#### NOTE BY THE SECRETARIAT

- 1. In October 1949, the Committee on Agricultural Problems established the Working Party on Standardization of Perishable Foodstuffs. <sup>1</sup>
- 2. The Working Party was entrusted with the task of "determining common standards for perishable foodstuffs" and studying the steps to be taken at the international level in order to secure the general adoption of "standards and control systems".
- 3. It should be noted that the standards drawn up by the Working Party concern only goods moving in trade between and to European countries and that they are applied at the dispatch stage and therefore by the competent service of the exporting countries. The standards for individual kinds of fruit and vegetables are drawn up with the framework of a Protocol, adopted in 1958 and revised at the seventeenth session of the Working Party in 1964, which contains general provisions for this type of produce.
- 4. At its twelfth session, held in Geneva from 12-15 June 1961 the Working Party decided that the standards which it had approved in final form should henceforth be designated as European standards recommended by the Working Party on Standardization of Perishable Foodstuffs.
- 5. The standard contained in this document was approved in final form at the thirty-second session of the Working Party; the secretariat has been instructed to transmit this standard to Governments of member countries of the Economic Commission for Europe for official acceptance.

At the twenty-seventh session of the Committee on Agricultural Problems the Committee agreed to change the name of the Working Party to the "Working Party on Standardization of Perishable Produce" (ECE/AGRI/20, para. 43).

#### **UNECE STANDARD**

concerning the marketing and commercial quality control of

# HENS EGG PRODUCTS FOR USE IN THE FOOD INDUSTRY

moving in international trade between and to UNECE member countries (adopted in 1986)

#### I. SCOPE

This standard applies to hens egg products for use in the food industry for human consumption and which are marketed as "UN/ECE QUALITY". These products may be in the dried, liquid or frozen form.

# II. DEFINITIONS USED IN THE STANDARD

#### Egg products:

the products referred to in Section I above, fit for human consumption. Egg products may contain certain permitted food additives necessary for manufacture.

# Whole egg:

the homogeneous product obtained from the complete contents of broken out hens eggs-in-shell, in accordance with good manufacturing practice. Small quantities of egg albumen or egg yolk may be added to whole egg in order to standardize the product so that the compositional requirements set out in Section III are met.

#### Egg yolk:

the homogeneous product produced from the separation of the yolk of broken out hens-eggs-in-shell, in accordance with good manufacturing practice. Small quantities of egg albumen may be added to egg yolk in order to standardize the product.

# Egg albumen:

the homogeneous product obtained from the separation of the white of broken out hens eggs-in-shell, in accordance with good manufacturing practice.

# Liquid egg product:

the liquid product obtained from whole egg and/or egg yolk and/or egg albumen without the addition or removal of water.

# Frozen egg product:

a product obtained from a liquid egg product which has been subjected to a freezing or quick-freezing process, including deep freezing, and maintained in the frozen condition.

# **Dried egg product:**

a product obtained from a liquid egg product from which water has been removed by a drying process to give a powdered or granular product.

# **Concentrated egg product:**

an egg product with a higher solids content than the equivalent liquid or frozen product obtained by the removal of water.

# Blended egg product:

an egg product prepared in such a way that the proportion of the constituents of broken out hens eggs-in-shell is altered from the whole egg, egg yolk and egg albumen.

# Commodity lot or batch:

is a definite quantity of egg product produced under conditions which are presumed to be uniform, i.e., between any planned breaks in production.

#### Food additive:

means any substance defined as a food additive by the Codex Alimentarius Commission [Procedural Manual of the Codex Alimentarius Commission, 6th ed., 1986, p. 33].

# III. PROVISIONS CONCERNING QUALITY

#### (i) General

The purpose of this standard is to define the quality requirements which the produce must satisfy at all stages of marketing, after preparation and packaging; but the standard does not apply to products sold by retail.

# (ii) General quality criteria

Egg products must be homogeneous, fit for human consumption, practically free from shell fragments, and foreign matter. The taste, colour and odour of egg products shall be natural and characteristic of each product. In the case of dried egg products these shall be easily reconstituted.

