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(Acts whose publication is obligatory)

REGULATION (EEC) No 924/74 OF THE COMMISSION

of 10 April 1974

amending Regulation (EEC) No 1061/69 of 6 June 1969 specifying methods of analysis for the implementation of Regulation (EEC) No 1059/69

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, as last amended by the Act [2] annexed to the Treaty on the Accession of New Member States to the European Economic Community and the European Atomic Energy Community signed at Brussels on 22 January 1972, and in particular Article 3 thereof;

Whereas, so as to ensure a uniform treatment on importation into the Community of goods subject to Council Regulation (EEC) No 1059/69 [3], of 28 May 1969, laying down the trade arrangements applicable to certain goods resulting from the processing of agricultural products, as last amended by Council Regulation (EEC) No 1491/73 [4], Commission Regulation (EEC) No 1061/69 [5] of 6 June 1969 specified methods of analysis and other technical provisions required either to identify certain goods or to determine their composition;

Whereas the method of analysis provided for in the latter Regulation to determine milkfat content is proving to be insufficiently accurate in the case of goods containing not only milkfats but also certain other fats;

Whereas, for practical reasons, it is necessary not only to maintain the method of analysis mentioned above, but also to adopt a method which can give satisfaction in the above cases;

Whereas, in accordance with Article 4 (2) of Regulation (EEC) No 1059/69, the methods of analysis and other technical provisions concerned must be adopted in accordance with the procedure laid down in Article 3 (2) and (3) of Regulation (EEC) No 97/69;

Whereas the measures provided for in this Regulation are in accordance with the Opinion of the Committee on the Common Customs Tariff Nomenclature,

HAS ADOPTED THIS REGULATION:

Article 1

Regulation (EEC) No 1061/69 shall be amended as follows:

(a) Article 3 shall be amended as follows:
'The milkfat content by weight of a product shall be ascertained either by the method set out in Annex III, Chapter 1, or, if it appears necessary, by the method set out in Annex III, Chapter 2.'

(b) in Annex III, there shall be inserted after the title: 'Chapter 1'

(c) Annex III shall be completed by the Chapter 2 annexed to this Regulation.

Article 2

This Regulation shall enter into force on 1 May 1974.

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(1) OJ No L 14, 21. 1. 1969, p. 1.
(2) OJ No L 73, 27. 3. 1972, p. 14.
(3) OJ No L 141, 12. 6. 1969, p. 1.
(4) OJ No L 151, 7. 6. 1973, p. 1.
(5) OJ No L 141, 12. 6. 1969, p. 24.

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

Done at Brussels, 10 April 1974.

For the Commission

The President

François-Xavier ORTOLI

ANNEX

CHAPTER 2

DETERMINATION OF THE SEMI-MICRO VALUES

(Analytical method, Page 8 i — 1960, of the Office International du Cacao et du Chocolat)

I. Butyric semi-micro value (BsmV):

1. *Definition:*

The butyric value is the number of ml of 0·1 N potassium hydroxide required to neutralize the soluble and volatile fatty acids isolated by saponification from 5 g of fat, these acids being soluble in a solution of sulphuric acid saturated with potassium sulphate and caprylic acid.

2. *Reagents:*

(a) Alcoholic solution of potassium hydroxide:

Mix 40 ml of potassium hydroxide (d = 1·5; 49 % w/w KOH) and 40 ml distilled water and make up to 1 litre with 95 % vol. alcohol. The alcohol concentration of the completed reagent should not exceed 90 % vol. It will be checked by titrating with phenolphthalein as indicator: 5 ml of the reagent should require 25 to 27 ml of 0·1 N hydrochloric acid. If the potash is too weak, add the required amount of potassium hydroxide (d = 1·5).

(b) Glycerine $C_3H_5(OH)_3$

Pharmaceutically pure (d = 1·23; 88 % w/w).

(c) Sulphuric acid 25 % vol. H_2SO_4

1 vol. conc. sulphuric acid (d = 1·84) + 3 vol. distilled water.

(d) Solution of potassium sulphate K_2SO_4

Saturated aqueous solution of potassium sulphate at 20 °C (d = 1·08; 10 % w/w).

(e) Coco-Soap solution

Saponify 50 g of pure coconut fat (refined but non-hardened, m. p. 24-26 °C) with 50 g of glycerine, 15 g KOH and 20 ml water, on an open flame in a 1 litre flask. After cooling to below 100 °C, carefully dilute to 500 ml.

(f) Sodium hydroxide 0·01 N NaOH

The strength should be checked every day (indicator: phenolphthalein).

(g) Indicator

Add 1 g of phenolphthalein to a 100 ml graduated flask and make up to the mark with 95 % vol. ethyl alcohol.

