIII

Methods of analysis of cheeses under private storage

1. The method of analysis laid down in the Appendix shall be used to ensure that cheese made exclusively from ewe's milk, goat's milk or buffalo milk or from a mixture of ewe's milk, goat's milk and buffalo milk does not contain cow's milk casein.

Cow's milk casein is considered to be present if the cow's milk casein content of the analysed sample is equal to or higher than the content of the reference sample containing 1 % cow's milk as laid down in the Appendix.

2. Methods for detecting cow's milk casein in cheeses referred to in paragraph 1 may be used provided that:

(a) the detection limit is maximum 0,5 % and

(b) there are no false-positive results and

(c) cow's milk casein is detectable with the required sensitivity even after long ripening periods, as may occur in usual commercial conditions.

If any of the above mentioned requirements is not met, the methods laid down in the Appendix shall be used.**]**

(**1**) Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs (OJ L 364, 20.12.2006, p. 5).

(**2**) Commission Regulation (EC) No 401/2006 of 23 February 2006 laying down the methods of sampling and analysis for the official control of the levels of mycotoxins in foodstuffs (OJ L 70, 9.3.2006, p. 12).

(**3**) Commission Regulation (EU) No 234/2010 of 19 March 2010 laying down certain detailed rules for the application of Council Regulation (EC) No 1234/2007 on the granting of export refunds on cereals and the measures to be taken in the event of disturbance on the market for cereals (OJ L 72, 20.3.2010, p. 3).

(**4**) Regulation (EC) No 853/2004 of the European Parliament and of the Council of 29 April 2004 laying down specific hygiene rules for food of animal origin (OJ L 139, 30.4.2004, p. 55).