Text consolidated by Valsts valodas centrs (State Language Centre) with amending regulations of:

21 June 2016 [shall come into force on 22 December 2016];

19 April 2022 [shall come into force on 23 April 2022].

If a whole or part of a paragraph has been amended, the date of the amending regulation appears in square brackets at the end of the paragraph. If a whole paragraph or sub-paragraph has been deleted, the date of the deletion appears in square brackets beside the deleted paragraph or sub-paragraph.

Republic of Latvia

Cabinet

Regulation No. 623

Adopted 3 November 2015

**Requirements for the Quality, Classification, and Supplementary Labelling of Edible Caseins and Caseinates**

*Issued pursuant to*

*Section 4, Paragraph four and Section 13, Paragraph three, Clause 3 of the Law on the Supervision of the Handling of Food*

**I. General Provisions**

1. The Regulation prescribes the requirements for the quality and classification of edible acid caseins, edible rennet caseins, and edible caseinates intended for the production of foodstuffs (hereinafter – the edible caseins and caseinates) and the procedures for assessing conformity of the abovementioned products with such requirements, and also the supplementary labelling requirements.

[*21 June 2016*]

2. This Regulation shall apply to:

2.1. the edible acid casein – a milk product obtained by curdling skimmed milk with an acid or products obtained from milk with the subsequent removal of whey and the rinsing and drying of the coagulate obtained;

2.2. the edible rennet casein – a milk product obtained by curdling skimmed milk or products obtained from milk with the subsequent removal of whey and the rinsing and drying of the coagulate obtained. The coagulate is obtained by using rennet or other coagulating enzymes which conform to the requirements laid down in Regulation (EC) No 1332/2008 of the European Parliament and of the Council of 16 December 2008 on food enzymes and amending Council Directive 83/417/EEC, Council Regulation (EC) No 1493/1999, Directive 2000/13/EC, Council Directive 2001/112/EC and Regulation (EC) No 258/97;

2.3. the edible caseinates – milk products obtained by drying an edible casein treated with neutralizing agents or coagulate curd of an edible casein.

[*21 June 2016*]

**II. Requirements for the Quality, Classification, and Supplementary Labelling**

3. [21 June 2016]

4. [21 June 2016]

5. [21 June 2016]

6. Acids such as lactic acid, hydrochloric acid, sulphuric acid, citric acid, acetic acid, and orthophosphoric acid, and also lactic starter or whey soured with a lactic starter may be used as coagulants in the production of the edible acid casein.

[*21 June 2016*]

7. Agents neutralizing the edible casein or solvents such as sodium, potassium, calcium, ammonia, and magnesium hydroxides, carbonates, phosphates, and citrates may be used in the production of edible caseinates.

8. The trade names of edible caseins and caseinates referred to in Annexes 1 and 2 to this Regulation shall only be used to denote these specific products, provided that they conform to the definitions and quality requirements laid down in this Regulation. Products not meeting the criteria referred to in Annexes 1 and 2 to this Regulation shall be assigned such names and labelling as not to mislead consumers in respect of characteristics, quality, or use thereof.

9. The labelling of edible caseins and caseinates on the packaging, container, or label shall contain the following information which is easily visible, clearly legible, and indelible:

9.1. the name – “edible acid casein”, “edible rennet casein”, or “edible caseinate”;

9.2. the net weight in kilograms or grams;

9.3. the name of such food business operator or the business name and address under whose name or business name this product is marketed or, if the abovementioned operator does not carry out a business activity in the European Union, the importer into the European Union market;

9.4. the country of origin if the edible caseins and caseinates are imported from third countries;

9.5. the date of manufacture or identification number of the batch.

[*21 June 2016*]

10. The name of the edible caseinate shall be accompanied on its labelling by the designation of the relevant cation or cations – sodium, potassium, calcium, ammonium, or magnesium.

11. If the edible caseins or caseinates are marketed in the form of mixtures, the labelling shall indicate:

11.1. “mixture of X” where X is the relevant name of the edible caseins or caseinates in decreasing order of weight;

11.2. the cation or cations if the mixture contains edible caseinates;

11.3. the protein content of the mixture if the mixture contains edible caseinates.

12. The information referred to in Paragraphs 9 and 11 of this Regulation shall be indicated on the labelling in conformity with the requirements laid down in the Official Language Law (this does not exclude a possibility of indicating this information in several languages).

[*21 June 2016*]

13. The information referred to in Sub-paragraphs 9.2, 9.3, 9.4, and 11.3 of this Regulation may only be indicated in accompanying documents of the edible caseins and caseinates.

14. If the minimum milk protein content in the edible caseins and caseinates exceeds the value provided for in Paragraph 1 of Annex 1 to this Regulation, this may be indicated, as appropriate, on the labelling on the product packaging, container, or label.

[*21 June 2016*]

**III. Requirements for the Sampling of Edible Caseins and Caseinates**

15. The requirements and procedures for sampling edible caseins and caseinates for chemical analyses are laid down in Annex 3 to this Regulation.

