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Legislation

Contents

I *Acts whose publication is obligatory*

.....

II *Acts whose publication is not obligatory*

Council

73/44/EEC:

Council Directive of 26 February 1973 on the approximation of the laws of the Member States relating to the quantitative analysis of ternary fibre mixtures..... 1

73/45/Euratom:

Council Decision of 8 March 1973 amending the statutes of the Euratom Supply Agency following the Accession of new Member States to the Community..... 20

Commission

73/46/EEC:

Fourth Commission Directive of 5 December 1972 establishing Community methods of analysis for the official control of feedingstuffs 21

73/47/EEC:

Commission Directive of 5 December 1972 amending the second Commission Directive of 18 November 1971 establishing Community methods of analysis for the official control of feedingstuffs 35

73/48/ECSC:

Commission Decision of 4 January 1973 extending the authority for the application of two special tariff measures to the rail carriage of Lorraine iron ore to Belgium and Luxembourg 36

73/49/ECSC:

Commission Decision of 4 January 1973 authorizing three special tariffs for the carriage of iron ore from Western France to Belgium, Luxembourg and the Saar... 38

(Contents continued)

73/50/EEC:

Commission Decision of 8 February 1973 applying the provisions of Article 3 (2) (a) of Regulation (EEC) No 142/69 in favour of two Italian sugar manufacturers 40

73/51/EEC:

Commission Decision of 8 February 1973 amending the Commission Decision of 22 December 1972 authorizing the Federal Republic of Germany to sell butter at reduced price in the form of concentrated butter 41

73/52/EEC:

Commission Decision of 21 February 1973 discontinuing the twenty-third partial invitation to tender for white sugar issued under Regulation (EEC) No 1897/72.... 42

73/53/EEC:

Commission Decision of 26 February 1973 concerning protective measures to be applied by the Member States, against swine vesicular disease 43

II

(Acts whose publication is not obligatory)

COUNCIL

COUNCIL DIRECTIVE

of 26 February 1973

on the approximation of the laws of the Member States relating to the quantitative analysis of ternary fibre mixtures

(73/44/EEC)

THE COUNCIL OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community, and in particular Article 100 thereof;

Having regard to the proposal from the Commission;

Whereas the Council Directive of 26 July 1971 ⁽¹⁾ on the approximation of the laws of the Member States relating to textile names lays down provisions on labelling based on the fibre composition of textile products;

Whereas the methods used for official tests in the Member States to determine the fibre composition of textile products should be uniform, as regards both the pre-treatment of the sample and the quantitative analysis of the product;

Whereas Article 13 of the abovementioned Council Directive provides that the sampling and analytical methods to be used in all Member States for the purpose of determining the fibre composition of products will be specified in separate Directives; whereas under these circumstances, the Council

adopted in its Directive of 17 July 1972 ⁽²⁾ on the approximation of the laws of the Member States relating to certain methods for the quantitative analysis of binary fibre mixtures, provisions concerning the preparation of test samples and test specimens which are applicable to ternary fibre mixtures;

Whereas the object of this Directive is to lay down provisions governing the quantitative analysis of ternary fibre mixtures;

Whereas the particular methods relating to the analysis of certain binary mixtures are described in detail in the Directive of 17 July 1972; whereas present experience does not permit the specification of a single standard procedure as yet; whereas several variants must be proposed for the selective dissolution of the components;

Whereas general rules valid for the analysis of all ternary mixtures must however be worked out; whereas the object of these rules is to specify the various methods which could suitably be applied and the method of calculating the percentage composition for each variant;

Whereas technical specifications must be adapted rapidly to keep pace with technical progress; whereas to this end the procedure laid down in Article 6 of the Directive of 17 July 1972 should be applied;

⁽¹⁾ OJ No L 185, 16. 8. 1971, p. 16.

⁽²⁾ OJ No L 173, 31. 7. 1972, p. 1.

HAS ADOPTED THIS DIRECTIVE:

Article 1

This Directive concerns the quantitative analysis of ternary fibre mixtures by the methods of manual separation, chemical separation or a combination of both.

Article 2

With regard to the preparation of test samples and test specimens, the provisions contained in Annex I to the Council Directive of 17 July 1972 on the approximation of the laws of the Member States relating to certain methods for the quantitative analysis of binary fibre mixtures shall be applicable.

Article 3

The Member States shall take all necessary steps to ensure that the provisions laid down — both in Annex I to this Directive and in Annex I to the Directive referred to in Article 2 — be applied in official tests, to determine the composition of textile products composed of a ternary fibre mixture put on the market in accordance with the Council Directive of 26 July 1971 on the approximation of the laws of the Member States relating to textile names.

Article 4

Any laboratory responsible for the testing of ternary mixtures shall show in the test report all the factors mentioned in point V of Annex I.

Article 5

Any amendments to the specifications in Annexes I, II and III that are necessary to conform with technical progress shall be adopted in accordance with the procedure laid down in Article 6 of the Directive of 17 July 1972.

Article 6

1. Member States shall put into force the provisions necessary to comply with this Directive within a period of eighteen months of its notification and shall immediately inform the Commission thereof.

2. Member States shall ensure that the text of the main provisions of internal law adopted by them in the field governed by this Directive are communicated to the Commission.

Article 7

This Directive is addressed to the Member States.

Done at Brussels, 26 February 1973.

For the Council

The President

E. GLINNE

ANNEX I

QUANTITATIVE ANALYSIS OF TERNARY FIBRE MIXTURES

GENERAL

Introduction

Methods for the quantitative analysis of fibre mixtures are based on two processes, the manual separation and the chemical separation of fibre types.

The method of manual separation should be used whenever possible since it generally gives more accurate results than the chemical method. It can be used for all textiles whose component fibres do not form an intimate mixture, as for example in the case of yarns composed of several elements each of which is made up of only one type of fibre or fabrics in which the warp is of a different type of fibre from the weft; or knitted fabrics capable of being unravelled made up of yarns of different types.

In general, the methods of quantitative chemical analysis are based on the selective solution of the individual components. There are four possible variants of this method:

1. Using two different test specimens, a component (a) is dissolved from the first test specimen, and another component (b) from the second test specimen. The insoluble residues of each specimen are weighed and the percentage of each of the two soluble components is calculated from the respective losses in mass. The percentage of the third component (c) is calculated by difference.
2. Using two different test specimens, a component (a) is dissolved from the first test specimen and two components (a and b) from the second test specimen. The insoluble residue of the first test specimen is weighed and the percentage of the component (a) is calculated from the loss in mass. The insoluble residue of the second test specimen is weighed; it corresponds to component (c). The percentage of the third component (b) is calculated by difference.
3. Using two different test specimens, two components (a and b) are dissolved from the first test specimen and two components (b and c) from the second test specimen. The insoluble residues correspond to the two components (c) and (a) respectively. The percentage of the third component (b) is calculated by difference.
4. Using only one test specimen, after removal of one of the components, the insoluble residue formed by the two other fibres is weighed and the percentage of the soluble component is calculated from the loss in mass. One of the two fibres of the residue is dissolved, the insoluble component is weighed and the percentage of the second soluble component is calculated from the loss in mass.

Where a choice is possible, it is advisable to use one of the first three variants.

Where chemical analysis is used, the expert responsible for the analysis must take care to select methods employing solvents which dissolve only the correct fibre(s), leaving the other fibre(s) intact.

By way of example, a table is given in Annex III which contains a certain number of ternary mixtures, together with methods for analysing binary mixtures which can, in principle, be used for analysing these ternary mixtures.

In order to reduce the possibility of error to a minimum, it is recommended that, whenever possible, chemical analysis using at least two of the four abovementioned variants should be made.

Mixtures of fibres used during processing and, to a lesser extent, in finished textiles may contain non-fibrous matter, such as fats, waxes or dressings, or water-soluble matter, either occurring naturally or added to facilitate processing. Non-fibrous matter must be removed before analysis. For this reason a method of pre-treatment for removing oils, fats, waxes and water-soluble matter is also given.

In addition, textiles may contain resins or other matter added to confer special properties. Such matter, including dyestuffs in exceptional cases, may interfere with the action of the reagent on the soluble components and/or it may be partially or completely removed by the reagents. This type of added matter may thus cause errors and should be removed before the sample is analysed. If it is impossible to remove such added matter the methods for quantitative chemical analysis given in Annex III attached are no longer applicable.

Dye in dyed fibres is considered to be an integral part of the fibre and is not removed.

Analyses are conducted on the basis of dry mass and a procedure is given for determining dry mass.

The result is obtained by applying to the dry mass of each fibre the recovery rates listed in Annex II to the Directive on the approximation of the laws of the Member States relating to textile names.

Before proceeding with any analysis, all the fibres present in the mixture must be identified. In some chemical methods, the insoluble component of a mixture may be partially dissolved in the reagent used to dissolve the soluble component(s). Wherever possible, reagents have been chosen that have little or no effect on the insoluble fibres. If a loss in mass is known to occur during the analysis, the result should be corrected; correction factors are given for this purpose. These factors have been determined in several laboratories by treating, with the appropriate reagent as specified in the method of analysis, fibres cleaned by the pre-treatment. These correction factors apply only to undergraded fibres and different correction factors may be necessary if the fibres have been degraded before or during processing. If the fourth variant, in which a textile fibre is subjected to the successive action of two different solvents, must be used, correction factors must be applied for possible losses in mass undergone by the fibre in the two treatments. At least two determinations should be made, both in the case of manual separation and in the case of chemical separation.

I. GENERAL INFORMATION ON METHODS FOR THE QUANTITATIVE CHEMICAL ANALYSIS OF TERNARY FIBRE MIXTURES

Information common to the methods given for the quantitative chemical analysis of ternary fibre mixtures.

I.1. *Scope and field of application*

The field of application of each method for analysing binary fibre mixtures specifies to which fibres the method is applicable. (See Annex II, Directive relating to certain methods for the quantitative analysis of binary fibre mixtures).

I.2. *Principle*

After the identification of the components of a mixture, the non-fibrous material is removed by suitable pre-treatment and then one or more of the four variants of the process of selective solution described in the introduction is applied. Except where this presents technical difficulties, it is preferable to dissolve the major fibre component so as to obtain the minor fibre component as final residue.

I.3. *Apparatus and Reagents*

I.3.1. *Apparatus*

- I.3.1.1. Filter crucibles and weighing bottles large enough to contain such crucibles, or any other apparatus giving identical results.
- I.3.1.2. Vacuum flask.
- I.3.1.3. Desiccator containing self-indicating silica gel.
- I.3.1.4. Ventilated oven for drying specimens at 105 ± 3 °C.
- I.3.1.5. Analytical balance, accurate to 0.0002 g.
- I.3.1.6. Soxhlet extractor or other apparatus giving identical results.

I.3.2. *Reagents*

- I.3.2.1. Light petroleum, redistilled, boiling range 40 to 60 °C.
- I.3.2.2. Other reagents are specified in the appropriate parts of the text of the method. All reagents used should be chemically pure.
- I.3.2.3. Distilled or deionized water.

I.4. *Conditioning and testing atmosphere*

Because dry masses are determined, it is unnecessary to condition the specimens or to conduct analyses in a conditioned atmosphere.

I.5. *Laboratory test sample*

Take a laboratory test sample that is representative of the laboratory bulk sample and sufficient to provide all the specimens, each of at least 1 g, that are required.

I.6. *Pre-treatment of the laboratory test sample*

Where a substance not to be taken into account in the percentage calculations (See Article 12 (2) (d) of the Directive relating to textile names) is present, it should first be removed by a suitable method that does not affect any of the component fibres.

For this purpose, non-fibrous matter which can be extracted with light petroleum and water is removed by treating the air-dry test sample in a Soxhlet extract selector with light petroleum for one hour at a minimum rate of 6 cycles per hour. Allow the light petroleum to evaporate from the sample which is then directly extracted with water by soaking for one hour at room temperature followed by soaking at 65 ± 5 °C for a further one hour, agitating the liquid from time to time, specimen/water ratio 1 : 100. Remove the excess water from the sample by squeezing, suction, or centrifuging and then allow the sample to become air-dry.

Where non-fibrous matter cannot be extracted with light petroleum and water, it should be removed by substituting for the water method described above a suitable method that does not substantially alter any of the fibre constituents. However for some unbleached natural vegetable fibres (eg jute, coir) it is to be noted that normal pre-treatment with light petroleum and water does not remove all the natural non-

fibrous substances; nevertheless additional pre-treatment is not applied unless the sample contains finishes insoluble in both light petroleum and water.

Analytical reports should include full details of the methods of pre-treatment used.

I.7. *Test procedure*

I.7.1. *General instructions*

I.7.1.1. *Drying*

Conduct all drying operations for not less than 4 hours and not more than 16 hours at $105 \pm 3^\circ\text{C}$ in a ventilated oven with the oven door closed throughout. If the drying period is less than 14 hours, the specimen must be checkweighed to determine whether its mass is constant. The mass may be considered as constant if, after a further drying period of 60 minutes, its variation is less than 0.05 %.

Avoid handling crucibles and weighing bottles, specimens or residues with bare hands during the drying, cooling and weighing operations.

