II

(Acts whose publication is not obligatory)

COMMISSION

COMMISSION DIRECTIVE

of 6 February 1987

amending Annex II to Council Directive 72/276/EEC on the approximation of the laws of the Member States relating to certain methods for the quantitative analysis of binary textile fibre mixtures

(87/184/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard 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], and in particular Article 15a (2) thereof,

Whereas Directive 71/307/EEC requires labelling to indicate the nature of the fibres in textile products, checks on the conformity of these products with the indications given on the label being carried out by analysis;

Whereas pursuant to Article 13 (2) of the above Directive, Annex II to Council Directive 72/276/EEC [3], as last amended by Directive 81/75/EEC [4], lays down 15 uniform methods of analysis for most of the textile products composed of binary mixtures that are on the market;

Whereas in the light of experience gained gradually by national laboratories and the results of interlaboratory tests carried out under the auspices of the Commission, it is necessary:

— to redraft method No 2 by the incorporation of the use of a stable and easily prepared additional reagent,

— to amend certain points in method No 8 so as to simplify the test procedures and ensure uniform results,

— to delete method No 12, which has proved to be insufficiently accurate;

Whereas textile products consisting of mixtures of chlorofibre, certain modacrylics, certains elastanes, acetate, triacetate and certain other fibres which are also subject to the labelling obligation stipulated in Directive 71/307/EEC are not covered by Directive 72/276/EEC; whereas uniform methods of analysis applicable to these products should therefore be established;

Whereas the provisions of this Directive are in accordance with the opinion of the Committee for Directives relating to Textile Names and Labelling,

HAS ADOPTED THIS DIRECTIVE:

Article 1

Section 2 of Annex II to Directive 72/276/EEC is hereby amended as follows:

1. Special methods Nos 2, 8 and 12 are amended in accordance with Annex I to this Directive.

2. Special method No 16 in Annex II to this Directive is hereby added.

[1] OJ No L 185, 16. 8. 1971, p. 16.
[2] OJ No L 353, 15. 12. 1983, p. 8.
[3] OJ No L 173, 31. 7. 1972, p. 1.
[4] OJ No L 57, 4. 3. 1981, p. 23.

Article 2

1. Member States shall bring into force the provisions necessary to comply with this Directive not later than 1 September 1988, and shall forthwith inform the Commission thereof.

2. As soon as this Directive has been notified, Member States shall also ensure that the Commission is informed, in sufficient time for it to submit its comments, of any draft laws, regulations or administrative provisions which they intend to adopt in the field covered by the Directive.

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 6 February 1987.

For the Commission

Grigoris VARFIS

Member of the Commission

ANNEX I

Amendments to section 2 ('Special Methods') of Annex II to Directive 72/276/EEC

Method No 2:

The text is replaced by the following:

'METHOD No 2

CERTAIN PROTEIN FIBRES AND CERTAIN OTHER FIBRES

(Method using hypochlorite)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. certain protein fibres, namely: wool (1), animal hair (2 and 3), silk (4), protein (21)

with

2. cotton (5), cupro (19), modal (20), viscose (23), acrylic (24), chlorofibres (25), polyamide or nylon (28), polyester (29), polypropylene (31), elastane (37) and glass fibre (38).

If different protein fibres are present, the method gives the total of their amounts but not their individual quantities.

2. PRINCIPLE

The protein fibre is diseolved out from a know dry mass of the mixture, with a hypochlorite solution. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of dry protein fibre is found by difference.

Either lithium hypochlorite or sodium hypochlorite can be used for the preparation of the hypochlorite solution.

Lithium hypochlorite is recommended in cases involving a small number of analyses or for analyses conducted at fairly lengthy intervals. This is because the percentage of hypochlorite in solid lithium hypochlorite — unlike that in sodium hypochlorite — is virtually constant. If the percentage of hypochlorite is known, hypochlorite content need not be checked iodometrically for each analysis, since a constant weighed portion of lithium hypochlorite can be employed.

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1. **Apparatus**

(i) Erlenmeyer flask with ground-glass stopper, 250 ml;

(ii) Thermostat, adjustable to 20 (± 2) °C.

3.2. **Reagents**

(i) Hypochlorite reagent

(a) Lithium hypochlorite solution

This consists of a freshly prepared solution containing 35 (± 2) g/l of active chlorine (approximately 1 M), to which 5 (± 0,5) g/l of previously dissolved sodium hydroxide is added. To prepare, dissolve 100 grams of lithium hypochlorite containing 35 % active chlorine (or 115 grams containing 30 % active chlorine) in approximately 700 ml of distilled water, add 5 grams of sodium hydroxide dissolved in approximately 200 ml of distilled water and make up to 1 litre with distilled water. The solution which has been freshly prepared needs not be checked iodometrically;

(b) Sodium hypochlorite solution

This consists of a freshly prepared solution containing 35 (± 2) g/l of active chlorine (approximately 1 M) to which 5 (± 0,5) g/l of previously dissolved sodium hydroxide is added.

