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REGULATION (EEC) No 1061/69 OF THE COMMISSION

of 6 June 1969

specifying methods of analysis for the implementation of Regulation (EEC) No 1059/69 on the trade arrangements applicable to certain goods resulting from the processing of agricultural products

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community;

Having regard to Council Regulation (EEC) No 97/69[1] of 16 January 1969 on measures to be taken for uniform application of the nomenclature of the Common Customs Tariff, and in particular Article 3 thereof;

Whereas, in order to ensure that goods covered by Council Regulation (EEC) No 1059/69[2] of 28 May 1969 laying down the trade arrangements applicable to certain goods resulting from the processing of agricultural products are dealt with uniformly on import into the Community, it is necessary to specify methods of analysis and other technical provisions required either to identify certain goods or to determine their composition; whereas, in accordance with Article 4 (2) of that Regulation, such methods of analysis and other technical provisions must be adopted in accordance with the procedure laid down in Article 3 (2) and (3) of Regulation (EEC) No 97/69;

Whereas Regulation (EEC) No 1059/69 replaces Council Regulation No 160/66/EEC[3] of 27 October 1966 introducing trade arrangements in respect of certain goods resulting from the processing of agricultural products; whereas, for the purpose of implementing this latter Regulation, methods of analysis were fixed by Council Regulation No 83/67/EEC[4] of 18 April 1967 establishing tariff specifications relating to goods covered by Council Regulation No 160/66/EEC and determining the fixed components applicable to such goods and the quantities of basic products to be taken as having been used in their manufacture, as last amended by Regulation (EEC) No 2121/68[5]; whereas, subject to certain changes which have proved necessary in practice, those methods of analysis should continue to be applied; whereas, with regard to the detection of common wheat flour or meal in macaroni, spaghetti and similar products, it is, however, advisable to adopt a new method ensuring more accurate results;

Whereas the provisions of this Regulation are in accordance with the Opinion of the Committee on the Common Customs Tariff Nomenclature;

HAS ADOPTED THIS REGULATION:

Article 1

1. Where classification of goods referred to in Article 1 of Regulation (EEC) No 1059/69 under one or other of the subheadings of the Common Customs Tariff depends on the starch content by weight, such content shall be determined by reference to the quantity of starch in the anhydrous state contained in such goods.

2. The starch content by weight of goods shall be ascertained by the modified Ewers polarimetric method as set out in the Annex to Commission Regulation No 228/67/EEC[6] of 28 June 1967 on the determination of the starch content of compound feeding-stuffs, the starch and ash content of brans and the denaturing of flour of manioc and of other roots.

However, where the goods in question contain starches other than native but do not also contain

[1] OJ No L 14, 21.1.1969, p. 1.
[2] OJ No L 141, 12.6.1969, p. 1.
[3] OJ No 195, 28.10.1966, p. 3361/66.
[4] OJ No 81, 26.4.1967, p. 1597/67.
[5] OJ No L 311, 28.12.1968, p. 1.
[6] OJ No 136, 30.6.1967, p. 2925/67.

sucrose or invert sugar, the starch content by weight of such goods shall be ascertained by the saccharification method set out in Annex I hereto.

For purposes of implementation of this paragraph, dextrins shall be treated as starches other than native.

Article 2

For the purpose of ascertaining whether common wheat flour or meal is present in macaroni, spaghetti and similar products falling within heading No 19.03 of the Common Customs Tariff, the method set out in Annex II shall be employed.

Article 3

The milkfat content by weight of a product shall be ascertained by the method set out in Annex III.

Article 4

The mannitol content of products falling within sub-heading No 29.04 C III of the Common Customs Tariff, calculated by reference to the sorbitol content, shall be ascertained by the method set out in Annex IV.

Article 5

This Regulation shall enter into force on 1 July 1969.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 6 June 1969.

For the Commission

The President

Jean REY

ANNEX I

DETERMINATION OF STARCH BY THE SACCHARIFICATION METHOD

I. **Principle**

Starch is converted by acid hydrolysis into reducing sugars which are determined by volume using Fehling's solution.

