COMMISSION RECOMMENDATION

of 6 February 1987

on quantitative methods of analysis for the identification of acrylic and modacrylic fibres, chlorofibres and trivinyl fibres

(87/185/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community, and in particular the second indent of Article 155 thereof,

Whereas Annex I to Council Directive 71/307/EEC of 26 July 1971 on the approximation of the laws of the Member States relating to textile names [1], as last amended by Directive 83/623/EEC [2], which requires mandatory labelling for textile products, sets out the fibre names and descriptions; whereas the fibres making up these products and named on the label must conform with those descriptions;

Whereas that Annex specifies that fibres referred to in items 24, 25, 27 and 35 must have in the chain specific percentages of a given monomer pattern, which is the only criterion allowing them to be identified and in some cases distinguished from each other;

Whereas during verification of the conformity of textile products with the composition stated on the label it may prove necessary to check these percentages in order to identify the abovementioned fibres; whereas for that purpose analytical laboratories must have suitable methods for the quantitative determination of nitrogen and chlorine making up the monomer patterns of those chemical fibres;

Whereas in the current state of the art it is not always possible to determine the quantities of nitrogen or chlorine inherent exclusively in the abovementioned monomer patterns, since such quantities can be determined only for the product as a whole, including any proportion of the substances due to the presence in the fibre of matter such as colouring agents or additives containing nitrogen and chlorine; whereas under these circumstances it is not possible to lay down binding methods of analysis;

Whereas it is nevertheless expedient, in order to ensure the necessary uniformity of the results of checks on the conformity of textile products carried out in the Community, to provide laboratories with methods for the quantitative determination of nitrogen and chlorine that are as precise as possible, with due regard to the current state of the art, and which those laboratories may use; whereas such methods have been drawn up on the basis of numerous interlaboratory tests carried out under the auspices of the Commission; whereas their use should therefore be recommended in accordance with the views expressed by the Committee for Directives relating to Textile Names and Labelling,

HEREBY FORMULATES THIS RECOMMENDATION:

Article 1

It is hereby recommended that the laboratories conducting verifications of the conformity of the composition of textile products use, for the identification of the fibres referred to in items 24, 25, 27 and 35 of Annex I to Directive 71/307/EEC, the methods for quantitative analysis given in the Annex to this recommendation.

Article 2

The Member States shall inform the Commission of the measures taken on the basis of this recommendation.

Article 3

This recommendation is addressed to the Member States.

Done at Brussels, 6 February 1987.

For the Commission

Grigoris VARFIS

Member of the Commission

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[1] OJ No L 185, 16. 8. 1971, p. 16.
[2] OJ No L 353, 15. 12. 1983, p. 8.

ANNEX

RECOMMENDED QUANTITATIVE ANALYSIS METHODS FOR THE IDENTIFICATION OF ACRYLIC AND MODACRYLIC FIBRES AND CHLOROFIBRES AND TRIVINYL FIBRES

Note

The methods given in A and B below make it possible to verify the permitted percentages of monomer patterns for acrylic and modacrylic fibres and chlorofibres and trivinyl fibres under items 24 and 27 and 25 and 35 respectively in Annex I to Directive 71/307/EEC.

However, these methods should not be considered as the only means of verifying the conformity of the above fibres with their descriptions. Should any doubt arise, the results obtained with these methods may be checked by other suitable techniques, especially when the results are on the limits for the relevant fibre.

A. METHOD FOR THE QUANTITATIVE DETERMINATION OF NITROGEN FOR THE IDENTIFICATION OF ACRYLIC AND MODACRYLIC FIBRES REFERRED TO IN ITEMS 24 AND 27 OF ANNEX I TO DIRECTIVE 71/307/EEC

1. Field of application

This method can be used after removal of the non-fibrous matter, for the qualitative determination of acrylic and modacrylic fibres in the pure state and after separation from a mixture.

The method can only be applied when additives, and in particular nitrogenous substances can be completely removed beforehand: dyes are considered as additives here, in derogation of Council Directive 72/276/EEC [1] on the analysis of binary mixtures.