16. An inspector authorised by the Food and Veterinary Service (hereinafter – the inspector) shall take a sample for official control according to the official control programme developed by the Food and Veterinary Service. The inspector shall mark and seal a sample at a sampling point.

17. The inspector shall take a parallel sample for analysis upon request of a food business. The parallel sample shall be taken concurrently with the sample for official control, and they shall be accompanied by a sampling report.

18. Sampling equipment shall be made of stainless steel or another adequate material which does not cause changes in the sample and does not affect analytical results. The equipment shall have a smooth surface, without any cracks, and its corners shall be rounded.

19. Samples containers and lids thereof shall be made of such a material and designed in such a manner as to protect the sample and not to cause any changes therein which can affect analytical results. Samples containers and lids thereof can be made of glass, metal, such as stainless steal, and plastics, such as polypropylene. If the samples containers are transparent or semi-transparent, they shall be stored in a dark place after placing the sample therein. The samples containers and lids thereof shall be clean and dry.

20. The form and volume of a sample container shall conform to the sampling requirements laid down in this Regulation. Disposable plastic, laminated and also aluminium foil samples containers or suitable plastic bags with a wire, plastic clip, or another closure may be used.

21. A suitable plug or screw-capped metal or plastic lid with an air-proof gasket, where appropriate, shall be used to seal samples containers. Plugs and gaskets shall be made of insoluble, non-absorbent material not penetrable by fats which does not affect the smell, taste, odour, other characteristics or content of the sample. Plugs shall be covered with or made of non-absorbent non-aromatic materials.

22. The sample container shall be immediately sealed after placing the sample therein. Sample storage temperature may not be higher than 25 °C.

23. The samples referred to in Paragraphs 15 and 16 of this Regulation shall be transported to a laboratory as soon as possible but not later than within 24 hours after sampling. During transportation, the samples shall be protected from exposure to surrounding odours, direct sunlight, and temperature if it is higher than 25 °C.

**IV. Preparation of the Samples of Edible Caseins and Caseinates for Chemical Analysis**

24. The methods for chemical analysis of the samples of edible caseins and caseinates and the procedures for the performance thereof shall be laid down in Annex 4 to this Regulation. The abovementioned methods shall be used in order to determine:

24.1. the moisture:

24.1.1. in the edible acid casein;

24.1.2. in the edible rennet casein;

24.1.3. in caseinates;

24.2. the protein content:

24.2.1. in the edible acid casein;

24.2.2. in the edible rennet casein;

24.2.3. in caseinates;

24.3. the total acidity in the edible acid casein;

24.4. the ash (including P2O5):

24.4.1. in the edible acid casein;

24.4.2. in the edible rennet casein;

24.5. the pH value in caseinates.

[*21 June 2016*]

25. The mass of the analysed sample shall be at least 200 grams. An analytical balance, accurate to 0.1 milligrams, shall be used to weigh the sample.

26. The laboratory sample shall be mixed thoroughly by shaking it repeatedly and turning the container over. Then the entire laboratory sample shall be transferred into an air-proof container of a sufficient volume.

27. Approximately 50 grams of the laboratory sample mixed thoroughly shall be carried to a test sieve with a receiver. The test sieve shall be constructed of wire netting, the nominal dimension of holes thereof shall be 500 micrometres, and the diameter of the sieve shall be 200 millimetres, and it shall have a lid.

[*19 April 2022*]

27.1Following the recommendation of the Ministry of Agriculture, the national standardisation authority shall publish on its website a list of standards which can be applied to determine the technical parameters (perforation margin and wire diameter) of sieves to be used for the test referred to in Paragraph 27 of this Regulation.

[*19 April 2022*]

27.2Where the standards referred to in Paragraph 27.1of this Regulation are applied, it shall be deemed that the preparation of samples of edible caseins and caseinates and the chemical analysis thereof conform to the essential requirements laid down in this Chapter that are covered by such standards or parts thereof.

[*19 April 2022*]

28. If 50 grams of the sample pass through the sieve fully or at least in the amount of 95 percent, the laboratory sample shall be used for testing.

29. If 50 grams of the sample do not pass through the sieve fully, it shall be ground in a grinder until it meets the sieving criteria referred to in Paragraph 27 of this Regulation. A bead mill may not be used for grinding, and during grinding neither excessive heat can be generated nor moisture can be lost. The entire sifted sample shall be immediately transferred into an air-proof container of a sufficient volume and mixed thoroughly by repeatedly shaking and inverting, and precluding any change in the moisture content of the product. After preparation of the sample to be analysed, the analyses referred to in Paragraph 23 of this Regulation shall be performed as soon as possible.

30. The sample shall always be stored in an air-proof and moisture-proof container.

31. For the purpose of dissolving, dilution, and washing, distilled water or demineralised water of a least equivalent degree of purity as distilled water shall be used.