#### (iii) Source material

The only permitted source material of egg for egg products shall be as follows:

- (a) hens eggs-in-shell complying with -
  - 1. UN/ECE Standard No. 42 concerning the Marketing and Quality Control of Eggs in Shell for direct consumption moving in trade between and to European countries.
  - 2. UN/ECE Standard No. 43 concerning the Marketing and Quality Control of Eggs in Shell for Processing moving in trade between and to European countries.
- (b) The contents of hens eggs-in-shell satisfying the above UN/ECE standards but which have cracked shells and provided that they are broken out and treated as soon as possible. <sup>2</sup>
- (c) Egg products which satisfy the requirements specified in this standard.
- (d) Egg products which satisfy the requirements of this standard except the criteria applying to the fat content and egg solids (dry matter) content as specified in Section III (iv).
- (e) Unpasteurized egg products otherwise conforming to the requirements specified in (c) and (d) above and in this case they may be imported and used only under the authority and

The legislation of the Federal Republic of Germany prohibits the use of eggs with cracked shells where the membrane has been ruptured if the egg content was not immediately removed after the rupture of the membrane.

supervision of the official agency having jurisdiction.

- (f) The permitted source material shall not include:
  - egg products obtained by the crushing of eggs-in-shell <sup>3</sup> <sup>4</sup>
  - albumen retrieved from shells by centrifugal separation. 4

#### (iv) Compositional criteria

Liquid, frozen or dried whole egg, egg yolk and egg albumen shall comply with the specifications given below. The percentages expressing composition shall be based on the egg portion only of the egg product.

PRODUCT	EGG SOLIDS	FAT CONTENT	FREE FATTY ACIDS
	CONTENT	(minimum per cent)	(maximum per cent)*
	(minimum percent)		
Liquid and frozen whole egg	23.5	9.8	-
Liquid and frozen egg yolk	43.0	26.0	-
Liquid and frozen egg albumen	10.5	-	-
Dried whole egg	95.0	39.0	3.5
Dried egg yolk	95.0	56.0	3.5
Pan dried egg albumen	84.0	-	-
Spray dried egg albumen	92.0	-	-

<sup>\*</sup> Free fatty acids (expressed as oleic acid) maximum per cent of the fat content of the product.

The composition of blended and concentrated egg products will depend on the specifications of manufacturers.

# (v) Treatment of egg products

- (a) Egg products shall be pasteurized in accordance with Section 4.4.4.5 (Pasteurization) of the Joint FAO/WHO Codex Alimentarius Commission Code of Hygienic Practice for Egg Products. The egg products must be treated in an establishment approved by the official agency having jurisdiction.
- (b) Egg products may be desugared or adjusted for pH.

# (vi) Microbiological criteria 5

In addition to any national requirements, the microbiological condition of the egg products shall be in conformity with the following minimum requirements:

#### Salmonellae:

(a) Salmonella organisms should not be recovered from any of ten sample units examined when the test is carried out according to the method described (n n 10, c = 0, m = 0).  $^6$ 

<sup>&</sup>lt;sup>3</sup> The legislation of the Federal Republic of Germany prohibits the use of eggs with cracked shells where the membrane has been ruptured if the egg content was not immediately removed after the rupture of the membrane.

<sup>&</sup>lt;sup>4</sup> Reservation of the United Kingdom which is of the opinion that source material obtained by crushing of eggs-inshell and centrifugal separation should be permitted by the standard.

Recommended International Code of Hygienic Practice for Egg Products (Addendum 1-1978 CAC/RCP 15-1976), Section 5, page 32, Sampling Plan and Microbiological Limits (paragraphs 4 and 4.1 only).

n = The number of sample units to be examined.

m = The value at or below which no concern is recognized.

(b) In products intended for special dietary purposes, salmonella organisms should not be recovered from any of thirty sample units examined (n = 30, c = 0, m = 0).

# Mesophilic aerobic bacteria

Mesophilic aerobic bacteria should not be recovered from any of five sample units examined when the test is carried out according to the method described in a number exceeding one million per gramme, nor in a number exceeding 50,000 per gramme from three or more of the five sample units examined. (n = 5, c = 2,  $m = 5 \times 10^4$ ,  $M = 10^6$ ).

#### Coliform bacteria

Coliform bacteria should not be recovered from any of five sample units examined, when the test is carried out according to the method described, in a number exceeding 1,000 per gramme, not in a number exceeding ten per gramme from three or more of the five sample units examined. (n = 5, c = 2, m = 10, M =  $10^{3}$ ).