2. Principle

The nitrogen content is determined and the percentage by mass of the acrylonitrile unit present in the fibre is calculated by means of a conversion factor.

3. Apparatus and reagents

3.1. *Apparatus*

(i) Kjeldahl digestion flask, 500 ml capacity

(ii) Kjeldahl distillation apparatus

(iii) Titration apparatus, allowing precision of 0,05 ml.

3.2. *Reagents*

All reagents used must be analytical grade and the water must be distilled.

(i) Anhydrous potassium sulphate

(ii) Copper sulphate ($CuSO_4 . 5H_20$)

(iii) Concentrated sulphuric acid, (d = 1,84 at 20 °C)

(iv) Sulphuric acid 0,1 N (standard solution)

(v) Sodium hydroxide solution 400 g/l: dissolve 400 grams of sodium hydroxide in 400/500 ml of water and bring up to a volume of 1 litre with water

(vi) Sodium hydroxide solution 0,1 N (standard solution)

(viii) Phenolphthalein solution, as indicator.

4. Procedure

As regards sampling, drying, weighing and pretreatment, follow the instructions given in Section 1 'General information of methods for the quantitative chemical analysis of textile fibre mixtures' of Directive 72/276/EEC.

[1] OJ No L 173, 31. 7. 1972.

Transfer aproximately 0,5 g of the dry sample, accurately weighed, into the Kjeldahl flask and add 10 grams of potassium sulphate, 1 gram of copper sulphate and 25 ml of concentrated sulphuric acid (density : 1,84). Gently swirl the contents of the flask until all the fibres are thoroughly wetted out by the acid.

Fix the flask in an inclined position under a suction hood and reheat slowly and carefully over a bunsen burner or other heating apparatus until the fibres are destroyed. Then increase the heat in order to keep the solution simmering (approximately 350 °C) for 30 minutes, i. e. until the solution becomes almost colourless.

Allow the flask to cool and carefully dilute the contents with 150 ml water.

Pour about 100 ml of the 0,1 N sulphuric acid solution into a 250 ml conical flask which is placed beneath the condenser so that the outlet of the delivery tube is just below the level of the liquid.

Connect the Kjeldahl flask to the distillation apparatus and add 120 ml of sodium hydroxide solution (400 g/l) slowly and carefully.

Heat to simmering and collect at least 100 ml of the distillate so as to recover the entire quantity of ammonia.

After distillation lower the conical flask so that the outside of the condenser is approximately 20 mm above the level of the liquid and distill for a further minute.

Wash the outside of the tube with distilled water and collect the washing liquid in the concial flask.

Titrate the distillate with the standardized 0,1 N sodium hydroxide solution, using the phenolphthalein solution as an indicator.

5. **Calculation and expression of the results**

The percentage of nitrogen in the sample, when dry, is calculated by :

$$A\,\% = \frac{14\,(V_1 n_1 - V_2 n_2)}{10\,.\,m}$$

where

$A\,\%$ = percentage of nitrogen in the dry and pure sample ;

V_1 = volume in millilitres of sulphuric acid solution 0,1 N ;

n_1 = normality of sulphuric acid solution ;

V_2 = volume in millilitres of sodium hydroxide solution 0,1 N ;

n_2 = normality of sodium hydroxide solution ;

m = mass in grams of the pretreated dried test specimen.

Calculation of the acrylonitrile monomer unit to the first decimal place is by the formula :

— acrylonitrile unit % = $A\,\% \times 3{,}788$.

6. **Precision of the method**

The confidence limits of the results obtained by this method are not greater than ± 1 for a confidence level of 95 %.

B. METHOD FOR THE QUANTITATIVE DETERMINATION OF CHLORINE FOR THE IDENTIFICATION OF CHLOROFIBRES AND TRIVINYL FIBRES REFERRED TO IN ITEMS 25 AND 35 OF ANNEX I TO DIRECTIVE 71/307/EEC

1. **Field of application**

This method can be used, after removal of non-fibrous matter, for the identification of chlorofibres and trivinyl fibres in the pure state and after separation from a mixture.