Dry specimens in a weighing bottle with its cover beside it. After drying, stopper the weighing bottle before removing it from the oven, and transfer it quickly to the desiccator.

Dry the filter crucible in a weighing bottle with its cover beside it in the oven. After drying, close the weighing bottle and transfer it quickly to the desiccator.

Where apparatus other than a filter crucible is used, drying operations should be conducted in the oven so as to determine the dry mass of the fibres without loss.

I.7.1.2. *Cooling*

Conduct all cooling operations in the desiccator, placed beside the balance, until the cooling of the weighing bottles is complete, and in any case for not less than 2 hours.

I.7.1.3. *Weighing*

After cooling, complete the weighing of the weighing bottle within 2 minutes of its removal from the desiccator; weigh to an accuracy of 0.0002 g.

I.7.2. *Procedure*

Take from the pre-treated laboratory test sample a test specimen of at least 1 g (in mass). Cut yarn or cloth into lengths of about 10 mm, dissected as much as possible. Dry the specimen(s) in (a) weighing bottle(s) cool it (them) in the desiccator and weigh it (them). Transfer the specimen(s) to the glass vessel(s) specified in the appropriate section of the Community method, reweigh the weighing bottle(s) immediately and obtain the dry mass(es) of the specimen(s) by difference; complete the test as specified in the appropriate section of the applicable method. Examine the residue(s) microscopically to check that the treatment has in fact completely removed the soluble fibre(s).

I.8. *Calculation and expression of results*

Express the mass of each component as a percentage of the total mass of fibre in the mixture. Calculate the results on the basis of clean dry mass, adjusted by (a) the conventional recovery rates and (b) the correction factors necessary to take account of loss of non-fibrous matter during pre-treatment and analysis.

I.8.1. Calculation of percentages of mass of clean dry fibres disregarding loss of fibre mass during pre-treatment.

I.8.1.1. — VARIANT 1 —

Formulae to be applied where a component of the mixture is removed from one specimen and another component from a second specimen:

$$P_1\% = \left[\frac{d_2}{d_1} - d_2 \times \frac{r_1}{m_1} + \frac{r_2}{m_2} \times \left(1 - \frac{d_2}{d_1} \right) \right] \times 100$$

$$P_2\% = \left[\frac{d_4}{d_3} - d_4 \times \frac{r_2}{m_2} + \frac{r_1}{m_1} \times \left(1 - \frac{d_4}{d_3} \right) \right] \times 100$$

$$P_3\% = 100 - (P_1\% + P_2\%);$$

$P_1\%$ is the percentage of the first clean dry component (component in the first specimen dissolved in the first reagent);

$P_2\%$ is the percentage of the second clean dry component (component in the second specimen dissolved in the second reagent);

$P_3\%$ is the percentage of the third clean dry component (component undissolved in both specimens);

m_1 is the dry mass of the first specimen after pre-treatment;

m_2 is the dry mass of the second specimen after pre-treatment;

r_1 is the dry mass of the residue after removal of the first component from the first specimen in the first reagent;

r_2 is the dry mass of the residue after removal of the second component from the second specimen in the second reagent;

d_1 is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen ⁽¹⁾;

d_2 is the correction factor for loss in mass in the first reagent of the third component undissolved in the first specimen ⁽¹⁾;

d_3 is the correction factor for loss in mass in the second reagent, of the first component undissolved in the second specimen ⁽¹⁾;

d_4 is the correction factor for loss in mass in the second reagent, of the third component undissolved in the second specimen ⁽¹⁾.

I.8.1.2. — VARIANT 2 —

Formulae to be applied where a component (a) is removed from the first test specimen, leaving as residue the other two components (b + c), and two components (a + b) are removed from the second test specimen, leaving as residue the third component (c):

$$P_1\% = 100 - (P_2\% + P_3\%)$$

$$P_2\% = 100 \times \frac{d_1 r_1}{m_1} - \frac{d_1}{d_2} \times P_3\%$$

$$P_3\% = \frac{d_4 r_2}{m_2} \times 100$$

$P_1\%$ is the percentage of the first clean dry component (component of the first specimen soluble in the first reagent);

⁽¹⁾ The values of d are indicated in the corresponding sections of the Directives relating to the various methods of analysing binary mixtures.

$P_2\%$ is the percentage of the second clean dry component (component soluble, at the same time as the first component of the second specimen, in the second reagent);

$P_3\%$ is the percentage of the third clean dry component (component insoluble in both specimens);

m_1 is the dry mass of the first specimen after pre-treatment;

m_2 is the dry mass of the second specimen after pre-treatment;

r_1 is the dry mass of the residue after removal of the first component from the first specimen in the first reagent;

r_2 is the dry mass of the residue after removal of the first and second components from the second specimen in the second reagent;

d_1 is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen ⁽¹⁾;

d_2 is the correction factor for loss in mass in the first reagent, of the third component undissolved in the first specimen ⁽¹⁾;

d_4 is the correction factor for loss in mass in the second reagent, of the third component undissolved in the second specimen ⁽¹⁾.

I.8.1.3. — VARIANT 3 —

Formulae to be applied where two components (a + b) are removed from a specimen, leaving as residue the third component (c), then two components (b + c) are removed from another specimen, leaving as residue the first component (a):

$$P_1\% = \frac{d_3 r_2}{m_2} \times 100$$

$$P_2\% = 100 - (P_1\% + P_3\%)$$

$$P_3\% = \frac{d_2 r_1}{m_1} \times 100$$

$P_1\%$ is the percentage of the first clean dry component (component dissolved by the reagent);

$P_2\%$ is the percentage of the second clean dry component (component dissolved by the reagent);

$P_3\%$ is the percentage of the third clean dry component (component dissolved in the second specimen by the reagent);

m_1 is the dry mass of the first specimen after pre-treatment;

m_2 is the dry mass of the second specimen after pre-treatment;

r_1 is the dry mass of the residue after removal of the first and second components from the first specimen with the first reagent;

r_2 is the dry mass of the residue after removal of the second and third components from the second specimen with the second reagent;

d_2 is the correction factor for loss in mass with the first reagent of the third component undissolved in the first specimen ⁽¹⁾;

⁽¹⁾ The values of d are indicated in the corresponding sections of the Directives relating to the various methods of analysing binary mixtures.

d_3 is the correction factor for loss in mass with the second reagent of the first component undissolved in the second specimen ⁽¹⁾.

I.8.1.4. — VARIANT 4 —

Formulae to be applied where two components are successively removed from the mixture using the same specimen:

$$P_1\% = 100 - (P_2\% + P_3\%)$$

$$P_2\% = \frac{d_1 r_1}{m} \times 100 - \frac{d_1}{d_2} \times P_3\%$$

$$P_3\% = \frac{d_3 r_2}{m} \times 100$$

$P_1\%$ is the percentage of the first clean dry component (first soluble component);

$P_2\%$ is the percentage of the second clean dry component (second soluble component);

$P_3\%$ is the percentage of the third clean dry component (insoluble component);

m is the dry mass of the specimen after pre-treatment;

r_1 is the dry mass of the residue after elimination of the first component by the first reagent;

r_2 is the dry mass of the residue after elimination of the first and second components by the first and second reagents;

d_1 is the correction factor for loss in mass of the second component in the first reagent ⁽¹⁾;

d_2 is the correction factor for loss in mass of the third component in the first reagent ⁽¹⁾;

d_3 is the correction factor for loss in mass of the third component in the first and second reagents ⁽²⁾.

I.8.2. Calculation of the percentage of each component with adjustment by conventional recovery rates and, where appropriate, correction factors for losses in mass during pre-treatment operations:

Given:

$$A = 1 + \frac{a_1 + b_1}{100} \quad B = 1 + \frac{a_2 + b_2}{100} \quad C = 1 + \frac{a_3 + b_3}{100}$$

then:

$$P_{1A}\% = \frac{P_1 A}{P_1 A + P_2 B + P_3 C} \times 100$$

$$P_{2A}\% = \frac{P_2 B}{P_1 A + P_2 B + P_3 C} \times 100$$

$$P_{3A}\% = \frac{P_3 C}{P_1 A + P_2 B + P_3 C} \times 100$$

$P_{1A}\%$ is the percentage of the first clean dry component, including moisture content and loss in mass during pre-treatment;

⁽¹⁾ The values of d are indicated in the corresponding sections of the Directives relating to the various methods of analysing binary mixtures.

⁽²⁾ Wherever possible d_3 should be determined in advance by experimental methods.

$P_{2A}\%$ is the percentage of the second clean dry component, including moisture content and loss in mass during pre-treatment;

$P_{3A}\%$ is the percentage of the third clean dry component, including moisture content and loss in mass during pre-treatment;

P_1 is the percentage of the first clean dry component obtained by one of the formulae given in I.8.1;

P_2 is the percentage of the second clean dry component obtained by one of the formulae given in I.8.1;

P_3 is the percentage of the third clean dry component obtained by one of the formulae given in I.8.1;

a_1 is the conventional recovery rate of the first component;

a_2 is the conventional recovery rate of the second component;

a_3 is the conventional recovery rate of the third component;

b_1 is the percentage of loss in mass of the first component during pre-treatment;

b_2 is the percentage of loss in mass of the second component during pre-treatment;

b_3 is the percentage of loss in mass of the third component during pre-treatment.

Where a special pre-treatment is used the values b_1 , b_2 and b_3 should be determined, if possible, by submitting each of the pure fibre constituents to the pre-treatment applied in the analysis. Pure fibres are those free from all non-fibrous material except those which they normally contain (either naturally or because of the manufacturing process), in the state (unbleached, bleached) in which they are found in the material to be analysed.

Where no clean separate constituent fibres used in the manufacture of the material to be analysed are available, average values of b_1 , b_2 and b_3 as obtained from tests performed on clean fibres similar to those in the mixture under examination, must be used.

If normal pre-treatment by extraction with light petroleum and water is applied, correction factors b_1 , b_2 and b_3 may generally be ignored, except in the case of unbleached cotton, unbleached flax and unbleached hemp where the loss due to pre-treatment is usually accepted as 4 % and in the case of polypropylene as 1 %.

In the case of other fibres, losses due to pre-treatment are usually disregarded in calculations.

I.8.3. *Note*

Calculation examples are given in Annex II to this Directive.

II. *METHOD OF QUANTITATIVE ANALYSIS BY MANUAL SEPARATION OF TERNARY FIBRE MIXTURES*

II.1. *Scope*

This method is applicable to textile fibres of all types provided they do not form an intimate mixture and that it is possible to separate them by hand.

II.2. *Principle*

After identification of the textile components, the non-fibrous matter is removed by a suitable pre-treatment and then the fibres are separated by hand, dried and weighed in order to calculate the proportion of each fibre in the mixture.

II.3. *Apparatus*

- II.3.1. Weighing bottles or other apparatus giving identical results.
- II.3.2. Desiccator containing self-indicating silica gel.
- II.3.3. Ventilated oven for drying specimens at 105 ± 3 °C.
- II.3.4. Analytical balance accurate to 0.0002 g.
- II.3.5. Soxhlet extractor, or other apparatus giving identical results.
- II.3.6. Needle.
- II.3.7. Twist tester or similar apparatus.

II.4. *Reagents*

- II.4.1. Light petroleum, redistilled, boiling range 40°—60 °C.
- II.4.2. Distilled or deionized water.

II.5. *Conditioning and testing atmosphere*

See I.4.

II.6. *Laboratory test sample*

See I.5.

II.7. *Pre-treatment of laboratory test samples*

See I.6.

II.8. *Procedure*

II.8.1. *Analysis of yarn*

Take from the pre-treated laboratory test sample a specimen of mass not less than 1 g. For a very fine yarn, the analysis may be made on a minimum length of 30 m, whatever its mass.

Cut the yarn into pieces of a suitable length and separate the fibre types by means of a needle and, if necessary, a twist tester. The fibre types so obtained are placed in pre-weighed weighing bottles and dried at 105 ± 3 °C to constant mass, as described in I.7.1 and I.7.2.

II.8.2. *Analysis of cloth*

Take from the pre-treated laboratory test sample a specimen of mass not less than 1 g, not including a selvage with edges carefully trimmed to avoid fraying and running parallel with weft or warp yarns, or in the case of knitted fabrics in the line of the wales and courses. Separate the different types of fibres and collect them in pre-weighed weighing bottles and proceed as described in II.8.1.

II.9. *Calculation and expression of results*

Express the mass of each component fibre as a percentage of the total mass of the fibres in the mixture. Calculate the results on the basis of clean dry mass, adjusted by (a) the conventional recovery rates and (b) the correction factors necessary to take account of losses in mass during pre-treatment operations.

- II.9.1. Calculation of percentage masses of clean dry fibre, disregarding loss in fibre mass during pre-treatment:

$$P_1\% = \frac{100 m_1}{m_1 + m_2 + m_3} = \frac{100}{1 + \frac{m_2 + m_3}{m_1}}$$

$$P_2\% = \frac{100 m_2}{m_1 + m_2 + m_3} = \frac{100}{1 + \frac{m_1 + m_3}{m_2}}$$

$$P_3\% = 100 - (P_1\% + P_2\%);$$

$P_1\%$ is the percentage of the first clean dry component;

$P_2\%$ is the percentage of the second clean dry component;

$P_3\%$ is the percentage of the third clean dry component;

m_1 is the clean dry mass of the first component;

m_2 is the clean dry mass of the second component;

m_3 is the clean dry mass of the third component.