32. If “dissolving”, “solution”, or “dilution” is referred to in this Regulation without any additional information, it shall mean “aqueous solution” or “dilution with water”.

33. All chemicals used shall be of the level of purity of the analytical reagent.

34. The result entered into the test report shall be the average value obtained in at least two cases of determination with sufficient repeatability. The test report shall be prepared and stored in a laboratory.

35. Testing results shall be indicated as a percentage of the mass of the sample to be analysed in grams per 100 grams.

36. The test report shall indicate:

36.1. the method of analysis used and the results obtained;

36.2. the information on the procedure which has not been specified in the description of the method of analysis or which is optional;

36.3. the circumstances which could have affected the results obtained;

36.4. the information necessary for the identification of the sample.

**V. Closing Provision**

37. Cabinet Regulation No. 435 of 21 June 2005, Requirements for the Quality, Classification, and Labelling of Edible Caseins and Caseinates and the Procedures for the Conformity Assessment Thereof (*Latvijas Vēstnesis*, 2005, No. 100), is repealed.

**Informative Reference to European Union Directives**

[*21 June 2016*]

The Regulation contains legal norms arising from:

1) [21 June 2016];

2) First Commission Directive 85/503/EEC of 25 October 1985 on methods of analysis for edible caseins and caseinates;

3) First Commission Directive 86/424/EEC of 15 July 1986 laying down methods of sampling for chemical analysis of edible caseins and caseinates;

4) Directive (EU) 2015/2203 of the European Parliament and of the Council of 25 November 2015 on the approximation of the laws of the Member States relating to caseins and caseinates intended for human consumption and repealing Council Directive 83/417/EEC.

Prime Minister Laimdota Straujuma

Minister for Agriculture Jānis Dūklavs

**Annex 1**

Cabinet Regulation No. 623

3 November 2015

**Requirements for the Quality and Classification of Edible Caseins and Caseinates**

[*21 June 2016*]

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| No. | Indicators | Trade name of the product | | |
| edible acid casein | edible rennet casein | edible caseinates |
| 1. | Milk protein content in dried extract,  not less than (%) | 90 | 84 | 88 |
| 2. | Casein content in milk protein,  not less than (%) | 95 | 95 | 95 |
| 3. | Milk fat in dried extract, not more than (%) | 2.0 | 2.0 | 2.0 |
| 4. | Moisture, not more than (%) | 12 | 12 | 8 |
| 5. | Titratable acidity (0.1N NaOH),  not more than ml/g | 0.27 | – | – |
| 6. | Ash (including P2O5) (%) | not more than 2.5 | not less than 7.5 | – |
| 7. | Anhydrous lactose, not more than (%) | 1.0 | 1.0 | 1.0 |
| 8. | Sediment (burnt particles),  not more than mg/25 g | 22.5 | 15 | 22.5 |
| 9. | pH value | – | – | 6.0–8.0 |
| 10. | Lead content, not more than mg/kg | 0.75 | 0.75 | 0.75 |
| 11. | Solid impurities 25 g | not acceptable | | |

**Annex 2**

Cabinet Regulation No. 623

3 November 2015

**Organoleptic Characteristics of Edible Caseins and Caseinates**

[*21 June 2016*]

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| No. | Indicators | | Edible acid casein | Edible rennet casein | Edible caseinates |
| 1. | Odour | | Characteristic odour of the product | | Attenuated foreign smells are acceptable |
| 2. | Appearance | colour | From white to cream | | |
| texture | Not containing grains which do not crack under light pressure | | |
| 3. | Solubility in water | | Insoluble | | Almost fully soluble in distilled water, except for calcium caseinates |

Minister for Agriculture Jānis Dūklavs

**Annex 3**

Cabinet Regulation No. 623

3 November 2015

**Sampling Procedures for Chemical Analyses of Edible Caseins and Caseinates**

[*21 June 2016*]

1. The requirements laid down in this Annex shall apply to the sampling of the edible acid casein, edible rennet casein, and caseinates.

2. Sampling equipment shall conform to the requirements referred to in Paragraph 17 of this Regulation:

2.1. testers of appropriate lengths referred to in Paragraph 7 of this Annex which can reach the bottom of the container or packaging of the product;

2.2. a wide spoon, scoop, or dipper;

2.3. samples containers in accordance with Paragraphs 18, 19, and 20 of this Regulation.

3. It shall be ensured that a sample is not exposed to humid air either during sampling or afterwards. After sampling, a container into which the sample has been transferred shall be sealed.

4. At least 200 g of the product from one batch shall be taken for the sample. A clean and dry tester shall be used to drill in the product. Container or packaging of the product shall be tilted aside, where necessary. Opening of tester shall be directed downwards, and drilling speed shall be steady. When the tester has reached the bottom of the container or packaging, the tester shall be rotated 180 degrees and pulled out. Content shall be placed into a sample container. In order to take the quantity of the product necessary for the sample, one or more holes shall be made. The sample container shall be immediately sealed after placing the sample therein.