# IV. PROVISIONS CONCERNING FOOD ADDITIVES

The use of food additives shall be in accordance with the legislation of the importing country. 8

# V. PROVISIONS CONCERNING CONTAMINANTS

Egg products shall not contain contaminants such as pesticide residues, antibiotics, hormones or heavy metal contaminants in amounts greater than those specified in the legislation of the importing country. <sup>9</sup>

M = The value beyond which the lot is rejected.

c = The maximum number of sample units with values between m and M for the lot to be acceptable.

These criteria are employed in describing 3-class plans. In a 2-class plan M is not applicable.

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In applying this provision Governments should refer to the recommendations of the Codex Alimentarius Commission.

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# VI. PROVISIONS CONCERNING HYGIENE

- (i) The hygiene requirements for the production of egg products and the premises, equipment and personnel used or engaged in their production should be as specified in the Joint FAO/WHO Codex Alimentarius Commission Code of Hygienic Practice for Egg Products.
- (ii) In addition, egg products shall satisfy the appropriate tests as specified in Section IX (i) of this standard.
- (iii) The permitted source material of Section III (iii) (b) of the standard not prepared in an egg products processing establishment shall be prepared in accordance with the requirements prescribed in the International Code of Hygienic Practice for Egg Products (CAC RCP 15-1976, Codex Alimentarius Commission ALINORM 85 13).

# VII. PROVISIONS CONCERNING PACKING, TRANSPORT, AND STORAGE

- (i) Egg products shall be packed in such a manner as to protect them adequately, and to prevent contamination. The packaging material shall not impart any taste, odour or colour to the egg products and shall be in accordance with legislation of the importing country.
- (ii) The transport of egg products should be in accordance with the requirements of the Joint FAO/WHO Codex Alimentarius Commission Code of Hygienic Practice for Egg Products. In addition the transport of frozen egg products should conform to the Agreement on the International Carriage of Perishable Foodstuffs and on the Special Equipment to be used for such Carriage (ATP).
- (iii) The storage of egg products should be in accordance with the requirements of the Joint FAO/WHO Codex Alimentarius Commission Code of Hygienic Practice for Egg Products.

#### VIII. PROVISIONS CONCERNING MARKING

# A. Marking of Packages

Packages containing egg products shall bear the following particulars in characters which are conspicuous, clearly legible and indelible:

- (i) the appropriate description of the product as indicated below:
  - (a) liquid whole egg
  - (b) frozen whole egg
  - (c) dried whole egg
  - (d) liquid egg yolk
  - (e) frozen egg yolk
  - (f) dried egg yolk
  - (g) liquid egg albumen
  - (h) frozen egg albumen
  - (i) pan dried egg albumen
  - (j) spray dried egg albumen
  - (k) liquid blended/concentrated egg
  - (1) frozen blended/concentrated egg

- (m) dried blended/concentrated egg
- (ii) The following indications, as appropriate:
  - (a) the indication "pasteurized", or the indication "heat-treated" where the albumen has been heat-treated
  - (b) the indication "desugared" where the product has been desugared
  - (c) the indication "acidified" where the product has been so treated.
- (iii) When products are marketed as UN/ECE Quality they must bear the marking "UN/ECE QUALITY".
- (iv) A list of ingredients, including food additives present, in descending order by weight, except where no food additives have been used.
- (v) The name, or trade name, and address of the manufacturer, packer, distributor, exporter, importer, or vendor.
- (vi) An identification number of the egg product processing establishment and the commodity lot number, each lot being allocated a sequential number.
- (vii) The country of origin where its omission could mislead the consumer. Where the egg product undergoes processing in a second country which changes its nature, the country in which the processing is performed shall be considered as the country of origin for the purposes of this provision.
- (viii) The net weight in either SI (Système international) units, or avoirdupois.
- (ix) Date marking: either the date of manufacture or date of minimum durability ("best before").
- (x) The minimum percentage of egg solids content and the minimum percentage of fat content, based on the egg portion only of the egg product, as far as concentrated and blended egg products are concerned.

# B. Marking of bulk containers

Where liquid egg products are marketed in a tanker churn or other suitable bulk container the information specified in sections (i) to (v), (viii) to (x) above may be provided in accompanying documents. The information provided for in (vi) above shall appear on the container.