- II.9.2. For calculation of the percentage of each component with adjustment by conventional recovery rates and, where appropriate, by correction factors for losses in mass during pre-treatment: see I.8.2.

III. *METHOD OF QUANTITATIVE ANALYSIS OF TERNARY FIBRE MIXTURES BY A COMBINATION OF MANUAL SEPARATION AND CHEMICAL SEPARATION*

Wherever possible, manual separation should be used, taking account of the proportions of components separated before proceeding to any chemical treatment of each of the separate components.

IV. *PRECISION OF THE METHODS*

The precision indicated in each method of analysis of binary mixtures relates to the reproducibility (see Annex II of the Directive relating to certain methods for the quantitative analysis of binary textile fibre mixtures).

Reproducibility refers to the reliability, ie the closeness of agreement between experimental values obtained by operators in different laboratories or at different times using the same method and obtaining individual results on specimens of an identical homogeneous mixture.

Reproducibility is expressed by confidence limits of the results for a confidence level of 95 %.

By this is meant that the difference between two results in a series of analyses made in different laboratories would, given a normal and correct application of the method to an identical and homogeneous mixture, be exceeded only in 5 cases out of 100.

To determine the precision of the analysis of a ternary mixture the values indicated in the methods for the analysis of binary mixtures which have been used to analyse the ternary mixture are applied in the usual way.

Given that in the four variants of the quantitative chemical analysis of ternary mixtures, provision is made for two dissolutions (using two separate specimens for the first three variants and a single specimen for the fourth variant) and, assuming that E_1 and E_2

denote the precision of the two methods for analysing binary mixtures, the precision of the results for each component is shown in the following table:

Component fibre	Variants	1	2 and 3	4
a		E_1	E_1	E_1
b		E_2	$E_1 + E_2$	$E_1 + E_2$
c		$E_1 + E_2$	E_2	$E_1 + E_2$

If the fourth variant is used, the degree of precision may be found to be lower than that calculated by the method indicated above, owing to possible action of the first reagent on the residue consisting of components b and c, which would be difficult to evaluate.

V. TEST REPORT

- V.1. Indicate the variant(s) used to carry out the analysis, the methods, reagents and correction factors.
- V.2. Give details of any special pre-treatments (See I.6).
- V.3. Give the individual results and the arithmetic mean, each to the 1st decimal place.
- V.4. Wherever possible, state the precision of the method for each component, calculated according to the table in section IV.

ANNEX II

EXAMPLES OF THE CALCULATION OF PERCENTAGES OF THE COMPONENTS OF CERTAIN TERNARY MIXTURES USING SOME OF THE VARIANTS DESCRIBED IN POINT I.8.1 OF ANNEX I

Consider the case of a fibre mixture which when qualitatively analysed gave the following components: 1. carded wool; 2. nylon (polyamide); 3. unbleached cotton.

VARIANT No 1

Using this variant, that is using two different specimens and removing one component (a = wool) by dissolution from the first specimen and a second component (b = polyamide) from the second specimen the following results can be obtained:

1. Dry mass of the first specimen after pre-treatment (m_1) = 1.6000 g
2. Dry mass of the residue after pre-treatment with alkaline sodium hypochlorite (polyamide + cotton) (r_1) = 1.4166 g
3. Dry mass of the second specimen after pre-treatment (m_2) = 1.8000 g
4. Dry mass of the residue after treatment with formic acid (wool + cotton) (r_2) = 0.9000 g

Treatment with alkaline sodium hypochlorite does not entail any loss in mass of polyamide, while unbleached cotton loses 3 %, therefore $d_1 = 1.0$ and $d_2 = 1.03$.

Treatment with formic acid does not entail any loss in mass of wool or unbleached cotton, therefore d_3 and $d_4 = 1.0$.

If the values obtained by chemical analysis and the correction factors are substituted in the formula under point I.8.1.1 of Annex I, the following result is obtained:

$$P_1\% \text{ (wool)} = \left[\frac{1.03}{1.0} - 1.03 \times \frac{1.4166}{1.6000} + \frac{0.9000}{1.8000} \times \left(1 - \frac{1.03}{1.0} \right) \right] \times 100 = 10.30$$

$$P_2\% \text{ (polyamide)} = \left[\frac{1.0}{1.0} - 1.0 \times \frac{0.9000}{1.8000} + \frac{1.4166}{1.6000} \times \left(1 - \frac{1.0}{1.0} \right) \right] \times 100 = 50.00$$

$$P_3\% \text{ (cotton)} = 100 - (10.30 + 50.00) = 39.70$$

The percentages of the various clean dry fibres in the mixture are as follows:

Wool	10.30 %
Polyamide	50.00 %
Cotton	39.70 %

These percentages must be corrected according to the formulae under point I.8.2 of Annex I in order to take account also of the conventional recovery rates and of the correction factors for any losses in mass after pre-treatment.

As indicated in Annex II to the Directive relating to textile names, the conventional recovery rates are as follows: carded wool 17.0 %, polyamide 6.25 %, cotton 8.5 %, also, unbleached cotton shows a loss in mass of 4 % after pre-treatment with light petroleum and water. Thus:

$$P_{1A} \% (\text{wool}) = \frac{10.30 \times \left(1 + \frac{17.0 + 0.0}{100}\right)}{10.30 \times \left(1 + \frac{17.0 + 0.0}{100}\right) + 50.00 \times \left(1 + \frac{6.25 + 0.0}{100}\right) + 39.70 \times \left(1 + \frac{8.5 + 4.0}{100}\right)} \times 100 = 10.97$$

$$P_{2A} \% (\text{polyamide}) = \frac{50.00 \times \left(1 + \frac{6.25 + 0.0}{100}\right)}{109.8385} \times 100 = 48.37$$

$$P_{3A} \% (\text{cotton}) = 100 - (10.97 + 48.37) = 40.66.$$

The composition of the yarn is therefore as follows:

Polyamide	48.4 %
Cotton	40.6 %
Wool	11.0 %
	<u>100.0 %</u>

VARIANT 4

Consider the case of a fibre mixture which when qualitatively analysed gave the following components: carded wool, viscose, unbleached cotton.

Suppose that using variant 4, that is successively removing two components from the mixture of one single specimen, the following results are obtained:

1. Dry mass of the specimen after pre-treatment (m_1) = 1.6000 g
2. Dry mass of the residue after the first treatment with alkaline sodium hypochlorite (viscose + cotton): (r_1) = 1.4166 g
3. Dry mass of the residue after the second treatment of the residue r_1 with zinc chloride/formic acid (cotton): (r_2) = 0.6630 g

Treatment with alkaline sodium hypochlorite does not entail any loss of mass in viscose, while unbleached cotton loses 3 %, therefore $d_1 = 1.0$ and $d_2 = 1.03$.

As a result of treatment with zinc chloride/formic acid, the mass of cotton increases by 4 %, so that $d_3 = (1.03 \times 0.96) = 0.9888$ rounded to 0.99, (d_3 being the correction factor for the respective loss or increase in mass of the third component in the first and second reagents).

If the values obtained by chemical analysis and the correction factors are substituted in the formulae given in point I.8.1.4 of Annex I the following result is obtained:

$$P_2 \% (\text{viscose}) = \frac{1.0 \times 1.4166}{1.6000} \times 100 - \frac{1.0}{1.03} \times 40.98 = 48.75 \%$$

$$P_3 \% (\text{cotton}) = \frac{0.99 \times 0.6630}{1.6000} \times 100 = 41.02 \%$$

$$P_1 \% (\text{wool}) = 100 - (48.75 + 41.02) = 10.23 \%$$

As has already been indicated for variant 1, these percentages must be corrected by the formulae indicated in point I.8.2 of Annex I.

$$P_{1A} \% (\text{wool}) = \frac{10.23 \times \left(1 + \frac{17.00 + 0.0}{100}\right)}{10.23 \times \left(1 + \frac{17.00 + 0.0}{100}\right) + 48.75 \times \left(1 + \frac{13 + 0.0}{100}\right) + 41.02 \times \left(1 + \frac{8.5 + 4.0}{100}\right)} \times 100 = 10.57\%$$

$$P_{2A} \% (\text{viscose}) = \frac{48.75 \times \left(1 + \frac{13 + 0.0}{100}\right)}{113.2041} \times 100 = 48.65 \%$$

$$P_{3A} \% (\text{cotton}) = 100 - (10.57 + 48.65) = 40.78 \%$$

The composition of the mixture is therefore:

Viscose	48.6 %
Cotton	40.8 %
Wool	10.6 %
	<u>100.0 %</u>

ANNEX III
TABLE OF TYPICAL TERNARY MIXTURES WHICH MAY BE ANALYSED USING
COMMUNITY METHODS OF ANALYSIS OF BINARY MIXTURES
(for illustration purposes)

Mixture No	Component fibres			Variant (1)	Number of Community binary method and reagent
	1st component	2nd component	3rd component		
1	wool or hair	viscose, cupro or certain types of modal	cotton	1 and/or 4	2 (alkaline sodium hypochlorite) and 3 (zinc chloride/formic acid)
2	wool or hair	polyamide 6 or 6-6	cotton, viscose, cupro or modal	1 and/or 4	2 (alkaline sodium hypochlorite) and 4 (formic acid 80 % w/w)
3	wool, hair or silk	certain chlorofibres	viscose, cupro, modal or cotton	1 and/or 4	2 (alkaline sodium hypochlorite) and 9 (carbon disulphide/acetone 55.5/44.5 w/w)
4	wool or hair	polyamide 6 or 6-6	polyester, polypropylene, acrylic or glass fibre	1 and/or 4	2 (alkaline sodium hypochlorite) and 4 (formic acid 80 % w/w)
5	wool, hair or silk	certain chlorofibres	polyester, acrylic, polyamide or glass fibre	1 and/or 4	2 (alkaline sodium hypochlorite) and 9 (carbon disulphide/acetone 55.5/44.5 w/w)
6	silk	wool or hair	polyester	2	11 (sulphuric acid 75 % w/w) and 2 (alkaline sodium hypochlorite)
7	polyamide 6 or 6-6	acrylic	cotton, viscose, cupro or modal	1 and/or 4	4 (formic acid 80 % w/w) and 8 (dimethylformamide)
8	certain chlorofibres	polyamide 6 or 6-6	cotton, viscose, cupro or modal	1 and/or 4	8 (dimethylformamide) and 4 (formic acid 80 % w/w) or 9 (carbon disulphide/acetone 55.5/44.5 w/w) and 4 (formic acid 80 % w/w)
9	acrylic	polyamide 6 or 6-6	polyester	1 and/or 4	8 (dimethylformamide) and 4 (formic acid 80 % w/w)
10	acetate	polyamide 6 or 6-6	viscose, cotton, cupro or modal	4	1 (acetone) and 4 (formic acid 80 % w/w)
11	certain chlorofibres	acrylic	polyamide	2 and/or 4	9 (carbon disulphide/acetone 55.5/44.5 w/w) and 8 (dimethylformamide)

Mixture No	Component fibres			Variant (1)	Number of Community binary method and reagent
	1st component	2nd component	3rd component		
12	certain chlorofibres	polyamide 6 or 6-6	acrylic	1 and/or 4	9 (carbon disulphide/acetone 55.5/44.5 w/w) and 4 (formic acid 80 % w/w)
13	polyamide 6 or 6-6	viscose, cupro, modal or cotton	polyester	4	4 (formic acid 80 % w/w) and 7 (sulphuric acid 75 % w/w)
14	acetate	viscose, cupro, modal or cotton	polyester	4	1 (acetone) and 7 (sulphuric acid 75 % w/w)
15	acrylic	viscose, cupro, modal or cotton	polyester	4	8 (dimethylformamide) and 7 (sulphuric acid 75 % w/w)
16	acetate	wool, hair or silk	cotton, viscose, cupro, modal, polyamide, polyester, acrylic	4	1 (acetone) and 2 (alkaline sodium hypochlorite)
17	triacetate	wool, hair or silk	cotton, viscose, cupro, modal, polyamide, polyester, acrylic	4	6 (dichloromethane) and 2 (alkaline sodium hypochlorite)
18	acrylic	wool, hair or silk	polyester	1 and/or 4	8 (dimethylformamide) and 2 (alkaline sodium hypochlorite)
19	acrylic	silk	wool or hair	4	8 (dimethylformamide) and 1 (sulphuric acid 75 % w/w)
20	acrylic	wool, hair or silk	cotton, viscose, cupro or modal	1 and/or 4	8 (dimethylformamide) and 2 (alkaline sodium hypochlorite)
21	wool, hair or silk	cotton, viscose, modal, cupro	polyester	4	2 (alkaline sodium hypochlorite) and 7 (sulphuric acid 75 % w/w)
22	viscose, cupro or certain types of modal	cotton	polyester	2 and/or 4	3 (zinc chloride/formic acid) and 7 (sulphuric acid 75 % w/w)
23	acrylic	viscose, cupro or certain types of modal	cotton	4	8 (dimethylformamide) and 3 (zinc chloride/formic acid)
24	certain chlorofibres	viscose, cupro or certain types of modal	cotton	1 and/or 4	9 (carbon disulphide/acetone 55.5/44.5 w/w) and 3 (zinc chloride/formic acid) or 8 (dimethylformamide) and 3 (zinc chloride/formic acid)