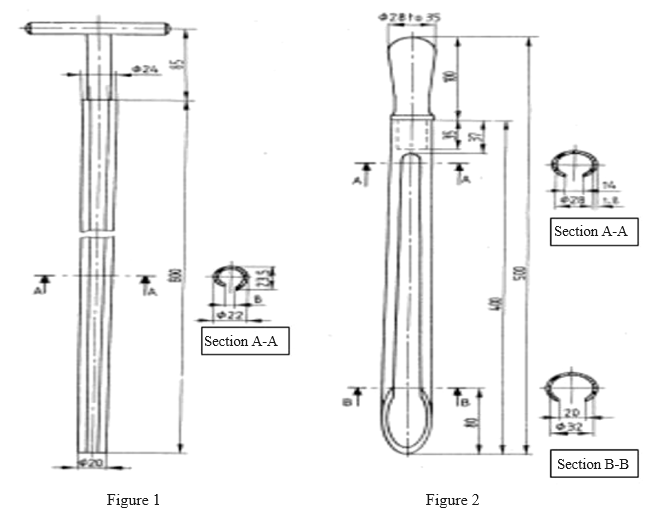
5. If the sample is taken from products which are packed in small retail packings, one or more packings with the same batch or code number shall be taken in order to obtain at least 200 g of the product or, if that is not possible, another method shall be used to create the sample. If the retail packing is damaged or open, it shall not be valid for the sample.

6. Samples shall be stored and transported in accordance with Paragraphs 21 and 22 of this Regulation.

7. Testers suitable for the sampling of caseins and caseinates from containers and large packings shall be presented in Figures 1 and 2 of this Annex:

7.1. A-type long testers (Figure 1);

7.2. B-type short testers (Figure 2).



8. A tester bit and a shank shall be made of highly polished metal, preferably stainless steel. A handle of the long tester shall be made of stainless steel. The short tester shall have a removable wooden or plastic handle which is fixed in the blade with the help of a bayonet stopper.

9. Characteristics of the tester:

9.1. the shape, material, and finish of the tester allows to clear the tester easily;

9.2. the protruded edge of the A-type long tester is sufficiently sharp;

9.3. the tip of the tester bit is sufficiently sharp to sample easily.

10. Testers shall correspond to the following dimensions (mm) (a tolerance of 10 % is acceptable):

|  |  |  |
| --- | --- | --- |
|  | A-type testers (long) | B-type testers (short) |
| 10.1. blade length | 800 | 400 |
| 10.2. metal thickness of the blade | 1 to 2 | 1 to 2 |
| 10.3. internal diameter of the blade tip | 18 | 32 |
| 10.4. blade diameter at the handle or shank | 22 | 28 |
| 10.5. opening width at the blade tip | 4 | 20 |
| 10.6. opening width at the handle or shank | 14 | 14 |

11. Instructions for use of testers:

11.1. if a powder does not flow freely, testers can be inserted vertically. A-type testers shall be completely filled by rotating and then pulled out vertically. B-type testers are already completely filled at the moment of pressing, and they shall be pulled out obliquely so that no losses occur at the bottom part;

11.2. if a powder flows freely, the container or packaging shall be tilted, the tester shall be inserted almost horizontally with the opening directed downwards and the pulled out with the opening directed upwards.

Minister for Agriculture Jānis Dūklavs

**Annex 4**

Cabinet Regulation No. 623

3 November 2015

**Methods for Chemical Analysis of Edible Caseins and Caseinates**

[*21 June 2016*]

**I. Determination of moisture content (method 1)**

1. In employing this method, the moisture content shall be determined in:

1.1. edible acid caseins;

1.2. edible rennet caseins;

1.3. caseinates.

2. The moisture content in caseins and caseinates shall constitute a loss in mass of the sample determined by employing the method described in this Chapter.

3. The principle of the method – drying of a test portion at a temperature of 102 ± 1 °C to constant weight and weighing to determine the loss in mass. The loss in mass shall be calculated as a percentage proportion to the sample weight.

4. The required equipment:

4.1. an analytical balance;

4.2. a flat-bottomed beaker of non-corrodible material such as aluminium or a container of stainless steel with a diameter between 60 to 80 mm and at least 25 mm high, and fitted with a tightly sealable lid that can be opened easily;

4.3. a drying cabinet with air circulation where temperature can be maintained at 102 ± 1 °C;

4.4. a dessicator containing freshly activated silica gel with a water content indicator or equivalent dehydrator;

4.5. a device suitable for taking containers, for example, laboratory tongs.