# IX. PROVISIONS CONCERNING METHODS OF ANALYSIS

(i) Pasteurization of egg products shall be determined by an appropriate test. Where the alpha-amylase test is used it should be performed as specified in the Joint FAO/WHO Codex Alimentarius Commission Code of Hygienic Practice for Egg Products.

#### **ANNEX**

# METHODS OF ANALYSIS AND SAMPLING

# 1. PREPARATION OF THE ANALYSIS SAMPLE

#### 1.1 General

The sample must be made homogeneous prior to analysis and kept in a hermetically sealed jar in a cool place.

For all frozen samples, the sample is allowed to thaw, or is warmed in a water bath of temperature less than 50 °C, homogenized and treated as for liquid samples in all analyses.

For all dried samples the sample is prepared for analysis by passing three times through a seive of approximately 1 mm square mesh to thoroughly break up any lumps.

# 2. REAGENTS

# 2.1 Water

- 2.1.1 Wherever water is stated for dissolution, dilution or washing purposes, distilled water, or water of at least equivalent purity shall be used.
- 2.1.2 Wherever reference is made to "dissolution", "solution" or "dilution" without further indication, "dissolution in water", "solution in water" or "dilution with water" is meant.

#### 2.2 Chemicals

All chemicals used shall be of recognized analytical reagent quality except where otherwise specified.

# 3. APPARATUS

# 3.1 Lists of apparatus

The lists of apparatus contain only those items with a specialized use and items with a special specification.

#### 3.2 Analytical balance

Analytical balance means a balance capable of weighing to the nearest 0.1 mg.

# 4. EXPRESSION OF RESULTS

# 4.1 Number of significant figures

The result shall not contain more significant figures than are justified by the precision of the method of analysis used.

# 5. TEST REPORT

The test report shall give all the information necessary for the complete identification of the sample.

# II. SAMPLING

# 1. SAMPLING OF LIQUID WHOLE EGG, LIQUID YOLK AND LIQUID ALBUMEN

# 1.1 Samples taken from process vessels

Samples for chemical examination are taken from sample cocks on process vessels or from the top.

It is essential to ensure that the sample cock has been properly cleaned and also that at least 10 litres of product is withdrawn prior to taking the sample. The contents of the vessel must be thoroughly mixed immediately prior to sampling.

# 1.2 Samples from churns, mobile containers, road tankers, etc.

Samples should be taken by "dipping" the well mixed container, etc., by means of a clean milk churn dipper.

# 2. SAMPLING OF FROZEN WHOLE EGG, FROZEN YOLK AND FROZEN ALBUMEN

Frozen Eggs - Obtain representative container or containers. Examine contents as to odour and appearance (condition of contents can be determined best by drilling to the centre of the container with an auger and noting the odour as the auger is withdrawn. If impossible to secure individual containers, the sample may consist of composite borings from the contents of each container). Take borings diagonally across the container from > 3 widely separated parts, starting from 2 to 5 centimetres in from the edge and extending to the opposite side as near to the bottom as possible. Pack the shavings tightly into a sample jar and fill it completely to prevent partial dehydration of the sample. Seal the jar tightly and store in freezer or with solid  $CO_2$ . Before analysing, warm the sample in a bath with water temperature held at < 50 and mix well.

# 3. SAMPLING OF DRIED WHOLE EGG, DRIED YOLK, AND DRIED ALBUMEN

#### 3.1 From spray drier outlet

Hold a clean container (ca 500 ml) beneath outlet and allow to fill.

<sup>&</sup>lt;sup>10</sup> Official Methods of Analysis of the AOAC edited by W. Horowitz, 14th Edition, published 1984 by AOAC, Washington.

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# 3.2 From sacks, etc.

Transfer to clean container (ca 500 ml) by means of clean spatula or scoop.

# METHOD 1 - DETERMINATION OF TOTAL SOLID MATTER (VACUUM OVEN, 99 °C)

# 1. SCOPE AND FIELD OF APPLICATION

The method allows the determination of the total solid matter content in:

- liquid whole egg product
- liquid yolk product
- liquid albumen product
- frozen whole egg product
- frozen yolk product
- frozen albumen product
- dried whole egg product
- dried yolk product
- dried albumen product
- liquid blended/concentrated egg product
- frozen blended/concentrated egg product
- dried blended/concentrated egg product

# 2. **DEFINITION**

Total solid matter content: the content of total solid matter as determined by the method specified.