Mixture No	Component fibres			Variant (1)	Number of Community binary method and reagent
	1st component	2nd component	3rd component		
25	acetone	viscose, cupro or certain types of modal	cotton	4	1 (acetone) and 3 (zinc chloride/formic acid)
26	triacetate	viscose, cupro or certain types of modal	cotton	4	6 (dichloromethane) and 3 (zinc chloride/formic acid)
27	acetate	silk	wool or hair	4	1 (acetone) and 11 (sulphuric acid 75 % w/w)
28	triacetate	silk	wool or hair	4	6 (dichloromethane) and 11 (sulphuric acid 75 % w/w)
29	acetate	acrylic	cotton, viscose, cupro or modal	4	1 (acetone) and 8 (dimethylformamide)
30	triacetate	acrylic	cotton, viscose, cupro or modal	4	6 (dichloromethane) and 8 (dimethylformamide)
31	triacetate	polyamide 6 or 6-6	cotton, viscose, cupro or modal	4	6 (dichloromethane) and 4 (formic acid 80 % w/w)
32	triacetate	cotton, viscose, cupro or modal	polyester	4	6 (dichloromethane) and 7 (sulphuric acid 75 % w/w)
33	acetate	polyamide 6 or 6-6	polyester or acrylic	4	1 (acetone) and 4 (formic acid 80 % w/w)
34	acetate	acrylic	polyester	4	1 (acetone) and 8 (dimethylformamide)
35	certain chlorofibres	cotton, viscose, cupro or modal	polyester	4	8 (dimethylformamide) and 7 (sulphuric acid 75 % w/w) or 9 (carbon disulphide/acetone 55.5/44.5 w/w) and 7 (sulphuric acid 75 % w/w)

(1) When variant 4 is used, first remove the first component using the first reagent.

COUNCIL DECISION

of 8 March 1973

amending the statutes of the Euratom Supply Agency following the Accession of new Member States to the Community

(73/45/Euratom)

THE COUNCIL OF THE EUROPEAN COMMUNITIES,

Article 2

Having regard to the Treaty establishing the European Atomic Energy Community, and in particular Article 54 (2) and (3) thereof;

Having regard to the proposal from the Commission;

HAS DECIDED:

Article X (1) and (2) of the Statutes of the Supply Agency shall be amended as follows:

'1. An Advisory Committee for the Agency shall be set up comprising 33 members.

2. Seats shall be allotted to nationals of the Member States as follows:

Article 1

Article V (1) and (2) of the Statutes of the Euratom Supply Agency ⁽¹⁾ shall be amended as follows:

'1. The capital of the Agency shall be 3 200 000 EMA units of account.

2. The capital shall be divided according to the following scale:

Belgium:	6 %
Germany:	21 %
Denmark:	3 %
France:	21 %
Ireland:	1 %
Italy:	21 %
Netherlands:	6 %
United Kingdom:	21 %

Belgium:	3 members
Germany:	6 members
Denmark:	2 members
France:	6 members
Ireland:	1 member
Italy:	6 members
Netherlands:	3 members
United Kingdom:	6 members

Done at Brussels, 8 March 1973.

For the Council

The President

W. De CLERCQ

⁽¹⁾ OJ No 27, 6. 12. 1958, p. 534/58.

COMMISSION

FOURTH COMMISSION DIRECTIVE

of 5 December 1972

establishing Community methods of analysis for the official control of feedingstuffs

(73/46/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community;

Having regard to the Council Directive of 20 July 1970 ⁽¹⁾ on the introduction of Community methods of sampling and analysis for the official control of feedingstuffs, as amended by the Directive No 72/275/EEC of 20 July 1972 ⁽²⁾, and in particular Article 2 thereof;

Whereas that Directive requires that official controls of feedingstuffs be carried out using Community methods of sampling and analysis for the purpose of checking compliance with requirements arising under the provisions laid down by law, Regulation or administrative action concerning the quality and composition of feedingstuffs;

Whereas Directives No 71/250/EEC of 15 June 1971 ⁽³⁾, No 71/393/EEC of 18 November 1971 ⁽⁴⁾ and No 72/199/EEC of 27 April 1972 ⁽⁵⁾ have already established a number of Community methods of analysis; whereas the progress of work since then makes it advisable to adopt a fourth set of methods;

Whereas the measures provided for in this Directive are in accordance with the Opinion of the Management Committee for Feedingstuffs;

HAS ADOPTED THIS DIRECTIVE:

Article 1

The Member States shall require that analyses for official controls of feedingstuffs as regards moisture contents of animal and vegetable fats and oils and

magnesium and crude fibre contents of feedingstuffs be carried out according to the methods described in Annex I to this Directive.

The general provisions set out in Part 1 (Introduction) of the Annex to the First Commission Directive No 71/250/EEC of 15 June 1971 shall be applicable to the methods described in Annex I to this Directive.

Article 2

The Member States shall require that analyses for official controls of feedingstuffs as regards their contents of retinol (vitamin A), thiamine (aneurine, vitamin B₁), ascorbic and dehydroascorbic acids (vitamin C) be carried out according to the methods described in Annex II to this Directive.

The general provisions set out in Part 1 (Introduction) of the Annex to the First Commission Directive 71/250/EEC of 15 June 1971, with the exception of the part dealing with preparation of the sample to be analysed, shall be applicable to the methods described in Annex II to this Directive.

Article 3

The Member States shall, not later than 1 January 1974, bring into force the laws, regulations or administrative provisions necessary to comply with this Directive. They shall forthwith notify the Commission thereof.

Article 4

This Directive is addressed to the Member States.

Done at Brussels, 5 December 1972.

For the Commission

The President

S. L. MANSHOLT

⁽¹⁾ OJ No L 170, 3. 8. 1970, p. 2.

⁽²⁾ OJ No L 171, 29. 7. 1972, p. 39.

⁽³⁾ OJ No L 155, 12. 7. 1971, p. 13.

⁽⁴⁾ OJ No L 279, 20. 12. 1971, p. 7.

⁽⁵⁾ OJ No L 123, 29. 5. 1972, p. 6.

ANNEX I

1. DETERMINATION OF MOISTURE IN ANIMAL AND VEGETABLE FATS AND OILS

1. Purpose and Scope

This method makes it possible to determine the water and volatile substances content of animal and vegetable fats and oils.

2. Principle

The sample is dried to constant weight at 103 °C. The loss in mass is determined by weighing.

3. Apparatus

- 3.1. Flat-bottomed dish, of a corrosion-resistant material, 8 to 9 cm in diameter and approximately 3 cm high.
- 3.2. Mercury thermometer with a strengthened bulb and expansion tube at the top end, graduated from approximately 80 °C to at least 110 °C, and approximately 10 cm in length.
- 3.3. Sand bath or electric hot-plate.
- 3.4. Desiccator, containing an efficient drying agent.
- 3.5. Analytical balance.

4. Procedure

Weigh out to the nearest mg approximately 20 g of the homogenized sample into the dry, weighed dish (3.1) containing the thermometer (3.2). Heat on the sand bath or hot-plate (3.3), stirring continuously with the thermometer, so that the temperature reaches 90 °C in about 7 minutes.

Reduce the heat, watching the frequency with which bubbles rise from the bottom of the dish. The temperature must not exceed 105 °C. Continue to stir, scraping the bottom of the dish, until bubbles stop forming.

In order to ensure complete elimination of moisture, reheat several times to 103 °C ± 2 °C, cooling to 93 °C between successive heatings. Then leave to cool to room temperature in the desiccator (3.4) and weigh. Repeat this operation until the loss in mass between two successive weighings no longer exceeds 2 mg.

N.B. An increase in the mass of the sample after repeated heating indicates an oxidation of the fat, in which case calculate the result from the weighing carried out immediately before the mass began to increase.

5. Calculation of results

The moisture content, as a percentage of the sample, is given by the following formula:

$$(M_1 - M_2) \cdot \frac{100}{M_0}$$

where:

M_0 = mass, in grammes, of the test sample;

M_1 = mass, in grammes, of the dish with its contents before heating;

M_2 = mass, in grammes, of the dish with its contents after heating.

Results lower than 0.05 % must be recorded as 'lower than 0.05 %'.

Repeatability

The difference in moisture between the results of two parallel determinations carried out on the same sample must not exceed 0.05 %, in absolute value.

2. DETERMINATION OF MAGNESIUM

— by atomic absorption spectrophotometry —

1. Purpose and Scope

This method makes it possible to determine the quantity of magnesium in feeding stuffs. It is particularly appropriate for determining magnesium contents lower than 5 %.

2. Principle

The sample is ashed and dissolved in dilute hydrochloric acid. If it contains no organic substances, it is dissolved directly in dilute hydrochloric acid. The solution is diluted and the magnesium content determined by atomic absorption spectrophotometry at 285.2 nm, by comparison with standard solutions.

3. Reagents

- 3.1. Hydrochloric acid a.p. d: 1.16.
- 3.2. Concentrated hydrochloric acid a.p. d: 1.19.
- 3.3. Magnesium ribbon or wire, or magnesium sulphate heptahydrate, dried at room temperature.
- 3.4. Strontium salt solution (chloride or nitrate) at 2.5 % (w/v) strontium (= 76.08 g $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ a.p., or 60.38 g $\text{Sr}(\text{NO}_3)_2$ a.p.).
- 3.5. Standard magnesium solution: weigh out to the nearest mg 1 g magnesium (3.3) which has previously had its oxide coating carefully removed, or the corresponding quantity (10.143 g) of magnesium sulphate heptahydrate (3.3). Place in a 1 000 ml graduated flask, add 80 ml hydrochloric acid (3.1), leave to dissolve and make up to 1 000 ml with water. 1 ml of this solution contains 1.000 mg magnesium.

4. Apparatus

- 4.1. Platinum, silica or porcelain ashing crucibles.
- 4.2. Thermostatically controlled electric muffle furnace.
- 4.3. Atomic absorption spectrophotometer.

5. Procedure

5.1. Preparation of the sample solution

5.1.1. Feeding stuffs composed exclusively of mineral substances

Weigh out to the nearest mg 5 g of the sample into a 500 ml graduated flask with 250 to 300 ml water. Add 40 ml hydrochloric acid (3.1), bring to the boil and keep the liquid gently boiling for 30 minutes. Leave to cool, make up to volume with water, mix and filter into a dry beaker through a dry pleated filter. Discard the first 30 ml of the filtrate. In the presence of silica, treat 5 g of sample with a sufficient quantity (15–30 ml) of hydrochloric acid (3.2), evaporate to dryness on a water bath and transfer to an oven at 105 °C for one hour. Proceed as from the third sentence of 5.1.2.

5.1.2. Feeding stuffs composed predominantly of mineral substances

Weigh out to the nearest mg 5 g of the sample into a crucible and ash at 550 °C in the muffle furnace until an ash which is free from carbonaceous particles is obtained, and leave to cool. In order to eliminate silica, add to the ash a sufficient quantity (15–30 ml) of hydrochloric acid (3.2), evaporate to dryness on a water bath and transfer to an oven at 105 °C for one hour. Treat the residue with 10 ml hydrochloric acid (3.1) and transfer to a 500 ml graduated flask using warm water. Leave to cool and make to volume with water. Mix and filter into a dry beaker through a dry pleated filter. Discard the first 30 ml of the filtrate.

5.1.3. *Feedingstuffs composed predominantly of organic substances*

Weigh out to the nearest mg 5 g of the sample into a crucible and ash at 550 °C in the muffle furnace until an ash which is free from carbonaceous particles is obtained. Treat the ash with 5 ml hydrochloric acid (3.2), evaporate to dryness on a water bath and then dry for one hour in the oven at 105 °C in order to render the silica insoluble. Treat the ash with 5 ml hydrochloric acid (3.1), transfer to a 250 ml graduated flask using warm water, bring to the boil, leave to cool and make up to volume with water. Mix and filter into a dry beaker through a dry pleated filter. Discard the first 30 ml of the filtrate.

5.2. *Measurement by atomic absorption*

By diluting the standard solution (3.5) with water, prepare at least 5 reference solutions of increasing concentration, corresponding to the optimal measuring range of the spectrophotometer. Add to each solution 10 ml strontium salt solution (3.4) and then make up the volume to 100 ml with water. Dilute with water one aliquot part of the filtrate obtained from 5.1.1, 5.1.2 or 5.1.3, so as to obtain a magnesium concentration which is within the limits of concentration of the reference solutions. The hydrochloric acid concentration of this solution must not exceed 0.4 N. Add 10 ml strontium salt solution (3.4) and then make up the volume to 100 ml with water. Measure the absorption of the solution to be determined and of the reference solutions at 285.2 nm.