5. Procedure:

5.1. prepare a test sample in accordance with Paragraphs 24, 25, 26, 27, and 28 of this Regulation;

5.2. prepare a beaker or another container:

5.2.1. heat an unsealed container and its lid in the drying cabinet at a temperature of 102 ± 1 °C for at least an hour;

5.2.2. place the lid on the container, transfer the container into the dessicator, allow to cool to the weighing room temperature, and weigh to the nearest 0.1 mg (m0);

5.3. place the prepared test sample of 3–5 g in the container, close the container with the lid, and weigh to the nearest 0.1 mg (m1);

5.4. determine the moisture content:

5.4.1. open the container and place it in the drying cabinet together with the lid at a temperature of 102 ± 1 °C for four hours;

5.4.2. transfer the container into the dessicator, allow to cool to the weighing room temperature, and weigh to the nearest 0.1 mg;

5.4.3. open the container and re-heat it in the drying cabinet together with the lid for one hour, repeat the steps referred to in Sub-paragraph 5.4.2 of this Chapter;

5.4.4. if the mass obtained in accordance with Sub-paragraph 5.4.3 of this Chapter is at least 1 mg less than the mass obtained in accordance with Sub-paragraph 5.4.2 of this Chapter, repeat the steps referred to in Sub-paragraph 5.4.3;

5.4.5. if the mass increases, the smallest value of the obtained mass shall be used in the calculations referred to in Sub-paragraph 6.1 of this Chapter. The final weight shall be in m2 grams. The total drying time does not usually exceed six hours.

6. Expression of results:

6.1. the loss in mass of the dried sample which is expressed as a percentage proportion to mass shall be calculated as follows to the nearest 0.01 %:

[(m1 – m2)/(m1 – m0)] × 100 where

m0 – mass of the container and its lid in grams (Sub-paragraph 5.2 of this Chapter);

m1 – mass of the container, its lid, and test sample in grams before drying (Sub-paragraph 5.3 of this Chapter);

m2 – mass of the container, its lid, and test sample in grams after drying (Sub-paragraph 5.4.3 or 5.4.4 of this Chapter);

6.2. if the analysis has been performed accurately and in line with the conditions, the difference between the two individual results obtained by one analyser testing identical material and using the same equipment within a short time may not exceed 0.5 g of water per 100 g of the product. This repeatability interval shall be 95 % of all determination cases.

**II. Determination of protein content (method 2)**

1. In employing this method, the protein content shall be determined in:

1.1. edible acid caseins;

1.2. edible rennet caseins;

1.3. caseinates, except for caseinates containing ammonium caseinate or other ammonium or nitrogen compounds other than protein.

2. The protein content shall constitute nitrogen content determined by employing the method described in this Chapter, multiplied by 6.38 and expressed as a percentage proportion to mass.

3. The principle of the method – a test sample shall be mineralised together with a mixture of potassium sulphate and sulphuric acid in the presence of a catalyst – divalent copper sulphate – to turn organic nitrogen into ammonium sulphate. Ammonia solution shall be distilled by containing the released ammonia in a boric acid solution and titrated with a standard solution of hydrochloric acid. Nitrogen content shall be converted to protein content multiplying the result by the factor 6.38.

4. The required reagents:

4.1. concentrated sulphuric acid 1.84 g/ml;

4.2. anhydrous potassium sulphate (K2SO4);

4.3. divalent copper sulphate pentahydrate (CuSO4 ·5H2O);

4.4. sucrose (C12H22O11);

4.5. boric acid 40 g/l;

4.6. concentrated aqueous solution of sodium hydroxide 30 % (m/m) not containing carbonates;

4.7. hydrochloric acid 0.1 mol/l;

4.8. an indicator mixture prepared by mixing the same volume of 2 g/l of methyl red solution in at least 95 % (V/V) ethanol and 1 g/l of methylene blue solution in at least 95 % (V/V) ethanol.

5. The required equipment:

5.1. an analytical balance accurate to ± 1 mg;

5.2. a 500 ml Kjeldahl flask;

5.3. an evaporating installation which keeps the Kjeldahl flask in an inclined position and a heating appliance which does not heat the part of the flask above the surface of the liquid content;

5.4. a condenser with a straight inner pipe;

5.5. an outlet pipe which is connected to the condenser through a drop catcher using a specially created glass connection or a rubber tube. Where the rubber tube is used, ends of glass parts shall be located next to each other;

5.6. a drop catcher which is connected to the Kjeldahl flask and condenser by using soft tight rubber stoppers or plugs of other suitable material;

5.7. a 500 ml conical flask;

5.8. measuring cylinders, capacity 50 ml and 100 ml;

5.9. a 50 ml burette, graduated in 0.1 ml;

5.10. boiling stones for:

5.10.1. evaporation – small pieces of porcelain or glass balls;

5.10.2. distillation – fresh calcined pieces of pumice.