# 3. PRINCIPLE

The total solid content is determined by drying the sample in a vacuum oven at an absolute pressure less than 2.2 kPa and at a temperature of  $99\pm1$  °C.

# 4. APPARATUS

# 4.1 Metal weighing dishes,

flat bottomed, resistant to attack by the samples and the conditions of the test.

# 4.2 Vacuum drying oven,

thermostatically controlled at 99 ±1 °C, equipped with a thermometer and manometer.

# 4.3 Desiccator,

containing freshly activated silica gel with a water content indicator or an equivalent desiccant.

## 4.4 Water bath,

boiling.

# 4.5 Analytical balance

# 5. PROCEDURE

- 5.1 Dry a weighing dish and lid (4.1) to constant weight in the oven (4.2) at 99  $\pm$ 1 °C.
- 5.2 Allow the dish with lid to cool in the desiccator (4.3) to ambient temperature and weigh to the nearest 0.1 mg.
- 5.3 Accurately weigh approximately 5 g of liquid or frozen egg product samples or approximately 2 g of dried egg product sample into the weighed dish. Place dish on a boiling water bath (4.4) to evaporate most of the water in the samples.
- 5.4 Replace lid loosely on dish, put in the vacuum oven and dry for approximately 5 hours at 99 ±1 °C. Admit dry air into oven to restore to atmospheric pressure, tighten the lid on the dish and transfer to the desiccator. Allow to cool to ambient temperature and weigh.
- 5.5 Repeat process 5.4 but drying for 2 hour intervals until constant weight is obtained.

#### 6. EXPRESSION OF RESULTS

#### 6.1 Formula and Method of Calculation

The total solid matter content, expressed as a percentage by weight of the sample is given by:

$$\begin{array}{ccc} m_{1} & \\ & x & 100 \\ m_{o} & \end{array}$$

where:

m<sub>o</sub> is the mass, in g, of the test portion

m<sub>1</sub> is the mass, in g, of the test portion after drying and when constant weight has been obtained.

# 6.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst on the same sample shall not exceed 0,1 g dry matter per 100 g of sample.

# 7. NOTES

7.1 This method is the same in principle as that described in the 14th edition (1984) of the Official Methods of Analysis of the Association of Official Chemists, section 17.006 - 17.007.

# METHOD 2 - DETERMINATION OF FAT CONTENT

# 1. SCOPE AND FIELD OF APPLICATION

The method allows the determination of fat in:

- liquid whole egg product
- liquid yolk product
- frozen whole egg product
- frozen yolk product
- dried whole egg product
- dried yolk product
- liquid blended/concentrated egg product
- frozen blended/concentrated egg product
- dried blended/concentrated egg product

# 2. **DEFINITION**

The fat content of egg products: the fat content as determined by the method specified.

# 3. PRINCIPLE

The sample is hydroliyzed by hydrochloric acid and the fat released is extracted by petroleum ether, recovered and calculated as a percentage by weight of the original sample.

Samples contained added salt and sugar are further extracted using a Soxhlet extraction of the acid hydrolysis residues.

# 4. **REAGENTS**

# 4.1 Hydrochloric acid,

concentrated (assay 36.5 - 38 % HCl).

# 4.2 Diethyl ether

# 4.3 Petroleum ether,

with any boiling range between 30 and 60 °C.

# 5. APPARATUS

# 5.1 Mojonnier extraction tube

#### 5.2 Water bath

capable of being thermostatically controlled over the range 70 - 100 °C.

#### 5.3 Oven

capable of being thermostatically controlled at  $100 \pm 1$  °C.

# 5.4 Soxhlet apparatus

with suitable thimbles.