6. Calculation of results

Calculate the quantity of magnesium in the sample by relation to the reference solutions. Express the result as a percentage of the sample.

Repeatability

The difference between the results of two parallel determinations carried out on the same sample must not exceed 5 %, in relative value.

3. DETERMINATION OF CRUDE FIBRE

1. Purpose and Scope

This method makes it possible to determine in feeding stuffs the quantity of fat-free organic substances which are insoluble in acid and alkaline media and are conventionally described as crude fibre.

2. Principle

The sample, defatted where necessary, is treated successively with boiling solutions of sulphuric acid and potassium hydroxide of specified concentrations. The residue is separated by filtration in the presence of asbestos, washed, dried, weighed and ashed at 900 °C. The loss of weight resulting from ashing corresponds to the crude fibre present in the test sample.

3. Reagents

3.1. Sulphuric acid 0.26 N.

3.2. Treated asbestos: add to asbestos of the type used with a Gooch crucible approximately 5 times its weight of dilute hydrochloric acid (1 volume hydrochloric acid, d: 1.19 + 3 volumes of water). Boil the mixture for approximately 45 minutes, leave to cool and filter through a Buchner funnel. Wash the residue first with water until hydrochloric acid has disappeared from the washing water, and then with acetone (3.6). Dry the asbestos in the drying oven and then ash for 2 hours at 900 °C. Leave to cool and keep in a stoppered flask. Asbestos treated in this way may be used several times. It must meet the specifications given in 5. regarding the blank test.

- 3.3. Antifoaming emulsion (eg silicone).
- 3.4. Potassium hydroxide solution 0.23 N.
- 3.5. Hydrochloric acid 0.5 N.
- 3.6. Acetone.
- 3.7. Diethyl ether.

4. Apparatus

- 4.1. Beakers of at least 600 ml capacity, with measuring marks at the 200 ml level.
- 4.2. Porcelain discs approximately 80 mm in diameter and approximately 4 mm thick, perforated with approximately 32 holes, each approximately 4 mm in diameter.
- 4.3. Rubber-stoppered vacuum flasks of approximately 2 litre capacity, with measuring marks at the 800 ml level and fitted with glass funnels 120 mm in diameter.
- 4.4. Filter plates approximately 40 mm in diameter and approximately 4 mm thick, with slanting edges to fit the cone of the funnel (4.3), perforated with approximately 16 holes, each approximately 4 mm in diameter, and covered by a wire mesh, the mesh size being approximately 1 mm. Both plates and wire mesh must be resistant to acids and alkalis.
- 4.5. Platinum or silica ashing crucibles.
- 4.6. Thermostatically controlled electric muffle furnace.
- 4.7. Desiccator.
- 4.8. Asbestos filter: Suspend 2.0 g asbestos (3.2) in 100 ml water.

Filter under vacuum over a filter plate covered with a wire mesh (4.4) and placed in the funnel of a vacuum flask (4.3). Collect the filtrate and filter once more through the same filter. Discard the filtrate.

5. Procedure

Weigh out to the nearest mg 3 g of the sample and 2 g treated asbestos (3.2) into a beaker (4.1), add 200 ml sulphuric acid (3.1) and a few drops of antifoaming emulsion (3.3). Bring rapidly to the boil and leave to boil for exactly 30 minutes. To keep a constant volume, cover the beaker with a cooling device such as a 500 ml round-bottomed flask in which cold water is circulated. Stop boiling by adding approximately 50 ml cold water and filter immediately under vacuum through an asbestos filter previously prepared as shown in 4.8.

Wash the residue with 5 lots of approximately 100 ml of very hot water to obtain a final volume of filtrate of 800 ml. Transfer the residue quantitatively to the beaker (4.1) which has first been fitted with a porcelain disc (4.2) to regulate the boiling. Add 200 ml potassium hydroxide solution (3.4). Bring rapidly to the boil and leave to boil for exactly 30 minutes. Add approximately 50 ml cold water and filter immediately under vacuum through a fresh asbestos filter previously prepared as shown in 4.8. Wash the residue with very hot water until the washing water is neutral (test with litmus paper), then 3 times with acetone (3.6) (approximately 100 ml acetone in all).

Transfer the residue quantitatively to an ashing crucible (4.5), break up if necessary and dry to constant weight in the drying oven at 130 °C.

Leave to cool in the desiccator (4.7) and weigh rapidly.

Place the crucible in the muffle furnace (4.6) and leave to ash for 30 minutes at 900 °C. Leave to cool in the desiccator (4.7) and weigh rapidly.

Carry out a *blank test* applying the same procedure to the treated asbestos (3.2), but without the sample. Loss of weight resulting from ashing of the 6 g asbestos must not exceed 10 mg.

6. Calculation of results

The crude fibre content, as a percentage of the sample, is given by the ratio:

$$\frac{(a - b) \cdot 100}{3}$$

where:

a = loss of weight after ashing during the determination;

b = loss of weight after ashing during the blank test.

Repeatability

The difference between the results of two parallel determinations carried out on the same sample must not exceed:

0.3, in absolute value, for crude fibre contents lower than 10 %;

3 %, in relative value, for crude fibre contents equal to or greater than 10 %.

7. Observations

7.1. Feedingstuffs containing more than 10 % oils and fats must be defatted prior to analysis with diethyl ether (3.7). To do this, place the test sample (3 g weighed to the nearest mg) on an asbestos filter (4.8). Cover 3 times with approximately 50 ml diethyl ether (3.7) and each time filter carefully under vacuum. Transfer the defatted test sample and the asbestos quantitatively to a beaker (4.1) and continue the analysis as shown in 5.

7.2. Feedingstuffs containing oils and fats which cannot be extracted directly must be defatted as shown in 7.1 and defatted a further time after the acid attack has been washed from the residue.

To do this, wash this attack from the residue 3 times with acetone (3.6) (100 ml in all), then 3 times with 50 ml diethyl ether (3.7). Then transfer the residue quantitatively to a beaker (4.1) and continue the analysis as shown in the second paragraph of 5 (treatment with potassium hydroxide solution).

7.3. If the feedingstuffs are rich in calcium (more than 2 % calcium), place the test sample (3 g weighed to the nearest mg) in a beaker (4.1) with 100 ml hydrochloric acid 0.5 N (3.5) and leave to stand in a cool temperature for 5 minutes. Filter immediately and wash in cold water. Use as a filtration aid the 2.0 g asbestos specified for boiling with sulphuric acid. If filtration proves difficult, dilute the suspension with acetone (3.6). Then proceed as shown in 5.

ANNEX II

1. DETERMINATION OF RETINOL (VITAMIN A)

1. Purpose and Scope

This method makes it possible to determine the quantity of retinol (Vitamin A) in feedingstuffs, concentrates and premixes. The lower limit of the determination is 10 000 IU/kg for highly pigmented feeds and 4 000 IU/kg for others ⁽¹⁾. Products are classified in two groups, according to their presumed retinol content:

Group A: contents lower than 200 000 IU/kg;

Group B: contents equal to or greater than 200 000 IU/kg.

2. Principle

The sample is hydrolised when hot with a potassium hydroxide solution in an ethanol medium and in the presence of an antioxidant or in a nitrogen atmosphere. The mixture is extracted with 1,2-dichloroethane. The extract is evaporated to dryness and treated with light petroleum. The solution is chromatographed on a column of aluminium oxide (for

⁽¹⁾ 1 IU = 0.3 µg of retinol.

Group B products, chromatography is only required in certain cases). For Group A products the retinol is determined by spectrophotometry at 610 nm after development of a coloured complex according to the Carr-Price reaction; for Group B products by spectrophotometry in the UV at 325 nm.

3. Reagents

(a) *used for analysing products of Groups A and B*

- 3.1. 96 % (v/v) ethanol.
- 3.2. 10 % (w/v) sodium ascorbate solution a.p., or
- 3.3. Purified nitrogen.
- 3.4. 50 % (w/v) potassium hydroxide solution a.p.
- 3.5. Potassium hydroxide solution 1 N a.p.
- 3.6. Potassium hydroxide solution 0.5 N a.p.
- 3.7. 1,2-dichlorethane a.p.
- 3.8. Light petroleum, boiling range: 30–50 °C. If necessary, purify as follows: stir 1 000 ml light petroleum with 20 ml lots of concentrated sulphuric acid until the acid remains colourless. Remove the acid and wash the light petroleum successively with 500 ml water, twice with 250 ml of 10 % (w/v) sodium hydroxide solution and three times with 500 ml water. Remove the aqueous layer, dry the light petroleum for 1 hour over active carbon and anhydrous sodium sulphate, filter and distil.
- 3.9. Aluminium oxide, standardized according to Brockmann: ash for 8 hours at 750 °C, cool in a desiccator and keep in a brown glass bottle fitted with a ground-glass stopper. Before use in chromatography moisten as follows: place in a brown glass bottle 10 g aluminium oxide and 0.7 ml water, seal with a stopper, reheat for 5 minutes in a boiling water bath while shaking. Leave to cool. Verify the activity of the aluminium thus prepared by subjecting a known quantity of retinol (3.17) (ca. 500 IU) to the procedure of 5.3 and 5.4 and checking recovery.
- 3.10. Basic aluminium oxide, degree of activity 1 (Woelm, Merck or equivalent).
- 3.11. Pure diethyl ether. Remove peroxides and traces of water by chromatography on a column of basic aluminium oxide (3.10). (25 g aluminium oxide per 250 ml diethyl ether.)
- 3.12. Light petroleum solutions (3.8) at 4, 8, 12, 16 and 20 % (v/v) diethyl ether (3.11).
- 3.13. Sodium sulphide solution 0.5 molar in 70 % (v/v) glycerine, prepared from sodium sulphide a.p.

(b) *used exclusively for analysing Group A products*

- 3.14. Crystallizable benzene a.p.
- 3.15. Chloroform a.p. Remove the ethanol, phosgene and traces of water by chromatography on a column of basic aluminium oxide (3.10) (50 g aluminium oxide per 200 ml chloroform; it is advisable to chromatograph the first 50 ml of the eluate a second time).
- 3.16. Carr-Price reagent: stir approximately 25 g antimony trichloride a.p. (kept in a desiccator) with 100 ml chloroform (3.15) until the solution is saturated. A slight deposit of antimony trichloride causes no problem. Add 2 ml acetic anhydride a.p. Keep in a refrigerator in a brown glass bottle with ground-glass stopper. The solution keeps for several weeks.
- 3.17. Retinol — standardized spectrophotometrically.

(c) *used exclusively for analysing Group B products*

- 3.18. Isopropanol, for chromatography.

4. Apparatus

- 4.1. Water bath.
- 4.2. Vacuum evaporation apparatus with round flasks of different capacities.
- 4.3. Glass chromatography tubes (length: 300 mm; internal diameter: about 13 mm).
- 4.4. Spectrophotometer with 10 mm cells. Measurements in the UV require silica cells.
- 4.5. UV lamps suitable for 365 nm.

5. Procedure

N.B. All operations must be carried out away from direct light, if necessary in brown glass equipment.

5.1. Test Sample

From the finely divided sample, take a test sample proportional to the presumed retinol content, thus:

0.1–1.0 g for concentrates (contents greater than 20 000 IU/g);

3.0–5.0 g for premixes (contents of between 400 and 20 000 IU/g);

10–20 g for mineral mixtures;

30 g for Group A products.

Immediately place the test sample in a 500 ml flask with a ground-glass stopper.

5.2. Hydrolysis and extraction ⁽¹⁾

Add successively to the test sample 40 ml ethanol (3.1), 2 ml sodium ascorbate solution (3.2) ⁽²⁾, 10 ml potassium hydroxide solution (3.4) and 2 ml sodium sulphide solution (3.13).

Heat for 30 minutes at 70–80 °C under a reflux condenser and then leave to cool under a stream of water. Add 50 ml ethanol (3.1) and 100 ml 1,2-dichlorethane (3.7) (taken with a pipette). Shake vigorously and then decant the supernatant liquid into a decanting container. Add to the container 150 ml potassium hydroxide solution (3.5), shake for 30 seconds and leave to stand until the layers are separated. Collect the dichlorethane layer (lower layer) in a decanting container, add 40 ml potassium hydroxide solution (3.6), shake for 10 seconds and leave to stand until the layers are separated. Collect the dichlorethane layer in a decanting container and wash 6–8 times with 40 ml lots of water until free of alkali (phenolphthalein test). Collect the dichlorethane layer and remove the last traces of water using strips of filter paper.

Evaporate to dryness an aliquot part of the solution under vacuum and on the water bath at 40 °C. Rapidly treat the residue with 5 ml light petroleum (3.8).

For Group A products, chromatograph as shown in 5.3.1.

For Group B products, transfer the solution to a 50 ml graduated flask, make up to volume with light petroleum (3.8), mix and measure the optical density as shown in 5.4.2.

5.3. Chromatography

5.3.1. Group A products

Fill a chromatography tube (4.3) to a height of 200 mm with 10 g aluminium oxide (3.9) previously slurred with light petroleum (3.8). Place in the tube the solution obtained in 5.2 and immediately add 20 ml light petroleum (3.8). Elute successively with 10 ml lots of the light petroleum solutions at 4, 8, 12, 16 and 20 % diethyl ether (3.12) under pressure or partial vacuum, the rate of flow being 2 to 3 drops per second.