The method is only applicable when additives, and in particular chlorates, can be completely removed beforehand : dyes are considered as additives here, in derogation of Directive 72/276/EEC on the analysis of binary mixtures

2. **Principle**

After combustion of the sample in an oxygen atmosphere, the chlorine content is determined and the percentage by mass of the vinyl or vinylidene chloride is calculated by means of conversion factors.

3. Apparatus and reagents

3.1. *Apparatus*

(i) Approximately 500 ml open-necked combustion flask in borosilicate glass or similar material fitted with a ground borosilicate glass stopper to which a platinum wire 0,5-0,7 mm in diameter and around 100 mm in length is welded (figure 1).

The flask and the stopper must be fitted with a device to ensure that the stopper is kept in place and thus prevent losses of gas during combustion.

(ii) Device for remote ignition of the material (infrared recommended).

(iii) Filter paper with low chlorine and ash content.

3.2. *Reagents*

All reagents used must be analytical grade and the water must be distilled.

(i) Potassium hydroxide 0,01 N (standard solution)

(ii) 30 % hydrogen peroxide (m/m)

(iii) Oxygen cylinder

(iv) Perchloric acid (70 % m/m ; d = 1,67)

(v) 0.1 % diphenyl carbazone alcoholic solution (m/m)

(vi) Mercury (II) perchlorate 0,01 N solution standardized with primary standard sodium chloride. The solution is prepared by dispersing, by means of shaking 1,1 grams of mercury oxide (yellow) in 800 ml of water and adding 1,54 ml of 70 % perchloric acid ; this is brought up to a volume of 1 000 ml and stadardized with sodium chloride solution, of known titre.

4. Procedure

For sampling, preliminary treatment, drying and weighing follow the instructions given under section I 'General information on the methods for the quantitative chemical analysis of textile fibre mixtures' of Directive 72/276/EEC.

Weigh accurately a dry sample of about 30 mg and place it on the filter paper which is folded (along the lines traced in Figure 2) and rolled up from bottom to top.

Wrap the platinum wire attached to the stopper tightly round the paper package containing the sample in such a way that the ignition 'touchpaper' protrudes freely.

Pour into the flask 10 ml of water, 20 ml of potassium hydroxide 0,01 N solution and 3-4 drops of hydrogen peroxide.

Fill the flask with oxygen for a few seconds until it is full. Light the 'touch-paper', stopper the flask immediately and secure the stopper.

Allow to cool, shake the flask vigorously several times over a period of 25-30 minutes so as to accelerate the absorption of the combustion products.

Fill the neck of the flask with a few millilitres of distilled water and then remove the stopper. Wash the platinum wire and the walls of the flask with 50-60 ml of water.

Boil the alkaline solution for 3-5 minutes and bring the solution to a pH of around 3,5 with 70 % perchloric acid.

Add 2 ml of the diphenyl carbazone indicator and titrate with 0,01 N mercury perchlorate solution until the colour changes from colourless to lilac.

Carry out control titration using the same quantities of reagent and filter paper used in the test.

(Instead of titration with mercury (II) perchlorate and chromatic indicator, potentiometric titration using a silver electrode with a silver nitrate solution of known titre may be employed).

5. **Calculation and expression of the results**

The percentage of chlorine in the fibre in question, when dry, is calculated by

$$Cl\ \% = \frac{(A-B) \times n \times 35{,}46 \times 100}{m}$$

where

Cl = chlorine content in the dry and pure sample ;

A = millilitres of mercury perchlorate 0,01 N solution consumed during titration of the sample ;

B = millilitres of mercury perchlorate 0,01 N solution consumed during titration of the control test ;

n = the normality of the mercury perchlorate solution ;

m = mass in milligrams of the pretreated dried test specimen.

The percentage of vinyl or vinidylene chloride monomers, approximated to the first decimal place, is calculated by one of the following formulae :

— vinyl chloride percentage = Cl % × 1,762

— vinylidene chloride percentage = Cl % × 1,367.

6. **Precision of the method**

The confidence limits of the results of the chlorine percentage obtained by this method are not greater than ± 0,5 for a confidence level of 95 %.

7. **Precautions**

— The combustion flask must not contain residue or fumes of organic solvent.