# 5.5 Analytical balance

#### 6. PROCEDURE

- 6.1 Accurately weigh, approximately 2 g liquid or frozen yolk product, 3 g of liquid or frozen whole egg product or 1 g dried yolk or whole egg product into a Mojonnier fat extraction tube (5.1), by difference. Slowly add, with vigorous shaking, 10 ml of hydrochloric acid (4.1) and, in the case of dried products, about 2 ml water, washing down any egg particles adhering to the sides of the tube.
- 6.2 Put the tube with sample in water bath (5.1) set at 70 °C, bring to boil and continue heating at boiling point for 30 minutes. Carefully shake the tube every 5 minutes during this time. After 30 minutes remove the tube, add water to nearly fill the lower bulb of the tube and cool to room temperature.
- 6.3 Add 25 ml of diethyl ether (4.2) to the tube with the treated sample and mix. Then add 25 ml of petroleum ether (4.3), mix and allow to stand until the solvent layer has cleared.
- 6.4 Draw off as much as possible of the ether-fat solution into a previously weighed clean dry 125 ml beaker/flask containing anti-bumping granules. Before weighing the beaker/flask dry it and a similar flask as counterpoise in an oven (5.3) at 100 °C and allow to stand in air until constant weight is obtained.
- 6.5 Re-extract the liquid remaining in the tube twice but use 15 ml of ether each time. Thoroughly shake on each addition of ether. Allow solutions to clear and draw off ether-fat solution into flask as previously.
- 6.6 Slowly evaporate the ether from the flask by carefully placing on a boiling water bath. Dry the fat by placing in the oven (5.3) at 100 °C till constant weight is reached (probably after about 90 mins). Remove flask and counterpoise from the oven and allow to cool to constant weight at ambient temperature in air (note: owing to the size of the flask and the nature of the material under test there is less error by cooling in air than by cooling in a desiccator). Correct the weight obtained by a blank determination on the reagents used.

# 7. EXPRESSION OF RESULTS

#### 7.1 Formula and Method of Calculation

The fat content, as a percentage by mass of the sample, is given by:

$$\begin{array}{ccc} m_{1} & \\ & x & 100 \\ m_{o} & \end{array}$$

where:

 $m_0$  is the mass, in g, of the test portion of the egg product sample (6.1)

m<sub>1</sub> is the mass, in g, of the fat obtained after extraction and blank correction.

# 7.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst on the same sample shall not exceed 0.3 g fat per 100 g of sample.

#### 8. NOTES

- 8.1 The fat content of an egg product containing salt and sugar is obtained using the above procedure except that the fat is further extracted from the acid solution obtained after the third extraction by the following procedure:
  - 8.1.1 Filter the aqueous layer remaining after extraction through a filter paper and wash filter paper with hot water until the washings do not affect the colour of blue litmus paper. Place the filter paper on a watch glass or Petri dish and dry for 1 hour in an oven (5.3) at 100 °C. Allow to cool and then insert into an extraction thimble of a Soxhlet apparatus (5.4) using tongs to handle the filter paper. Remove any traces of fat from the watch glass or Petri dish with cotton wool moistened with petroleum ether extraction solvent (4.3) and then place cotton wool in the thimble. Place the thimble in the extraction tube.
  - 8.1.2 Add extraction solvent (4.3) to the Soxhlet and extract for 4 hours by placing the extraction flask on a sand bath or water bath or some such similar apparatus. After extraction remove the solvent from the extraction flask and treat as in section 6.6.
  - 8.1.3 Add the weight of fat obtained after section 8.1.2 to the weight obtained after section 6.6 to give a corrected weight m<sub>1</sub>, the mass, in g, of the fat obtained after extraction.
- 8.2 This method is the same in principle as that described in the 14th edition (1984) of the Official Methods of Analysis of the Association of Official Analytical Chemists, sections 17.012 17.013.

The further Soxhlet extraction procedure (8.1) is the same in principle as that described in CAC/RM 55-1976 Determination of Fat in Foods for Infants and Children; Method 1.

# METHOD 3 - ALPHA - ANALYSE TEST

# 1. SCOPE AND FIELD OF APPLICATION

The efficiency of pasteurization is determined in:

- liquid whole egg product
- liquid yolk product
- frozen whole egg product
- frozen yolk product
- dried whole egg product
- dried yolk product
- liquid blended/concentrated egg product
- frozen blended/concentrated egg product
- dried blended/concentrated egg product.

# 2. **DEFINITION**

The efficiency of pasteurization: the absence/presence of active alpha-amylase by the method specified.

# 3. PRINCIPLE

The presence of any active alpha-amylase (present in unheated or insufficiently pasteurized egg product) is indicated by its ability to break down added starch so preventing the formation of a starch iodide complex on subsequent addition of an iodine solution.

# 4. REAGENTS, APPARATUS, PROCEDURE AND INTERPRETATION

The method to be employed is the method adopted in the Codex Alimentarius Commission Code of Hygiene Practice for Egg Products CAC/RCP - 15-1976, Annex I.