(1) For milk feeds and products with a tendency to agglomerate or swell, double the quantity of the reagents shown in the first and second paragraphs of 5.2.

(2) Sodium ascorbate need not be added when hydrolysis is carried out in a nitrogen atmosphere.

The carotene is eluted first⁽¹⁾. The retinol is generally eluted with the light petroleum solution at 20 % diethyl ether (3.12). The elution is followed under UV light (brief irradiation of the column with the mercury lamp). The fluorescent zone of the retinol is clearly separated from the yellow xanthophyll zones following it. Collect the eluate fraction containing the retinol in an Erlenmeyer flask.

5.3.2. Group B products

Chromatography must only be carried out if the optical density measurements obtained in 5.4.2 do not conform to the requirements given in 5.4.2.

If chromatography proves necessary, place in the chromatography column an aliquot part of the solution in the light petroleum obtained in 5.2, containing approximately 500 IU of retinol, and chromatograph as shown in 5.3.1.

5.4. Measurement of the optical density

5.4.1. Group A products

Evaporate to dryness under vacuum the eluate containing the retinol obtained in 5.3.1. Treat the residue with 2 ml benzene (3.14). Take 0.3 ml of this solution and add 3 ml of the Carr-Price reagent (3.16). A blue colouring develops. Measure the optical density with the spectrophotometer at 610 nm exactly 30 seconds after the reaction has begun. Determine the retinol content by reference to a standard curve obtained from benzene solutions of increasing retinol-standard concentrations treated with Carr-Price reagent (2 to 16 IU retinol-standard (3.17) per 0.3 ml benzene (3.14) + 3 ml Carr-Price reagent (3.16)). The standard curve must be checked regularly and frequently using the standard and a freshly prepared Carr-Price reagent solution.

5.4.2. Group B products

Take an aliquot part of the solution in light petroleum obtained in 5.2 containing approximately 200 IU retinol. Evaporate to dryness under vacuum and treat the residue with 25 ml isopropanol (3.18). Measure the optical density in the spectrophotometer at 325, 310 and 334 nm. The absorption maximum is located at 325 nm. The retinol content of the solution is calculated as follows:

$$E_{325} \cdot 18.30 = \text{IU of retinol/ml}$$

However, the ratio of the optical densities

$$E_{310} : E_{325} \text{ and } E_{334} : E_{325}$$

must be $6 : 7 = 0.857$.

If one of these ratios differs appreciably from this value (< 0.830 or > 0.880), the measurement of the optical densities must be preceded by chromatography in accordance with the method given in 5.3.2. If the measurement of the optical densities carried out after chromatography shows that the abovementioned ratios still differ appreciably from the value of 0.857 (< 0.830 or > 0.880), the determination must be carried out in accordance with the method given for Group A products.

6. Calculation of results

Calculate the retinol content of the sample taking into account the weight of the test sample and the dilutions carried out in the course of analysis. Express the results in IU of retinol per kg of feedingstuff or per kg of concentrate or premix.

(1) Carotene content may be determined by optical density measurement at 450 nm; $E \frac{1\%}{1\text{ cm}} = 2600$

Repeatability

The difference between the results of two parallel determinations carried out on the same sample must not exceed:

- 20 %, in relative value, for retinol contents lower than 75 000 IU/kg;
- 15 000 IU for contents between 75 000 and 150 000 IU/kg;
- 10 %, in relative value, for contents between 150 000 and 250 000 IU/kg;
- 25 000 IU for contents between 25 000 and 500 000 IU/kg;
- 5 %, in relative value, for contents greater than 500 000 IU/kg.

2. DETERMINATION OF THIAMINE (VITAMIN B₁, ANEURINE)

1. Purpose and Scope

This method makes it possible to determine the quantity of thiamine (aneurine, Vitamin B₁) in feeding stuffs, concentrates and premixes. The lower limit of the determination is 5 ppm.

2. Principle

The solution is treated when hot with dilute sulphuric acid and then hydrolysed enzymatically. The solution obtained is subjected to alkaline oxidation. The thiochrome formed is extracted with isobutanol and determined by fluorimetry.

3. Reagents

- 3.1. 100 µg/ml standard thiamine solution: dissolve 112.3 mg thiamine hydrochloride, previously dried under vacuum to constant weight, in 1 000 ml sulphuric acid 0.2 N (3.2). If stored in a cool, dark place, this solution keeps for one month.
- 3.2. Sulphuric acid 0.2 N.
- 3.3. Pure sodium bisulphite.
- 3.4. 20 % (w/v) potassium ferricyanide solution a.p.
- 3.5. 25 % (w/v) potassium hydroxide solution a.p.
- 3.6. Oxidizing mixture: mix 2 ml potassium ferricyanide solution (3.4) with 48 ml potassium hydroxide solution (3.5). This mixture does not keep for more than 4 hours.
- 3.7. Isobutanol a.p.
- 3.8. Sodium acetate solution 2.5 N.
- 3.9. Multienzymatic preparation containing protease, phosphatase and amylase (eg, Clarase).
- 3.10. 96 % (v/v) ethanol.

4. Apparatus

- 4.1. Water bath.
- 4.2. Centrifuge (3 500 rpm) with 30 to 50 ml tubes fitted with ground-glass stoppers.
- 4.3. Fluorimeter.

5. Procedure

5.1. Enzymatic hydrolysis

Place in each of two 250 ml graduated flasks, A and B, identical amounts of the finely divided sample containing approximately 100 µg thiamine and 125 ml sulphuric acid (3.2). Also add, to flask A only, 1.0 ml standard solution (3.1) (internal standard).

Shake the flasks vigorously, place on a boiling water bath and keep there for 15 minutes, shaking occasionally. Leave to cool to approximately 45 °C. Add to each flask 20 ml sodium acetate solution (3.8) and 0.5 g multienzymatic preparation (3.9), then leave to stand for 20 minutes at room temperature. Add 20 ml sodium acetate solution (3.8), make up to volume with water, homogenize and filter. Collect filtrates A and B after having discarded the first 15 ml. Prepare the following solutions:

5.1.1. *Reference solution T*

Place in a centrifuge tube (4.2) 5 ml filtrate A and approximately 10 mg sodium bisulphite (3.3). Immerse the tube in a boiling water bath for 15 minutes and then leave to cool to room temperature.

5.1.2. *Solutions A (internal standard) and B (sample)*

Place 5 ml filtrate A in a centrifuge tube (4.2) and 5 ml filtrate B in another centrifuge tube (4.2).

5.2. *Oxidation*

Add to solutions T, A and B 5 ml of the oxidising mixture (3.6) and, one minute later, 10 ml isobutanol (3.7). Stopper the tubes and shake vigorously for 5 seconds. Leave to stand for one minute and centrifuge so as to separate the layers. From each tube transfer 5 ml of the supernatant isobutanol layer to each of the 25 ml graduated flasks, make up to volume with ethanol (3.10) and homogenize (= extracts T, A and B).

5.3. *Measurement of fluorescence*

Carry out the measurements at the wavelength for which the fluorimeter gives an optimal response to the fluorescence of the thiochrome. Irradiate at approximately 365 nm.

Adjust the instrument to zero using extract T. Measure the intensity of fluorescence of extracts A and B.

6. *Calculation of results*

The thiamine content in mg/kg of the sample is given by the ratio:

$$\frac{d \cdot b}{(a - b) c}$$

where:

a = intensity of fluorescence of extract A (internal standard);

b = intensity of fluorescence of extract B (sample);

c = weight of the test sample in g;

d = amount of thiamine in µg added to the test sample (internal standard).

Repeatability

The difference between the results of two parallel determinations carried out on the same sample must not exceed:

10 %, in relative value, for contents lower than 500 mg/kg, and

5 %, in relative value, for contents equal to or greater than 500 mg/kg.

3. DETERMINATION OF ASCORBIC ACID AND DEHYDROASCORBIC ACID (VITAMIN C)

1. *Purpose and Scope*

This method makes it possible to determine the total quantity of ascorbic and dehydroascorbic acids (Vitamin C) in feedingstuffs, concentrates and premixes. The lower limit of the determination is 5 ppm. Products are classified in two groups, according to their presumed Vitamin C content:

Group A: contents lower than 10 g/kg;

Group B: contents equal to or greater than 10 g/kg.

2. Principle

The sample is suspended in a dilute solution of metaphosphoric acid and extracted with chloroform. The aqueous phase is treated with a solution of 2,6-dichlorophenol-indophenol in order to transform the ascorbic acid into dehydroascorbic acid, and then with a solution of 2,4-dinitrophenylhydrazine. The hydrazone formed is extracted with a mixture of ethyl acetate, glacial acetic acid and acetone. The solution is chromatographed on a column of silica gel, the eluate evaporated to dryness and the residue dissolved in dilute sulphuric acid. The optical density of the solution is measured by a spectrophotometer at 509 nm.

For Group A products the eluate resulting from chromatography on the column is further subjected to thin layer chromatography to isolate the hydrazone.

3. Reagents

- 3.1. Standard solution of 0.05 % L-ascorbic acid: dissolve 50 mg L-ascorbic acid a.p. in approximately 20 ml metaphosphoric acid solution (3.2) and make up to 100 ml with water. Prepare immediately before use.
- 3.2. 10 % (w/v) metaphosphoric acid solution: after grinding it in a mortar, dissolve in water 200 g metaphosphoric acid a.p. and make up to 2 000 ml with water. Keep at 4 °C. This is stable for one week.
- 3.3. Chloroform a.p.
- 3.4. Solution of 0.5 % (w/v) 2,6-dichlorophenol-indophenol a.p. Prepare immediately before use.
- 3.5. Filtration aid (S. and S. No 121 or equivalent).
- 3.6. Acid solution of 2 % (w/v) 2,4-dinitrophenylhydrazine: dissolve 2 g 2,4-dinitrophenylhydrazine in 100 ml dilute sulphuric acid (25 ml sulphuric acid a.p., d: 1.84, diluted by making up to 100 ml with water). Stored at a cool temperature this solution keeps for one week.
- 3.7. Nitrogen, or
- 3.8. Carbon dioxide.
- 3.9. Mixture of ethyl acetate a.p./glacial acetic acid/acetone a.p.: 96/2/2 in volume.
- 3.10. Mixture of dichloromethane a.p./glacial acetic acid: 97/3 in volume.
- 3.11. Silica gel, particle size: 0.05 to 0.2 mm.
- 3.12. Stahl grade silica gel H, for thin layer chromatography.
- 3.13. Dilute sulphuric acid: place 105 ml water in a 200 ml graduated flask, make up to volume with sulphuric acid a.p., d: 1.84.
- 3.14. Eluting solvent for thin layer chromatography: mix 75 ml diethyl ether a.p., 25 ml ethyl acetate a.p. and 4.0 ml 96 % (w/v) acetic acid a.p. Renew after 2 to 3 chromatographies.

4. Apparatus

- 4.1. Water bath fitted with a thermostat set at 20 °C.
- 4.2. Centrifuge (3 500 rpm) with 40 to 50 ml tubes fitted with ground-glass stoppers.
- 4.3. Rotary vacuum evaporator with 250 ml flasks.
- 4.4. Glass chromatography tubes (length: 100 mm, internal diameter: 20 mm), with a sintered disc (eg Allihn tubes).
- 4.5. Spectrophotometer or colorimeter with filters with 10 mm cells.
- 4.6. Apparatus for thin layer chromatography, with silica gel plates (3.12) coated to a depth of 0.5 to 0.6 mm. (Ready-made plates are appropriate.) Dry the plates for 2½ to 3 hours in the drying oven at 120 to 130 °C. Leave to cool and then keep in a desiccator for at least 24 hours before use.
- 4.7. Drying oven set at 120 to 130 °C.

5. Procedure

5.1. Extraction

Place in each of two 250 ml graduated flasks (with ground-glass stoppers), A and B, identical quantities of the finely divided sample containing about 200 µg vitamin C. Add to *flask A only* 0.4 ml standard solution (3.1) and mix, shaking gently (internal standard).

Add to each flask 30 ml chloroform (3.3) and 25 ml metaphosphoric acid solution (3.2) at 4 °C. Shake briefly and then leave to stand for 10 to 15 minutes. Add 25 ml water, stopper the flasks, shake vigorously for 10 seconds and leave to stand for 10 to 15 minutes in the water bath (4.1). Centrifuge to separate the aqueous phase from the chloroform phase. Carry out the operations simultaneously, as described below, on the aqueous extracts A (internal standard) and B.

5.2. Oxidation

Using a pipette, transfer 40 ml of the supernatant aqueous solution (slightly cloudy) obtained in 5.1 to a reaction tube fitted with a ground-glass stopper, add 0.5 to 1 ml 2,6-dichlorophenol-indophenol solution (3.4) and mix. A red colouring develops which should remain for at least 15 minutes. Then add approximately 300 mg filtration aid (3.5), shake and filter through a dry pleated filter. The filtrate need not necessarily be clear.