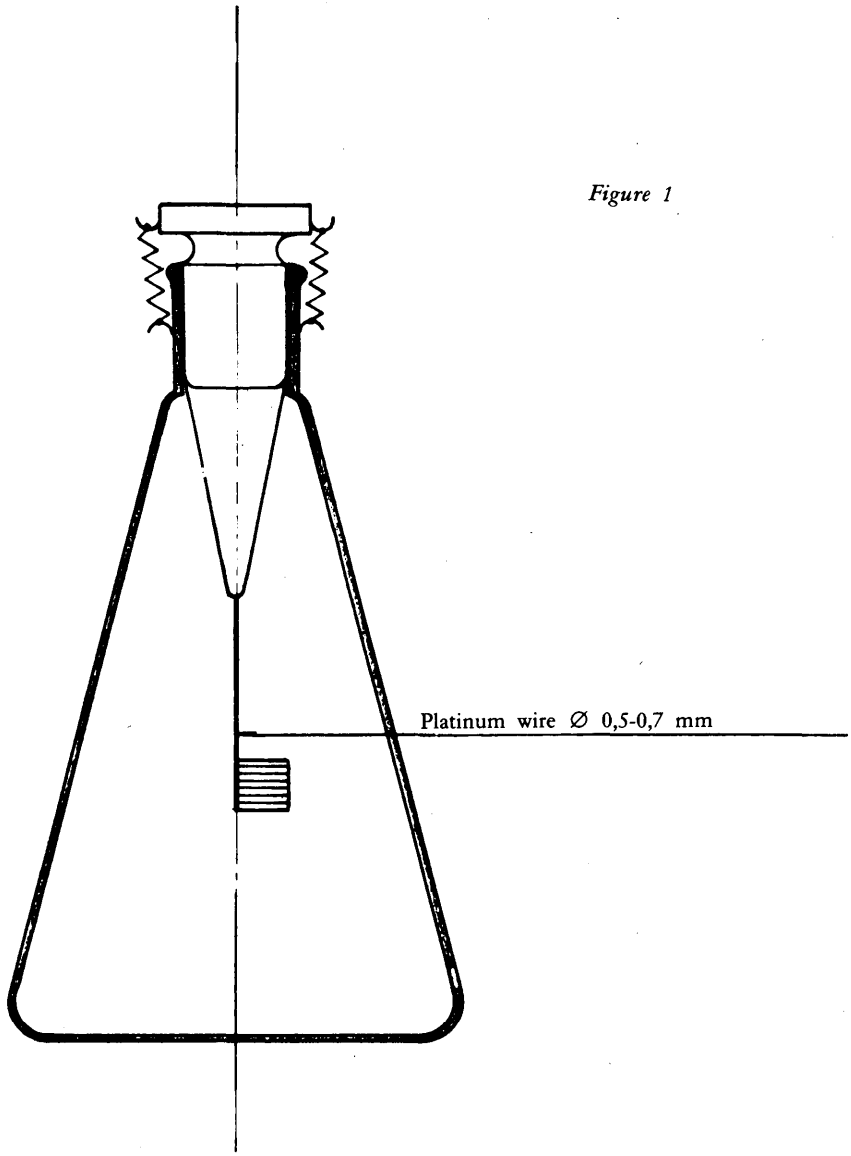
— Care must be taken when operating manually to avoid operator accidents caused by flask explosions.

— Once the sample is enclosed in the flask, the flask itself must be properly closed since the first stage of combustion produces high pressure.

Figure 1

Conical flask Schöninger

of 500 ml



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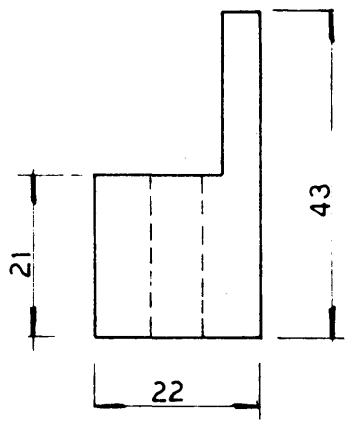

Figure 2