6. Procedure:

6.1. prepare a test sample in accordance with Paragraphs 24, 25, 26, 27, and 28 of this Regulation;

6.2. test for the presence of ammoniacal nitrogen if the presence of ammonium caseinate or another ammonium compound is possible. Place 1 g of the sample in the conical flask, add 10 ml of water and 100 mg of magnesium oxide. Rinse off the magnesium oxide stuck to walls of the flask, close the flask with a cork plug, placing a piece of wet red litmus paper between the plug and neck of the flask. Mix the contents of the flask thoroughly and heat it in the water bath at a temperature of 60–65 °C. If the litmus paper turns blue within 15 minutes, the sample contains ammonia, and the method referred to in this Chapter cannot be employed;

6.3. perform a blank test concurrently with determining the ammoniacal nitrogen content by replacing the sample with 0.5 g of sucrose. Use the same installation, the same quantity of reagent, and employ the same procedure. If titration results in the blank test exceed 0.5 ml 0.1 mol/l of acid, check the reagents and clear or replace the inadequate reagent or reagents;

6.4. transfer into the Kjeldahl flask 0.3–0.4 g of the test sample weighed to the nearest 0.1 mg;

6.5. determine the protein content:

6.5.1. transfer some pieces of porcelain or some glass balls and approximately 10 g of anhydrous potassium sulphate into the flask. Add 0.2 g of divalent copper sulphate pentahydrate and rinse it off the neck of the flask with a little water. Add 20 ml of concentrate sulphuric acid. Mix the contents of the flask. Heat the evaporating installation carefully until foaming ends, boil slowly until the solution is clear and a faint greenish-blue colour persists. Move the flask periodically during heating. Continue boiling for 90 minutes preventing local overheating and adjusting heating so that vapour is condensed in the middle of the neck of the flask. Allow to cool to room temperature. Carefully add approximately 200 ml of water and some pieces of pumice, mix and cool again;

6.5.2. transfer 50 ml of boric acid solution and four drops of indicator mixture into the conical flask. Mix. Place the conical flask under the condenser so that the contracted end of the outlet pipe is immersed in the boric acid solution. Place 80 ml of sodium hydroxide solution in the Kjeldahl flask using the measuring cylinder. During this procedure hold the flask in an inclined position so that the sodium hydroxide solution rinses edges of the flask creating a lower layer. Immediately connect the Kjeldahl flask to the condenser through the drop catcher to prevent any potential loss. Mix the contents of the Kjeldahl flask with a rotating movement carefully, then boil carefully avoiding foaming. Continue distillation until 150 ml of a distillate is collected within approximately 30 minutes. Temperature of the distillate must be below 25 °C. Lower the conical flask approximately 2 minutes before the end of the distillation so that the end of the outlet pipe is no longer immersed in the acid solution, and rinse the end with a little water. Stop the heating, disconnect the outlet pipe and rinse its external and internal walls with a little water collecting rinsing water in the conical flask;

6.5.3. titrate the distillate in the conical flask using a standard solution of hydrochloric acid.

7. Expression of results:

7.1. the protein content which is expressed as a percentage proportion to mass shall be calculated as follows to the nearest 0.1 %:

[(V1 – V2) × T × 14 × 100 × 6.38]/ m × 1 000 = [8.932 (V1 – V2) × T]/ m where

V1 – volume of the standard solution of hydrochloric acid used to determine the protein content (millilitres);

V2 – volume of the standard solution of hydrochloric acid used in the blank test (millilitres);

T – concentration of the standard solution of hydrochloric acid (mol/l);

m – mass of the test sample (grams);

7.2. if the analysis has been performed accurately and in line with the conditions, the difference between the two individual results obtained by one analyser testing identical material and using the same equipment within a short time may not exceed 0.5 g of protein per 100 g of the product. This repeatability interval shall be 95 % of all determination cases.

**III. Determination of total acidity (method 3)**

1. In employing this method, the total acidity of edible acid caseins shall be determined.

2. The total acidity in edible acid caseins shall be a volume of 0.1 mol/l of standard solution of sodium hydroxide in millilitres which is necessary to neutralise the extract obtained from 1 g of the product.

3. The principle of the method – a test sample shall be extracted at a temperature of 60 °C and filtered. The filtrate shall be titrated with a standard solution of sodium hydroxide using phenolphthalein as an indicator.

4. The required reagents:

4.1. water which is boiled for 10 minutes before use so that it contains no carbon dioxide;

4.2. 0.1 mol/l of standard solution of sodium hydroxide;

4.3. a neutralised phenolphthalein indicator solution in 10 g/l ethanol (95 % V/V).

5. The required equipment:

5.1. an analytical balance;

5.2. a 500 ml conical flask with a suitable ground glass plug;

5.3. a 100 ml pipette;

5.4. a pipette suitable for measuring 0.5 ml of indicator solution;

5.5. a 250 ml conical flask;

5.6. a measuring cylinder, capacity 250 ml;

5.7. a burette, graduated in 0.1 ml;

5.8. a water bath capable of being maintained at a temperature of 60 ± 2 °C;

5.9. a suitable filter.