II. **Apparatus and reagents**

1. 250 ml flask
2. 200 ml graduated flask
3. 25 ml graduated burette
4. Hydrochloric acid at 1·19 density
5. Potassium hydroxide solution
6. Decolourising charcoal
7. Fehling's solution
8. Methylene blue solution (1%)

III. **Method**

Into a 250 ml flask place a sample containing about 1 g of starch. Add 100 ml of distilled water and 2 ml of hydrochloric acid. Bring to the boil and reflux for three hours.

Transfer the contents of the flask and rinsings into a 200 ml graduated flask. Cool and nearly neutralise with potassium hydroxide solution. Add distilled water to 200 ml and filter through a little decolourising charcoal.

Then pour the solution into a graduated burette and reduce 10 ml of Fehling's solution by the following method:

Into a flat-bottomed flask of about 250 ml pour 10 ml of Fehling's solution (5 ml of solution A and 5 ml of solution B). Shake until clear and add 40 ml of distilled water and a small quantity of quartz or pumice.

Place the flask on a square asbestos plate with a round hole of about 6 cm diameter in the centre, the asbestos in turn resting on a piece of wire gauze. Heat the flask at such a rate that the liquid begins boiling after about two minutes.

From the burette, add to the boiling liquid successive quantities of the sugar solution until the blue colour of the Fehling's solution becomes hardly discernible; then add 2 or 3 drops of methylene blue solution as indicator, and complete the titration by adding further quantities of the sugar solution, drop by drop, until the blue colour of the indicator disappears.

For greater accuracy repeat the titration under the same conditions, but adding without a break almost all the sugar solution required to reduce the Fehling's solution. In this second titration, the reduction of the Fehling's solution should occur within three minutes.

The percentage by weight of starch in the sample is determined by means of the following formula:

$$\text{starch } \% = \frac{T \times 200 \times 100}{n \times p} \times 0{\cdot}90$$

where:

T is the quantity in grammes of anhydrous dextrose corresponding to 10 ml of Fehling's solution (5 ml of solution A and 5 ml of solution B). This titer corresponds to 0·04945 g of anhydrous dextrose when solution A contains 17·636 g of copper per litre.

n is the number of ml of the sugar solution used for titration.

p is the weight of the sample amount.

0·90 is the rate of conversion of anhydrous dextrose into starch.

Where necessary, the starch content so obtained is corrected by deducting the sugars previously existing in the sample calculated as starch.

IV. **Preparation of Fehling's solutions**

Solution A: In a graduated flask dissolve 69·278 g of pure crystallised copper sulphate—Analytical Reagent ($CuSO_4 \cdot 5\ H_2O$)—free from iron in distilled water and bring the volume to 1 litre with distilled water. The correct strength of this solution must be verified by a quantitative determination of the copper.

Solution B: In a graduated flask dissolve 100 g of sodium hydroxide and 346 g of double sodium potassium tartrate (Rochelle salt) in distilled water and bring the volume to 1 litre with distilled water.

The two solutions A and B must be mixed in equal quantities immediately before use. 10 ml of Fehling's solution (5 ml of solution A and 5 ml of solution B) is completely reduced, under the conditions described at III, by 0·04945 g of anhydrous dextrose.

ANNEX II

DETECTION OF COMMON WHEAT FLOUR OR MEAL IN MACARONI, SPAGHETTI AND SIMILAR PRODUCTS (PASTA)

(by the Young and Gilles method, modified by Bernaerts and Gruner)

I. Principle

An extract of the sample of the pasta for analysis is prepared by using a non-polar solvent.

This extract is chromatographed on a thin layer of silica gel so as to separate the sterols present in various band form fractions.

According to the number of brightly coloured bands it is possible to determine whether the product under examination has been manufactured exclusively from durum wheat or common wheat, or from a mixture of the two. It is also possible to determine whether eggs have been added.