3. *Method:*

(a) Saponification

Accurately weigh 500 to 520 mg of fat in a 50 ml flat-based flask. Execute a blank determination in the same way but using 500 mg of cocoa butter. The melted fat is introduced into the flask with a pipette (the given amount represents about 20 drops). Then add a few grains of pumice stone and 5 ml of alcoholic potassium hydroxide solution using a pipette. Connect the flask to a refrigerator and saponify on a boiling-water bath. When the solution is perfectly clear keep boiling for at least another five minutes. Then with a large orifice pipette (tip cut off) add 1 ml glycerine and continue to boil without refrigerator until the greater part of the alcohol has evaporated. This point is reached when the soap begins to lather strongly. Then lay the flask in a stove at 100 °C for an hour to eliminate the last traces of alcohol.

(b) Decomposition of the soaps

Immediately after removing the flask from the stove add 15 ml of saturated potassium sulphate solution with a pipette, close with a cork stopper and shake vigorously until the soap is homogeneous. Certain fats form fairly viscous soaps which are difficult to dissolve in the potassium sulphate solution. In this case it is recommended to put the flask back in the stove for a while and shake once again.

As soon as a homogeneous solution is formed allow to cool to room temperature then place the flask on a water bath at exactly 20 °C for 10 minutes. Then add in the following order, while shaking, 0·5 ml sulphuric acid, 1 ml of coco-soap solution and 0·1 mg purified infusorial earth. Replace the flask on the bath at 20 °C for about five minutes. Shake strongly and filter over a dry pleated filter paper 10 cm in diameter and collect the filtrate in a 12·5 ml Beckel tube till the mark is reached. If necessary, slightly press the contents of the filter with the round end of a test-tube to obtain the prescribed amount of filtrate.

(c) Distillation

Transfer the filtrate to a 100 ml flat-bottomed flask, rinse the Beckel tube with 5 ml distilled water (recently boiled and cooled) and add the latter to the filtrate. Add some powdered pumice stone to the flask; connect it to the distilling apparatus (Figure 1) and distil into an 11 ml Beckel tube (up to the mark).

As a source of heat use an oil bath at 175 °C whose level is about 1 cm higher than the contents of the flask. According to Grossfeld, the size of the distillator does not affect the result. It should however be kept at a minimum in order to lower as much as possible the amount of liquid on the walls of the refrigerating tube (sleeve length about 18 cm).

(d) Titration

Transfer the distillate to a 50 ml Erlenmeyer, add 1-2 drops of phenolphthalein and titrate with the 0·01 N sodium hydroxide until a distinctly pink coloration is obtained. Rinse the Beckel tube three times with the solution contained in the Erlenmeyer. In most cases the latter will lose its colour. Continue to titrate carefully until a faint pink coloration just appears, which should however persist for about 30 seconds.

(e) Calculation

The butyric semi-micro value is determined from the following formula:

$$\text{BsmV} = \frac{(a - b) \times 1{\cdot}4 \times 500}{E}$$

Commentaries:

a = ml of 0·01 N NaOH used for the titration

b = ml of 0·01 N NaOH used for the blank titration

E = mg of fat used for the determination.

FIGURE 1

Distilling Apparatus for the Determination of Butyric and Total (semi-micro) values

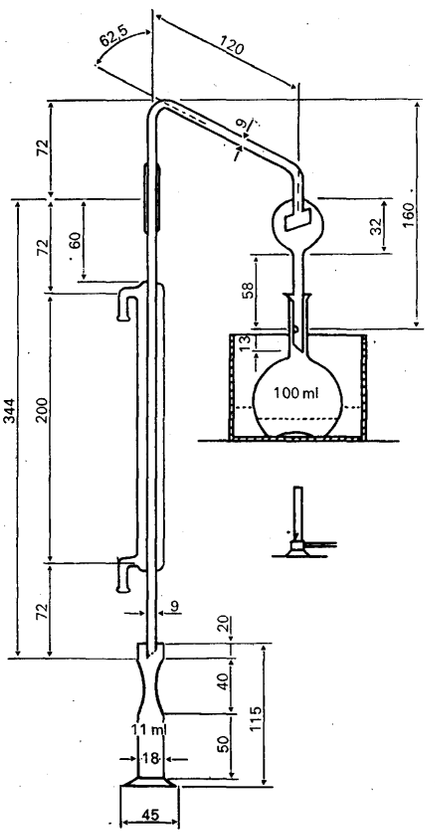
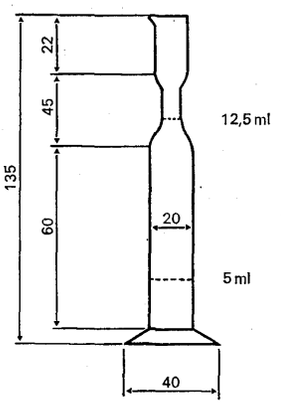


FIGURE 2

Beckel tube



II. **Total semi-micro value (TsmV):**

1. *Definition:*

The total value of the lower fatty acids indicates the number of ml of alkali required to neutralize all the fatty acids with a low molecular weight contained in 5 g of fat, the latter having been saponified and the lower fatty acids having been separated from the higher fatty acids with a solution of magnesium sulphate and in a very diluted medium.