5.3. Reaction with 2,4-dinitrophenylhydrazine and hydrazone extraction

Using a pipette, transfer 10 ml of the filtrate obtained in 5.2 to a centrifuge tube (4.2), add 2 ml 2,4-dinitrophenylhydrazine solution (3.6) and mix. Pass a stream of nitrogen (3.7) or carbon dioxide (3.8) rapidly into the tube, stopper the tube and immerse it for approximately 15 hours (overnight) in the water bath (4.1).

Then add 3 ml water, 20 ml of the ethyl acetate/glacial acetic acid/acetone mixture (3.9) and approximately 800 mg filtration aid (3.5). Stopper the tube, shake vigorously for 30 seconds and centrifuge. Place 15 ml of the supernatant phase in an evaporation flask and evaporate under reduced pressure in the rotary evaporator (4.3) until an oily residue is obtained. Dissolve the residue in 2 ml of the ethyl acetate/glacial acetic acid/acetone mixture (3.9) by re-heating at 50 °C, leave to cool, add 10 ml of the dichloromethane/glacial acetic acid mixture (3.10) and mix.

5.4. Chromatography on a column

Fill a chromatography tube (4.4) up to a level of 30 mm with the dichloromethane/glacial acetic acid mixture (3.10). Suspend (shaking vigorously) 5 g silica gel (3.11) in 30 ml of the dichloromethane/glacial acetic acid mixture (3.10); pour the suspension into the tube. Leave to stand and then compress under nitrogen (3.7) at low pressure. Decant into the tube the solution obtained in 5.3, rinse the flask with a small quantity of the dichloromethane/glacial acetic acid mixture (3.10) and decant into the tube, then fill the latter with the mixture (3.10) and proceed to wash the column with the same mixture (3 to 4 lots of approximately 5 ml) until a colourless eluate is obtained. Discard the part of the eluate which is coloured yellow.

Elute the reddish zone at the top of the column with the ethyl acetate/glacial acetic acid/acetone mixture (3.9), collect the eluate and evaporate to dryness.

5.4.1. *For Group A products (contents in vitamin C lower than 10 g/kg)*, dissolve the residue in 2.0 ml of the ethyl acetate/glacial acetic acid/acetone mixture (3.9) and chromatograph immediately on a thin layer as shown in 5.5.

5.4.2. *For Group B products (contents in vitamin C equal to or greater than 10 g/kg)*, treat the oily residue with 4.0 ml dilute sulphuric acid (3.13), shake vigorously to dissolve the residue completely and measure the optical density as shown in 5.6.

5.5. *Thin layer chromatography*

Carry out in duplicate the operations described as follows. Place in a thin line on the plate (4.6) 0.5 ml of the solution obtained in 5.4.1. Using the eluting solvent (3.14) develop for at least 20 minutes in a tank saturated with solvent vapour, until the pink-coloured hydrazone zone is clearly separated. Leave to dry in the open. Mark out the limit of the pink zone, scrape away the zone with a spatula and quantitatively transfer the powder into a chromatography tube (4.4).

Elute successively once with 2 ml and twice with 1.5 ml of the ethyl acetate/glacial acetic acid/acetone mixture (3.9). Collect the eluate in a small flask (the last part must be colourless). Evaporate to dryness, treat the oily residue with 4.0 ml dilute sulphuric acid (3.13), shake vigorously to dissolve the residue completely and measure the optical density.

5.6. *Measurement of the optical density*

Measure the optical density with a spectrophotometer at 509 nm 20 to 30 minutes after dissolving the residue in sulphuric acid. Carry out the measurements by comparison with dilute sulphuric acid (3.13).

5.7. *Blank test*

Carry out a blank test applying the same procedure but without the sample.

6. **Calculation of results**

The vitamin C content of the sample in g per kg is given by the ratio:

$$\frac{(c - a) \cdot 2}{(b - c) \cdot 10d}$$

where:

a = optical density of the blank;

b = optical density of the internal standard solution;

c = optical density of the sample solution;

d = weight, in grammes, of the test sample.

Repeatability

The difference between the results of two parallel determinations carried out on the same sample must not exceed:

10 %, in relative value, for vitamin C contents lower than 10 g/kg and

5 %, in relative value, for contents equal to or greater than 10 g/kg.

COMMISSION DIRECTIVE

of 5 December 1972

amending the second Commission Directive of 18 November 1971 establishing Community methods of analysis for the official control of feedingstuffs

(73/47/EEC)

THE COMMISSION OF THE EUROPEAN
COMMUNITIES,

Having regard to the Treaty establishing the
European Economic Community;

Having regard to the Council Directive of 20 July
1970 ⁽¹⁾ on the introduction of Community methods
of sampling and analysis for the official control of
feedingstuffs, as amended by the Directive of 20 July
1972 ⁽²⁾, and in particular Article 2 thereof;

Whereas on 18 November 1971 the Commission
adopted the second Directive ⁽³⁾ establishing
Community methods of analysis for the official
control of feedingstuffs;

Whereas Part 1 of the Annex to that Directive
provides for a method of determining moisture
which can be applied *inter alia* to the analysis of
animal and vegetable fats and oils;

Whereas moisture in animal and vegetable fats and
oils is to be determined as from 1 January 1974 by
another method which was fixed by the fourth
Commission Directive establishing Community
methods of analysis for the official control of
feedingstuffs;

Whereas therefore the obligation to apply the method
of determining moisture as specified in the second
Directive for the analysis of animal and vegetable fats
and oils should be suspended;

Whereas the measures provided for in this Directive
are in accordance with the Opinion of the Standing
Committee for Animal Feedingstuffs;

HAS ADOPTED THIS DIRECTIVE:

Article 1

The second Commission Directive establishing
Community methods of analysis for the official
control of feedingstuffs is amended as follows:

The second sentence of the first subparagraph of
point 1, Part 1 of the Annex is replaced by the
following:

'It does not cover the analysis of milk products as
straight feedingstuffs, the analysis of mineral
substances and mixtures composed predominantly
of mineral substances, the analysis of animal and
vegetable fats and oils or the analysis of the oil
seeds and oleaginous fruit defined in Council
Regulation No 136/66/EEC ⁽¹⁾ of 22 September
1966 on the establishment of a common
organization of the market in oils and fats.'

Article 2

The Member States shall put into force not later than
1 January 1973 the necessary laws, regulations and
administrative provisions in order to comply with the
provisions of this Directive. They shall forthwith
inform the Commission thereof.

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 5 December 1972.

For the Commission

The President

S. L. MANSHOLT

⁽¹⁾ OJ No L 170, 3. 8. 1970, p. 2.

⁽²⁾ OJ No L 171, 29. 7. 1972, p. 39.

⁽³⁾ OJ No L 279, 20. 12. 1971, p. 7.

COMMISSION DECISION

of 4 January 1973

extending the authority for the application of two special tariff measures to the rail carriage of Lorraine iron ore to Belgium and Luxembourg

(Only the French text is authentic)

(73/48/ECSC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Coal and Steel Community and in particular Articles 2 to 5 and 70 thereof;

Having regard to the Decision of the Commission of the European Communities No 68/349/ECSC of 31 July 1968 authorizing a special tariff for the rail carriage of Lorraine iron ore to Belgium (OJ No L 230, 19. 9. 1968, p. 8);

Having regard to the Decision of the Commission of the European Communities No 69/268/ECSC of 23 July 1969 authorizing a special tariff for the rail carriage of Lorraine iron ore to Luxembourg (OJ No L 220, 1. 9. 1969, p. 5);

Whereas, by a letter (ref. 1757) dated 31 December 1971 from the Office of the Permanent Representative of France to the European Communities, the French Government requested that the authority given by the Commission Decisions cited above in respect of the Franco-Belgian Tariff No 3232, Part III and of the Franco-Luxembourg Tariff No 3530, Part III A, be extended; and whereas, by a letter (ref. 17. 2. 31) dated 30 November 1971, the Luxembourg Government made a similar request in respect of Tariff No 3530, Part III A;

Whereas the Commission of the European Communities by the above-cited Decisions, taken pursuant to the fourth paragraph of Article 70 of the ECSC Treaty, authorized the two special tariff measures under reference for the rail carriage of Lorraine iron ore to Belgium and Luxembourg, after having ascertained that they were consistent with the Treaty; whereas its conclusion was based on the particular benefit which the railways concerned could derive from a reduced tariff and on the trifling effect which the tariff might have on normal conditions of competition in the relevant coal and steel markets; whereas the social policy objectives of the ECSC

Treaty were also being met in that the tariff measures would bring about an improvement in the living and working conditions of mineworkers in Lorraine;

Whereas these two measures have helped to halt the drop in exports of Lorraine iron ore to Belgium and Luxembourg and to stabilize the rail traffic at the level it was when the special tariff measures were applied;

Whereas the French and Luxembourg Governments support their request for extending the authority covering the two tariffs by stressing the importance which reduced tariffs, despite their adjustment as a result of a general tariff increase which took place in the meantime, still have for both the SNCF and the CFL;

Whereas Lorraine iron ore faces continuous competition from ores of better quality and at lower price which could be consigned to users in Belgium and Luxembourg by other routes and other means of transport;

Whereas the competition has recently become more acute; whereas, therefore, it will be more and more difficult to sell Lorraine ore within the Community unless the burden of transport costs is alleviated;

Whereas the circumstances which prompted the Commission to grant authority are still present, and whereas the effects of the tariff measures are not contrary to the principles of the Treaty; whereas, in particular, the application of the tariffs is beneficial to the carrier and does not affect the functioning of the common market;

Whereas the Commission must reserve the right to review its decision whenever the circumstances giving rise to it vary or no longer apply; whereas it feels that these measures and the authority to apply them should be reviewed within two years at most even if no such change has taken place; whereas they should therefore be authorized for a limited period only;

HAS ADOPTED THIS DECISION:

*Article 3**Article 1*

The authority given for the application to the rail carriage of Lorraine iron ore to Belgium and to Luxembourg of special tariff measures, namely the Franco-Belgian Tariff No 3232, Part III, and the Franco-Luxembourg Tariff No 3530, Part III A, shall continue to be effective until 31 December 1974.

This Decision is addressed to the French Republic and, as regards its application to the Franco-Luxembourg Tariff No 3530, Part III A, to the Grand Duchy of Luxembourg.

Done at Brussels, 4 January 1973.

Article 2

This Decision may be amended or repealed if the Commission considers that it is no longer warranted.

For the Commission

The President

S. L. MANSHOLT

COMMISSION DECISION

of 4 January 1973

authorizing three special tariffs for the carriage of iron ore from Western France to Belgium, Luxembourg and the Saar

(Only the French text is authentic)

(73/49/ECSC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Coal and Steel Community and in particular Articles 2 to 5 and 70 thereof;

Whereas, by a letter (ref. 1329) dated 5 November 1970 from the Office of the Permanent Representative of France to the European Communities, the French Government, pursuant to the fourth paragraph of Article 70 of the ECSC Treaty, addressed to the Commission of the European Communities a request for authority to apply the reduced Franco-Luxembourg Tariff No 3530.05 to the carriage of iron ore along the French Railways (SNCF) section of the journey from Rougé to Rodange;

Whereas, by a letter (ref. 7.2.31) dated 14 December 1970 from the Office of the Permanent Representative of Luxembourg to the European Communities, the Luxembourg Government requested authority to apply a reduced tariff on the Luxembourg Railways (CFL) section of the same journey;

Whereas, by a letter (ref. OA/mj) dated 29 December 1971 from the Office of the Permanent Representative of France to the European Communities, the French Government confirming its letter of 5 November 1970 further requested authority to apply to the carriage of iron ore from Western France, that is from the mines of Rougé, Segré and Halouse: to Belgium, the Franco-Belgian Tariff No 3232.01; to Luxembourg and the Saar, over a new extended route, Tariff No 3530.05 and the Franco-German Tariff No 3101, Part III, Scales 2 (a) and 2 (b) respectively;

Whereas the French and Luxembourg Governments base their requests on the ground that reduced tariffs would be of particular benefit to the railways in helping them to retain a heavy traffic-load; whereas, without these measures, the iron ore from the mines of Western France would not withstand the competition from richer ore transported by other

routes; whereas, therefore, it is to be feared that, as a result especially of general rate increases by the SNCF, steelmakers in Belgium, Luxembourg and the Saar would make more use of ore from other sources; whereas the trend in traffic confirms this danger, the volume of traffic to which the proposed tariff measures relate having fallen from 500 000 tons (200 000 to Belgium, 88 000 to Luxembourg and 200 000 to the Saar) in 1970 to about 330 000 tons in 1971; whereas despite the reduced tariffs it would still be profitable for the railways to carry the ore in question and thus help to improve their financial situation;

Whereas under the fourth paragraph of Article 70 of the ECSC Treaty the application of special tariffs in the interest of coal- or steel-producing undertakings requires the prior agreement of the Commission; whereas this provision refers not only to special tariffs adopted in the interest of these undertakings but to all special tariffs, whatever the reason for adopting them, which benefit one or more of these undertakings; whereas the agreement of the Commission will be granted so long as the special tariffs accord with the principles of the Treaty, and whereas it will, as a general rule, be presumed that they do so accord whenever the special tariff is warranted by the particular circumstances of the transport market; whereas, however, upon considering the application of a tariff measure, a carrier's interest in retaining a specific traffic load ought no longer to be taken into account whenever such measure would be prejudicial to the proper functioning of the common market in coal and steel in accordance with the provisions of the ECSC Treaty;