II. Apparatus and reagents

1. Homogeniser or grinder to obtain a grist that will pass through a standard sieve with a 0·200 mm mesh
2. Standard sieve with a 0·200 mm mesh
3. Evaporator with a water bath for evaporation under reduced pressure
4. Glass plate, aluminium sheet or other appropriate backing measuring 20 cm × 20 cm covered with a thin layer of silica gel. If the thin layer has to be prepared, silica gel mixed with about 13% plaster should be used, and it should be applied in a 0·25 mm layer with suitable apparatus in accordance with the manufacturer's instructions
5. Micropipette for measuring 20 microlitres
6. Container with lid suitable for the development of chromatograms
7. Atomiser
8. Petroleum ether with a boiling point between 40 and 60 °C, redistilled before use
9. Anhydrous ethyl ether for analysis
10. Carbon tetrachloride for chromatography, redistilled before use
11. Phosphomolybdic acid for analysis
12. 94° ethyl alcohol

III. Method

Grind about 20 g of the sample for analysis so that all of it passes through the sieve. Put the sample in an Erlenmeyer flask and cover with 150 ml petroleum ether. Leave at normal temperature until the following day. Shake from time to time.

Then filter on a Büchner funnel fitted with a filtering aid or on a sintered filter. Gradually transfer the clear solution thus obtained into a 100 ml calibrated flask. Evaporate the solvent under reduced pressure by heating the flask in a water bath at 40–50 °C. When the solvent has evaporated, heat under reduced pressure for a further ten minutes.

When the flask has cooled, determine the weight of the extract. Dilute the extract in ethyl ether on the basis of 1 ml ethyl ether per 60 mg of extract.

Activate the thin layers by bringing them to 130 °C for three hours. Leave to cool in a desiccator containing silica gel. Plates which are not used immediately can be preserved in the same desiccator.

Apply, drop by drop, 20 microlitres of the clear solution to form a band of constant width and 3 cm in length on a layer preferably newly activated. Let the solvent evaporate.

Develop the chromatogram under normal temperature with carbon tetrachloride using a chromatographic container the walls of which are covered with filter paper soaked in

solvent. After about an hour the solvent will reach a height of 18 cm. Remove the plate and leave the solvent to evaporate in the open. For better separation of the bands, develop the chromatogram a second time. Again let the solvent evaporate in the open.

Spray the thin layer of silica gel with a solution of 20% phosphomolybdic acid in ethyl alcohol. The colour of the layer must be a uniform yellow. Develop the bands by heating the sprayed plate at 110 °C for five minutes.

IV. **Interpretation of the chromatogram**

If the chromatogram shows a single main brightly coloured band with an Rf of about 0·4–0·5, the wheat used for the manufacture of the pasta in question is durum wheat. If, on the other hand, two main bands of equal brightness appear, the raw material used is common wheat. Mixtures of durum wheat and common wheat can be assessed by an evaluation of the relative brightness of the two bands.

If there are three bands (2 bands at the height where the main bands for common wheat are to be found, with a further band between them) eggs have been added to pasta. In this case, the raw material used is durum wheat if the middle band is brighter than the upper band. On the other hand, if the upper band is brighter than the middle band, the raw material used is common wheat.

ANNEX III

DETERMINATION OF MILKFAT CONTENT

I. **Principle**

After determining the percentage by weight of total fat contained in the sample, the butyric index of this total fat content should be determined; the milkfat content can then be determined by using 20 as the average butyric index of milkfat.

II. **Apparatus and reagents**

1. Distillation apparatus with refrigerated reflux condenser
2. Beckel tubes
3. Alcoholic solution of potassium hydroxide prepared by mixing 40 ml of potash solution (d = 1·5) with 40 ml distilled water, and diluting to one litre with 95—96% alcohol
4. Pure glycerine (d = 1·23)
5. Saturated solution of potassium sulphate
6. Sulphuric acid diluted (1 volume concentrated sulphuric acid (d = 1·84) and 3 volumes distilled water)
7. Coconut soap solution, obtained as follows:

   In a 1 litre flask, saponify 50 g of pure coconut fat, refined but not hardened (melting point 24–26 °C), with 50 g of glycerine, 15 g KOH and 20 ml of water. After cooling to below 100 °C, dilute to 500 ml
8. 1% alcoholic solution of phenolphthalein
9. Aqueous 0·01 N solution of sodium hydroxide

III. Method

First determine the percentage by weight of the total fat content of the sample to be analysed. Then determine the butyric index of this total fat content by the Grossfeld method, as follows:

Weigh accurately 500 to 550 mg of fat and transfer it into a 50 ml flat-bottomed flask with 5 ml of alcoholic solution of potassium hydroxide. Keep at moderate boil under reflux. When the fat has been completely saponified, remove refrigerated reflux condenser, and using a graduated pipette add 1 ml of glycerine. Continue to heat until most of the alcohol has evaporated (this is indicated by the appearance of heavy frothing). To evaporate the rest of the alcohol, place the flask in an oven at 100 °C for one hour.