2. *Reagents:*

(a) Alcoholic solution of potassium hydroxide:

Identical to that used for the butyric index. (Mix 40 ml of caustic potash — d = 1·5; 49 % w/w KOH — and 40 ml distilled water, then make up to 1 litre with 95 % vol. alcohol).

(b) Glycerine $C_3H_5(OH)_3$

Pharmaceutically pure (d = 1·23; 88 % w/w).

(c) Magnesium sulphate solution

15 g $MgSO_4 + 7\ H_2O$ per litre.

(d) Alcoholic phenolphthalein solution

90 % vol. alcohol containing 0·2 g phenolphthalein per litre.

(e) Sodium hydroxide

0·01 N NaOH

(f) Phosphoric acid H_3PO_4

(d = 1·146; 25 % w/w).

3. *Method:*

(a) Saponification

Accurately weigh 500-550 mg of fat for analysis in a 100 ml flat-bottomed flask. Execute a blank determination in the same way but using 2-3 drops of cocoa butter. (Add the liquid fat to the flask with a pipette). Add 5 ml alcoholic solution of potassium hydroxide and a few grains of pumice stone and connect the flask to a water condenser. Saponify on a boiling-water bath. When the solution is perfectly clear, maintain the boiling for at least five minutes. With a wide-orificed pipette (end cut off) add 1 ml of glycerine and boil — without the condenser — until the greater part of the alcohol has evaporated. This point is reached when the soap begins to lather strongly. Then place the flask in a stove at 100 °C for one hour to eliminate the last traces of alcohol.

(b) Decomposition of the soaps

Immediately on removal of the flask from the stove, add 50 ml of recently boiled distilled water with a pipette, while shaking constantly, to dissolve the soap. Finally add, while still shaking, 25 ml of magnesium sulphate solution, close the flask and allow to settle for about 13 hours (overnight) at room temperature.

Then filter over a 15 cm dry pleated filter paper collecting the filtrate in a 100 ml graduated cylinder. If the volume of the filtrate is less than 50 ml, slightly press the contents of the filter paper with the rounded end of a test-tube to obtain the required amount of filtrate.

(c) Distillation

Pipette exactly 50 ml of filtrate in a new 100 ml flat-bottomed flask (or an Erlenmeyer flask). Add a knife-tip (about 0·1 g) of pumice-stone powder and 1 ml of

phosphoric acid. With the semi-micro distillation apparatus (Figure 1) distil exactly 40 ml in an NP 20/4 50 ml graduated cylinder. As a source of heat use an oil bath at 175 °C the level of which is about 1 cm above the level of the flask contents.

(d) Titration

Transfer the distillate to a 100 ml Erlenmeyer flask. Rinse the condenser with 10 ml of alcoholic phenolphthalein solution which is collected in the graduated cylinder. After good draining of the condenser, pour the alcohol carefully into the Erlenmeyer containing the distillate. The added alcohol forms a layer on the surface of the liquid and dissolves the fatty acids insoluble in water which have formed drops at the surface. Shake carefully and titrate with 0·01 N of sodium hydroxide till the colour becomes distinctly pink. Towards the end of the titration add two drops at a time of sodium hydroxide. Rinse the graduated cylinder three times with the solution contained in the Erlenmeyer, which usually decolourises the solution. Continue to titrate carefully until a faint pink coloration appears which should however persist for at least 30 seconds.

(e) Calculation

The total semi-micro value is determined from the following formula:

$$TsmV = \frac{(a - b) \times 1{\cdot}52 \times 500}{E}$$

Commentaries:

a = ml of 0·01 N NaOH used for the titration

b = ml of 0·01 N NaOH used for the blank titration

E = mg of fat used for the determination.

III. **Residual semi-micro value (RsmV):**

1. *Definition*

The residual value is the difference between the total value for the lower fatty acids and the butyric value:

RsmV = TsmV — BsmV

As in the case of the total and butyric values, the residual value is proportional to the amount of certain fatty acids in the fat, in particular that of caprylic and caprinic acid. In this way, the residual value, together with the butyric value, is well suited for the determination of coconut fat in the presence of milk fat.

IV. **Calculation:**

By using the preceding semi-micro values we can calculate the milk fat and coconut fat content from the following two formulae:

% milk fat in the total fat = 5·09 × BsmV — 0·12 × RsmV

% coconut fat in the total fat = 2·76 × RsmV — 2·07 × BsmV

Note:

The addition of coconut fat can only be considered as definite if the ratio RsmV/BsmV is greater than or equal to 1·2.