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# METHOD 4. FREE FATTY ACIDS (CALCULATED AS OLEIC ACID)

# 1. SCOPE AND FIELD OF APPLICATION

The method determines the acidity of the diethyl ether extract, calculated as oleic acid, in:

- dried whole egg product
- dried yolk product
- dried blended/concentrated egg product.

# 2. **DEFINITION**

Free fatty acids content: the acidity of the diethyl ether extract, calculated as oleic acid, as determined by the method specified.

# 3. PRINCIPLE

The sample is extracted with diethyl ether. The ether is evaporated and the extracted residue is dissolved in toluene. The free fatty acid content is determined by titration against standard ethanolic sodium hydroxide solution using phenolphthalein indicator.

# 4. REAGENTS

# 4.1 Diethyl ether

# 4.2 Toluene.

Use best quality available. If not neutral titrate 50 ml against 0.05 mol/l standard ethanolic sodium hydroxide and correct subsequent results accordingly.

# 4.3 Phenolphthalein

1 % w/v in ethanol.

# 4.4 Ethanolic sodium hydroxide,

0.05 mol/l: dissolve a piece of metallic sodium, approximately 1 ml in volume, in 800 ml of absolute alcohol (ethanol). Titrate 10 ml standard 0.1 mol/l hydrochloric acid against this solution using phenolphthalein as indicator. Calculate the volume of ethanol needed to be added to the solution to make 0.05 mol/l. Standardize against 0.1 mol/l hydrochloric acid on each day the solution is used.

# 5. APPARATUS

# 5.1 Erlenmeyer flask,

lipped.

# 5.2 Water bath,

boiling.

# 5.3 Analytical balance

# **5.4** Oven,

capable of being thermostatically controlled at 100±1 °C.

#### 6. PROCEDURE

- 6.1 Accurately weigh approximately 2 g of dried egg sample into a small lipped Erlenmeyer flask (5.1), add 30 ml of diethyl ether (4.1) and thoroughly mix. Allow to clear and decant through a small filter paper into a flask. Repeat the extraction three further times using 20 ml of diethyl ether for each extraction.
- 6.2 Evaporate ether on a boiling water bath (6.2) and then dry the extract for 15 minutes in an oven (5.4) at 100 °C. Cool the extract, add 30 ml of toluene (4.2) 3-4 drops of phenolphthalein indicator solution (4.3) and titrate against standard ethanolic sodium hydroxide solution (4/4). The end point is reached when the yellow colour changes to orange.

# 7. EXPRESSION OF RESULTS

# 7.1 Formula and Method of Calculation

The free fatty acid content, calculated as oleic acid, of the sample is given by:

$$V_1$$
 x 2.81  $2m_0$ 

where:

 $V_1$  is the volume, in ml, of the standard 0.05 mol/l ethanolic sodium hydroxide used

M<sub>0</sub> is the mass, in g, of the sample taken

The free fatty acid content, calculated as oleic acid, and expressed on the fat portion of the egg product is given by:

$$V_1$$
 x 2.81 x 100  
2m<sub>0</sub> % fat

where:

 $V_1$  and  $m_0$  are as above

% fat is the percentage fat in the egg product as determined by method 2.

# 7.2 Repeatability

The difference between the results of two determinations when carried out simultaneously or in rapid succession by the same analyst on the same sample shall not exceed 0.3 g free fatty acid per 100 g of fat in the sample.

# 8. NOTES

- 8.1 The addition of salt or sugar to the egg product should not affect this determination.
- 8.2 This method is the same in principle as that described in the 14th edition (1984) of the Official Methods of Analysis of the Association of Official Analytical Chemists, sections 17.033 17.034.

# METHOD 5 - DETERMINATION OF EXTRANEOUS MATTER

(Provisional method <sup>11</sup>)

In order to determine the presence of shell residues or other extraneous matter, place 100 g of the substance under examination in a graduated cylinder of 1 000 ml capacity, add distilled water up to the mark (1 000 ml), mix carefully and pass through a sieve with perforations 1 mm in diameter. After sieving there should be no residue on the sieve.

For dried egg products, the test should be carried out on the reconstituted product.

<sup>&</sup>lt;sup>11</sup> This method has been tentatively accepted by the Group of Experts pending the development of a method which will detect particles of a size smaller than 1 mm.