6. Procedure:

6.1. prepare a test sample in accordance with Paragraphs 24, 25, 26, 27, and 28 of this Regulation;

6.2. weigh approximately 10 g of the test sample to the nearest 10 mg and place into the 500 ml conical flask;

6.3. determine the total acidity:

6.3.1. place 200 ml of freshly boiled and chilled water into the measuring cylinder, capacity 250 ml, heat to 60 °C;

6.3.2. close the flask, shake it and place in a water bath for 30 minutes at a temperature of 60 °C shaking it approximately every 10 minutes. Filter, cool the filtrate to approximately 20 °C. The filtrate should be clear;

6.3.3. using a 100 ml pipette, transfer 100 ml of the cooled filtrate into the 250 ml conical flask. Add 0.5 ml of phenolphthalein indicator solution by pipette suitable for measuring 0.5 ml of indicator solution;

6.3.4. titrate with a standard solution of sodium hydroxide to a pink colour that should persist for at least 30 seconds;

6.3.5. determine and record the titrated volume to the nearest 0.1 ml.

7. Expression of results:

7.1. the total acidity of acid edible casein shall be calculated as follows to two decimal places:

[20 × V × T]/ m where

V – volume of the standard solution of sodium hydroxide used for titration (ml);

T – concentration of the standard solution of sodium hydroxide (mol/l);

m – mass of the test sample (g);

7.2. if the analysis has been performed accurately and in line with the conditions, the difference between the two individual results obtained by one analyser testing identical material and using the same equipment within a short time may not exceed 0.02 ml 0.1 mol/l of sodium hydroxide per 1 g of the product. This repeatability interval shall be 95 % of all determination cases.

**IV. Determination of ash (also P2O5) (method 4)**

1. In employing this method, the ash (also P2O5) content shall be determined in edible acid caseins.

2. The principle of the method – a test sample shall be ashed in the presence of magnesium acetate at a temperature of 825 ± 25 °C to attract the entire organic phosphorus. The quantity of ash shall be calculated by weighing the residue and subtracting it from the mass of ash resulting from magnesium acetate.

3. The required reagent shall be 120 g/l of a magnesium acetate tetrahydrate solution. 120 g of magnesium acetate tetrahydrate [Mg(CH3CO2)2 4H2O] shall be dissolved in water and made up to the one litre mark with water.

4. The required equipment:

4.1. an analytical balance;

4.2. a 5 ml pipette;

4.3. quartz or platinum containers with a diameter of approximately 70 mm and 25–50 mm high;

4.4. a drying cabinet where temperature can be maintained at 102 ± 1 °C;

4.5. an electric furnace capable of being maintained at a temperature of 825 ± 25 °C;

4.6. a bath for boiling water;

4.7. a dessicator with efficient sorbent.

5. Procedure:

5.1. prepare a test in accordance with Paragraphs 24, 25, 26, 27, and 28 of this Regulation;

5.2. prepare containers:

5.2.1. heat two containers (A, B) in an electric furnace for 30 minutes at a temperature of 825 ± 25 °C. Allow the containers to cool a bit and then transfer them into the dessicator;

5.2.2. allow the container to cool to the weighing room temperature, and weigh to the nearest 0.1 mg;

5.3. weigh in one of the prepared containers (A), to the nearest 0.1 mg, approximately 3 g of the test sample;

5.4. determination of ash:

5.4.1. using a pipette, transfer 5 ml of magnesium acetate solution into the first container (A) in order to moisten the entire test sample and allow to stand for 20 minutes;

5.4.2. using a pipette, transfer 5 ml of magnesium acetate solution into the second container (B). Evaporate the content of both containers (A and B) on a boiling water bath until dry mass;

5.4.3. place both containers in the furnace for 30 minutes where temperature is maintained at 102 ± 1 °C. Heat the container A and its content on a small flame, cooking plate, or under infrared lamp until the test sample is completely charred but ensuring that it does not deflagrate. Place both containers (A and B) in the electric furnace where temperature is maintained at 825 ± 25 °C and heat for at least an hour until all carbon disappears from the container A;

5.4.4. allow both containers to cool a bit and then transfer them into the dessicator, cool to the weighing room temperature, and weigh to the nearest 0.1 mg;

5.4.5. continue heating in the electric furnace referred to in Sub-paragraph 4.5 of this Chapter for approximately 30 minutes, repeat cooling and weighing until mass becomes constant not exceeding 1 mg or begins to increase. Mark the minimum mass.

6. Expression of results:

6.1. the ash (including P2O5) content in the sample which is expressed as a percentage proportion to mass shall be calculated as follows to the nearest 0.01 %:

[[(m1 – m2) – (m3 – m4)] /m0] × 100 where

m0 – mass of the test sample (g);

m1 – mass of the container A and residue (g);

m2 – mass of the prepared container A (g);

m3 – mass of the container B and residue (g);

m4 – mass of the prepared container B (g);

6.2. if the analysis has been performed accurately and in line with the conditions, the difference between the two individual results obtained by one analyser testing identical material and using the same equipment within a short time may not exceed 0.1 g per 100 g of the product. This repeatability interval shall be 95 % of all determination cases.

**V. Determination of ash (also P2O5) (method 5)**

1. In employing this method, the ash (also P2O5) content shall be determined in edible rennet caseins.

2. The principle of the method – a test sample shall be ashed at a temperature of 825 ± 25 °C until constant mass. The residue of the ashed sample shall be weighed and calculated as a percentage proportion to the sample weight.