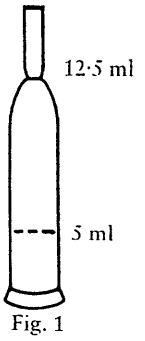


Fig. 1

Immediately after removal from the oven add, stirring well, 15 ml of saturated solution of potassium sulphate. Allow to cool to 20 °C, then add successively (stirring each time) 0·5 ml of dilute H_2SO_4, 1 ml of the coconut soap solution and about 0·1 g of purified kieselguhr.

Filter through a fluted filter 9 cm in diameter into the Beckel tube (Fig. 1) until the filtrate reaches the 12·5 ml level. It may be necessary to compress the residue onto the filter with a stirring rod to obtain sufficient filtrate. Transfer the filtrate into a 100 ml flask and rinse the Beckel tube with 5 ml of water. Pour the rinsings also into the bottle.

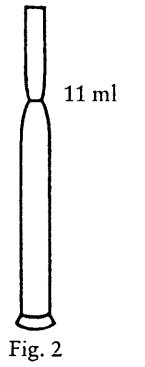


Fig. 2

Add fine pumice and distil 11 ml into the recipient shown (Fig. 2). Transfer to a beaker, add 1 to 2 drops of phenolphthalein solution, titrate with the aqueous sodium hydroxide solution until a persistent red colour is obtained, rinse the tube with this neutralised solution and repeat the titration until the red colour reappears.

The butyric index of the total fat content is calculated by means of the following formula:

$$\text{Butyric index} = \frac{n \times 1{\cdot}4 \times 500}{E} - 1{\cdot}2$$

where:

n is the number of ml of aqueous sodium hydroxide solution used in the titration

E is the number of mg of total fat used

The percentage by weight of milkfat in the sample is calculated by means of the following formula:

$$P = \frac{a \times b}{20}$$

where:

P is the percentage by weight of milkfat in the sample

a is the percentage by weight of total fat in the sample

b is the butyric index of the total fat determined as described above.

ANNEX IV

DETERMINATION OF THE MANNITOL CONTENT OF GOODS FALLING WITHIN SUB-HEADING No 29.04 C III OF THE COMMON CUSTOMS TARIFF, CALCULATED ON THE SORBITOL CONTENT

I. Principle

Gas chromatography is used to determine the mannitol content of goods falling within sub-heading No 29.04 C III of the Common Customs Tariff, calculated on their sorbitol content. For that purpose non-volatile products or their acetylated derivatives must first be processed.

II. **Apparatus and reagents**

1. Gas chromatograph with flame-ionisation detector
2. Column filled with '5% XE 60 on Aeropak 30' (Granulation 80–100 mesh)
3. Anhydrous pyridine for analysis
4. Acetone for analysis
5. Acetic anhydride for analysis
6. 25 ml calibrated flasks

III. Method

(a) *Preparation of the acetylated derivatives*

1. Solid products

Weigh about 200 mg of the product. Boil for one hour with 3 ml pyridine and 3 ml acetic anhydride so that the liquid flows back after condensation. Leave to cool and bring to 250 ml with acetone. Inject this solution in the chromatograph.

2. Products in aqueous solution

Weigh about 3 g of the product. Add 60 ml pyridine. Distil until the temperature of the vapour reaches 115°. Then add 30 ml acetic anhydride and boil for two hours so that the liquid flows back after condensation. Leave to cool and bring to 250 ml with acetone. Inject this solution in the chromatograph.

(b) *Conditions required to carry out chromatography*

Injection temperature: 300 °C

Temperature of the column: 210 °C

Flow of the carrier gas stream (for instance nitrogen): 25 ml/minute

Hydrogen flow: 25 ml/minute

Quantity injected: 1 microlitre

The mannitol peak appears first, then the sorbitol peak.

To determine the mannitol content of the goods under analysis, calculated on their sorbitol content, simply calculate the ratio of the area of the two peaks.