Whereas the tariff measures under consideration fall within the fourth paragraph of Article 70 of the ECSC Treaty by reason of their limited application and because they benefit the mines of Halouse, Segré and Rougé;

Whereas, as regards the interest of the railways, there exists in this case a real risk of loss of traffic already threatened by the fact that the steel undertakings can obtain their ore supplies by other routes and from other sources; whereas the keenness of the

competition is apparent from the considerable drop in sales of ore from Western France despite the tariff reductions already allowed; whereas, however, the threatened traffic is still of consequence and is profitable to the railways, which carry it under favourable conditions by whole trains, and to some extent according to prearranged schedules;

Whereas the indisputable interest which the tariff measures hold for the railways is in no way prejudicial to the proper functioning of the common market in coal and steel in accordance with the ECSC Treaty;

Whereas the competitive position of iron-ore producers of the Community is in no way affected by the tariff measures; whereas other iron ore produced in the Community cannot be substituted for the ore from Western France, which consist of different chemical substances and are mixed with other ore, particularly Lorraine ore;

Whereas the competitive position within the common market of steel undertakings in Belgium, Luxembourg and the Saar is not affected by the application of special tariffs, inasmuch as these undertakings can obtain ore by other routes on no less favourable terms;

Whereas the tariff measures have, besides, the effect of ensuring continued employment in the mining areas of Normandy and in the departments of Loire-Atlantique and Maine-et-Loire, thus keeping social disturbances at bay in regions classified, by reason of their economic structural problems, as French development areas;

Whereas in the circumstances the tariff measures are in accordance with the principles of the Treaty;

Whereas the Commission must reserve the right to review its decision whenever the circumstances giving

rise to it vary or no longer apply; whereas it feels that these measures and the authority to apply them should be reviewed within two years at most, even if no such change has taken place; whereas they should therefore be authorized for a limited period only;

HAS DECIDED AS FOLLOWS:

Article 1

Authority is given until 31 December 1974 for the application of the Franco-Belgian Tariff No 3232.01, the Franco-Luxembourg Tariff No 3530.05 and the Franco-German Tariff No 3101, Part III, Scales 2 (a) and 2 (b), to the carriage of iron ore from Western France to Belgium, Luxembourg and the Saar.

Article 2

This Decision may be amended or repealed if the Commission considers that it is no longer warranted.

Article 3

This Decision is addressed to the French Republic and, as regards its application to the Franco-Luxembourg Tariff No 3530.05, to the Grand Duchy of Luxembourg.

Done at Brussels, 4 January 1973.

For the Commission

The President

S. L. MANSHOLT

COMMISSION DECISION

of 8 February 1973

applying the provisions of Article 3 (2) (a) of Regulation (EEC) No 142/69 in favour
of two Italian sugar manufacturers

(Only the Italian text is authentic)

(73/50/EEC)

THE COMMISSION OF THE EUROPEAN
COMMUNITIES,

Having regard to the Treaty establishing the
European Economic Community;

Having regard to Commission Regulation (EEC) No
142/69 ⁽¹⁾ of 28 January 1969 laying down certain
detailed rules for the application of the quota system
for sugar, as last amended by Regulation (EEC) No
144/73 ⁽²⁾, and in particular Article 3 (3) thereof;

Whereas, by letters dated 10 September 1971
addressed to the Government of Italy, SpA
Zuccherificio del Molise, with its head office at
Bologna and a factory at Termoli (Capobasso) and
SpA Agricola Industriale Emiliana (AIE) with its head
office at Bologna and a factory at San Pietro in
Casale (Bologna) requested that, pursuant to Article
3 (2) (a) of Regulation (EEC) No 142/69, a certain
quantity of sugar produced at the factory San Pietro
in Casale under a contract to make up work between
the two undertakings should be treated as part of the
production of SpA Zuccherificio del Molise; whereas
the reason advanced by the applicants for the
application of the provisions in question was that
damages had occurred to the conveyor belt system at
the Termoli factory which could not be repaired in
the course of production;

Whereas by its Decision of 1 January 1972 the
Commission rejected the request of the two
manufacturers; whereas however this Decision was
later reversed by its Decision of 13 October 1972;

Whereas the Republic of Italy has checked the
information pursuant to Article 3 (3) of Regulation
(EEC) No 142/69; whereas, by agreement with that
Member State, the Commission has obtained further
information;

Whereas the difficulties encountered in the course of
production at the Termoli factory during the sugar
year 1971/72 appear to have been unforeseeable,
unavoidable, and in no way the fault of the principal;

Whereas, in the light of checks carried out, it appears
that the principal was obliged, because of the
difficulties abovementioned, to have a certain
quantity of beet purchased by itself processed by
another undertaking; whereas this quantity of beet
yielded 4 321 251 metric tons of white sugar;

Whereas, in these circumstances, an affirmative reply
should be given to the request of the two
manufacturers.

Whereas the measures provided for in this Decision
are in accordance with the Opinion of the
Management Committee for Sugar;

HAS ADOPTED THIS DIRECTIVE:

Article 1

The provisions of Article 3 (2) (a) of Regulation
(EEC) No 142/69 shall apply to the 4 321 251 metric
tons of white sugar produced under the contract to
make up work between SpA Zuccherificio del Molise,
as principal, and SpA Agricola Industriale Emiliana,
as processor, as notified by each one of them in their
letters to the Government of Italy of 10 September
1971.

Article 2

This Decision is addressed to SpA Zuccherificio del
Molise and SpA Agricola Industriale Emiliana, both
with head offices at Bologna, and to the Republic of
Italy.

Done at Brussels, 8 February 1973.

For the Commission

The President

François-Xavier ORTOLI

⁽¹⁾ OJ No L 20, 27. 1. 1969, p. 1.

⁽²⁾ OJ No L 18, 23. 1. 1973, p. 13.

COMMISSION DECISION

of 8 February 1973

amending the Commission Decision of 22 December 1972 authorizing the Federal Republic of Germany to sell butter at reduced price in the form of concentrated butter

(Only the German text is authentic)

(73/51/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community;

Having regard to Council Regulation (EEC) No 804/68 ⁽¹⁾ of 27 June 1968 on the common organization of the market in milk and milk products, as last amended by the Act ⁽²⁾ annexed to the Treaty ⁽³⁾ concerning 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 6 (7) thereof;

Having regard to Council Regulation (EEC) No 985/68 ⁽⁴⁾ of 15 July 1968 laying down general rules for intervention on the market in butter and cream, as last amended by Regulation (EEC) No 2714/72 ⁽⁵⁾, and in particular Article 7a thereof;

Whereas the Commission Decision of 22 December 1972 ⁽⁶⁾ authorizing the Federal Republic of Germany to sell butter at reduced price in the form of concentrated butter, fixed the quantity of butter to be processed by that Member State at 6 000 metric tons, taking account of the possibilities of disposal fixed on the basis of previous action;

Whereas because of the delay in implementing this measure and because its application is limited to 31

March 1973, the initial quantity provided for should be altered;

Whereas the measures provided for in this Decision are in accordance with the Opinion of the Management Committee for Milk and Milk Products;

HAS ADOPTED THIS DECISION:

Article 1

The expression '6 000 metric tons' appearing in Article 1 of the Commission Decision of 22 December 1972 is replaced by '4 000 metric tons'.

Article 2

This Decision is addressed to the Federal Republic of Germany.

Done at Brussels, 8 February 1973.

For the Commission

The President

François-Xavier ORTOLI

⁽¹⁾ OJ No L 148, 28. 6. 1968, p. 13.

⁽²⁾ OJ No L 73, 27. 3. 1972, p. 14.

⁽³⁾ OJ No L 73, 27. 7. 1972, p. 5.

⁽⁴⁾ OJ No L 169, 18. 7. 1968, p. 1.

⁽⁵⁾ OJ No L 291, 28. 12. 1972, p. 15.

⁽⁶⁾ OJ No L 303, 31. 12. 1972, p. 41.

COMMISSION DECISION

of 21 February 1973

discontinuing the twenty-third partial invitation to tender for white sugar issued under Regulation (EEC) No 1897/72

(73/52/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community;

Having regard to Council Regulation No 1009/67/EEC ⁽¹⁾ of 18 December 1967 on the common organization of the market in sugar, as last amended by Regulation (EEC) No 174/73 ⁽²⁾, and in particular Article 17 (4) thereof;

Whereas Commission Regulation (EEC) No 1897/72 ⁽³⁾ of 1 September 1972 on a standing invitation to tender to determine the export refund for white sugar, as last amended by Regulation (EEC) No 2847/72 ⁽⁴⁾, requires Member States to issue partial invitations to tender for the export of white sugar;

Whereas, pursuant to Article 4 (3) of Council Regulation (EEC) No 766/68 ⁽⁵⁾ of 18 June 1968 laying down general rules for granting export refunds on sugar, as last amended by Regulation (EEC) No 433/72 ⁽⁶⁾ the maximum amount of the refund is fixed for partial invitations to tender within three working days following the expiry of the time limit for the submission of tenders; whereas, when the maximum amount of the refund is being calculated, account must be taken of the supply situation and prices within the Community, prices and potential outlets on the world market and costs incurred in exporting sugar; whereas, however, pursuant to

Article 6 (1) of Commission Regulation (EEC) No 394/70 ⁽⁷⁾ of 2 March 1970 on detailed rules for granting export refunds on sugar, a decision may be taken to discontinue the tender;

Whereas the measures provided for in this Decision are in accordance with the Opinion of the Management Committee for Sugar;

HAS ADOPTED THIS DECISION:

Article 1

The twenty-third partial invitation to tender issued under Regulation (EEC) No 1897/72, the time limit for the submission of tenders in response to which expired on 21 February 1973, is hereby discontinued.

Article 2

This Decision is addressed to the Member States.

Done at Brussels, 21 February 1973.

For the Commission

P. J. LARDINOIS

Member of the Commission

⁽¹⁾ OJ No 308, 18. 12. 1967, p. 1.

⁽²⁾ OJ No L 25, 30. 1. 1973, p. 1.

⁽³⁾ OJ No L 201, 2. 9. 1972, p. 8.

⁽⁴⁾ OJ No L 299, 31. 12. 1972, p. 4.

⁽⁵⁾ OJ No L 143, 25. 6. 1968, p. 6.

⁽⁶⁾ OJ No L 53, 2. 3. 1972, p. 1.

⁽⁷⁾ OJ No L 50, 4. 3. 1970, p. 1.

COMMISSION DECISION

of 26 February 1973

concerning protective measures to be applied by the Member States, against swine vesicular disease

(73/53/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community;

Having regard to the Council Directive of 26 June 1964 ⁽¹⁾ on animal health problems affecting intra-Community trade in bovine animals and swine, as last amended by the Council Directive of 7 February 1972 ⁽²⁾, and in particular Article 9 thereof;

Having regard to the Council Directive of 26 June 1964 ⁽³⁾ on health problems affecting intra-Community trade in fresh meat, as last amended by the Council Directive of 27 October 1970 ⁽⁴⁾, and in particular Article 8 thereof;

Whereas the situation created by the outbreak of swine vesicular disease in parts of the Community could endanger Community pig stock; whereas consequently the Member States were fully justified in adopting certain emergency protective measures;

Whereas however, at the present time the seriousness of the disease and the speed with which it is spreading are not such as to justify import restrictions, even partial; whereas it is sufficient if the dispatching Member States in which there is an outbreak of swine vesicular disease grant the receiving Member States guarantees which are identical to more provided for by existing Community rules on foot-and-mouth disease, in order to prevent the disease from spreading; whereas in fact there is a certain similarity between the clinical appearance of the first symptoms of vesicular disease and of foot-and-mouth disease;

Whereas in implementation of the Community provisions referred to above, the measures to be applied by the countries concerned should be coordinated to this effect;

Whereas the measures provided for in this Decision are in accordance with the Opinion of the Standing Veterinary Committee;

HAS ADOPTED THIS DECISION:

Article 1

Member States in which there has been an outbreak of swine vesicular disease shall grant guarantees in respect of pigs and fresh pigmeat dispatched from their territories to the territory of another Member State, which are identical to those established for foot-and-mouth-disease by the Council Directive of 26 June 1964 on animal health problems affecting intra-Community trade in bovine animals and swine as last amended by the Council Directive of 7 February 1972, and in particular Articles 2 (i), 3 (2) (b) and 2 (c) (i) (ii) thereof.

Article 2

This Decision is addressed to the Member States.

Done at Brussels, 26 February 1973.

For the Commission

The President

François-Xavier ORTOLI

⁽¹⁾ OJ No 121, 29. 7. 1964, pp. 1977 and 2012/64.

⁽²⁾ OJ No L 38, 12. 2. 1972, p. 95.

⁽³⁾ OJ No 121, 29. 7. 1964, p. 2012/64.

⁽⁴⁾ OJ No L 239, 30. 10. 1970, p. 42.