3. The required equipment:

3.1. an analytical balance;

3.2. a quartz or platinum container with a diameter of approximately 70 mm and 25-50 mm high;

3.3. an electric furnace with air circulation capable of being maintained at a temperature of 825 ± 25 °C by using a thermostat;

3.4. a dessicator containing freshly activated silica gel with a water content indicator or equivalent dehydrator.

4. Procedure:

4.1. prepare a test sample in accordance with Paragraphs 24, 25, 26, 27, and 28 of this Regulation;

4.2. preparation of a container:

4.2.1. heat the container in an electric furnace for 30 minutes at a temperature of 825 ± 25 °C;

4.4.2. allow the container to cool a bit and then transfer it into the dessicator, cool to the weighing room temperature;

4.2.3. weigh the container to the nearest 0.1 mg;

4.3. weigh in the container, to the nearest 0.1 mg, approximately 3 g of the test sample;

4.4. determination of ash:

4.4.1. heat the container and its content on a small flame, cooking plate, or under infrared lamp until the test sample is completely charred but ensuring that it does not deflagrate;

4.4.2. transfer the container into the electric furnace at a temperature of 825 ± 25 °C and heat for at least an hour until all carbon disappears from the container;

4.4.3. allow the container to cool a bit and then transfer it into the dessicator, cool to the weighing room temperature, and weigh to the nearest 0.1 mg;

4.4.4. repeat heating in the electric furnace for approximately 30 minutes, and also repeat cooling and weighing until mass becomes constant not exceeding 1 mg or begins to increase. Mark the minimum mass.

5. Expression of results:

5.1. the ash (also P2O5) content in the sample which is expressed as a percentage proportion to mass shall be calculated as follows to the nearest 0.01 %:

[(m1 – m2)/ m0] × 100 where

m0 – mass of the test sample (g);

m1 – mass of the container and residue (g);

m2 – mass of the prepared container (g);

5.2. if the analysis has been performed accurately and in line with the conditions, the difference between the two individual results obtained by one analyser testing identical material and using the same equipment within a short time may not exceed 0.15 g of ash per 100 g of the product. This repeatability interval shall be 95 % of all determination cases.

**VI. Determination of pH value (method 6)**

1. In employing this method, the pH value shall be determined in caseinates.

2. The pH value in caseinates shall constitute the pH value of aqueous caseinate solution at a temperature of 20 °C determined by employing the method described in this Chapter.

3. The principle of the method – electrometric determination of the pH value for an aqueous caseinate solution by using the pH meter.

4. The required reagents:

4.1. water used in the procedure and preparation of reagents shall be freshly distilled preventing adsorption of carbon dioxide;

4.2. two standard borax buffer solutions for calibration of the pH meter the pH values of which at a temperature of 20 °C are known to the second decimal place and bracket the pH value of the tested sample, for example, a phthalate buffer solution of pH value approximately 4 and a borax buffer solution of pH value approximately 9.

5. The required equipment:

5.1. a balance accurate to 0.1 g;

5.2. a pH meter with the minimum sensitivity of 0.05 pH units and an appropriately calibrated electrode, such as glass, calomel, or another reference electrode;

5.3. a thermometer accurate to 0.5 ° C;

5.4. a 100 ml conical flask with a suitable ground glass plug;

5.5. a measuring cylinder, capacity 50 ml;

5.6. a blender;

5.7. a blender measuring cylinder, capacity 250 ml.

6. Procedure:

6.1. prepare a test sample in accordance with Paragraphs 24, 25, 26, 27, and 28 of this Regulation;

6.2. determine the pH value:

6.2.1. calibrate the pH meter – adjust the temperature of the buffer solution to 20 °C, calibrate the pH meter in accordance with the manufacturer’s instructions. Carry out calibration while holding the flask with the sample for 20 minutes in accordance with Sub-paragraph 6.2.2 of this Chapter. Where a series of samples is tested with one or more standard buffer solutions, check the calibration of the pH meter at least once every 30 minutes;

6.2.2. for the purpose of preparing a test solution, place 95 ml of water in the measuring cylinder, add 5.0 g of the test sample, and mix for 30 seconds by using the blender. Cover the flask with a slide and hold for 20 minutes at a temperature of approximately 20 °C;

6.2.3. place approximately 20 ml of solution in the measuring cylinder and immediately read the pH value of the solution by using the pH meter;

6.2.4. rinse glass electrodes with water.

7. Expression of results:

7.1. the pH value shall be registered by marking the value read from the pH meter to two decimal places as the pH value of aqueous caseinate solution;

7.2. if the analysis has been performed accurately and in line with the conditions, the difference between the two individual results obtained by one analyser testing identical material and using the same equipment within a short time may not exceed 0.05 pH units. This repeatability interval shall be 95 % of all determination cases.

Minister for Agriculture Jānis Dūklavs