Check the active chlorine content of the solution iodometrically before each analysis;

(ii) *Acetic acid, dilute solution*

Dilute 5 ml of glacial acetic acid to 1 litre with water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows: mix approximately 1 gram of the sample with approximately 100 ml of the hypochlorite solution (lithium or sodium hypochlorite) in the 250 ml flask and agitate thoroughly in order to wet out the sample.

Then heat the flask for 40 minutes in a thermostat at 20 °C and agitate continuously, or at least at regular intervals. Since the dissolution of the wool proceeds exothermically, the reaction heat of this method must be distributed and removed. Otherwise, considerable errors may be caused by the incipient dissolution of the non-soluble fibres.

After 40 minutes, filter the flask contents through a weighed glass-filter crucible and transfer any residual fibres into the filter crucible by rinsing the flask with a little hypochlorite reagent. Drain the crucible with suction and wash the residue successively with water, dilute acetic acid, and finally water, draining the crucible with suction after each addition. Do not apply suction until each washing liquor has drained under gravity.

Finally, drain the crucible with suction, dry the crucible with the residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d is 1,00, except for cotton, viscose and modal, for which d = 1,01, and unbleached cotton, for which d = 1,03.

6. PRECISION

On homogenous mixtures of textile materials, the confidence limits for results obtained by this method are not greater than ± 1 for a confidence level of 95 %.'

Method No 8

The text is hereby amended as follows :

In item 4 ('Test procedure'), the 5th, 6th and 7th paragraphs read as follows :

'Transfer any residual fibre to the crucible by washing out the beaker with dimethylformamide. Drain the crucible with suction. Wash the residue with about 1 litre of hot water at 70 — 80 °C, filling the crucible each time. After each addition of water, apply suction briefly but not until the water has drained under gravity. If the washing liquor drains through the crucible too slowly, slight suction may be applied.

Finally dry the crucible with the residue, cool and weigh them.'

Item 5 ('Calculation and expression of results') reads as follows :

'Calculate the results as described in the general instructions. The value of "d" is 1,00 except in the following cases :

wool	1,01
cotton	1,01
cupro	1,01
modal	1,01
polyester	1,01.'

Method No 12

This method is deleted.

ANNEX II

METHOD No 16

CHLOROFIBRES, CERTAIN MODACRYLICS, CERTAIN ELASTANES, ACETATES, TRIACETATES AND CERTAIN OTHER FIBRES

(Method using cyclohexanone)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of

1. acetate (17), triacetate (22), chlorofibre (25), certain modacrylics (27), certain elastanes (37)

with

2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (19), modal (20), viscose (23), polyamide or nylon (28), acrylic (24) and glass fibre (38).

Where modacrylics or elastanes are present a preliminary test must first be carried out to determine whether the fibre is completely soluble in the reagent.

It is also possible to analyze mixtures containing chlorofibres by using method No 9 or 15.

2. PRINCIPLE

The acetate and triacetate fibres, chlorofibres, certain modacrylics, and certain elastanes are dissolved out from a known dry mass with cyclohexanone at a temperature close to boiling point. The residue is collected, washed, dreid and weighed ; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of chlorofibre, modacrylic, elastane, acetate and triacetate is found by difference.

3. APPARATUS AND REAGENTS (other than those described in the general instructions)

3.1. **Apparatus**

(i) Hot extraction apparatus suitable for use in the test procedure in section 4. (See figure : this is a variant of the apparatus described in *Melliand Textilberichte* 56 (1975) 643-645) ;

(ii) Filter crucible to contain the specimen ;

(iii) Porous baffle (porosity grade 1) ;

(iv) Reflux condenser that can be adapted to the distillation flask ;

(v) Heating device.

3.2. **Reagents**

(i) Cyclohexanone, boiling point 156 °C ;

(ii) Ethyl alcohol, 50 % by volume.

NB: Cyclohexanone is flammable and toxic. Suitable precautions must be taken in its use.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and then proceed as follows :

Pour into the distillation flask 100 ml of cyclohexanone per gram of material, insert the extraction container in which the filter crucible, containing the specimen and the porous baffle, slightly inclined, have previously been placed. Insert the reflux condenser. Bring to the boil and continue extraction for 60 minutes at a minimum rate of 12 cycles per hour. After extraction and cooling remove the extraction container, take out the filter crucible and remove the porous baffle. Wash the contents of the filter crucible three or four times with 50 % ethyl alcohol heated to about 60 °C and subsequently with 1 litre of water at 60 °C.

Do not apply suction during or between the washing operations. Allow the liquid to drain under gravity and then apply suction.

Finally dry the crucible with the residue, cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

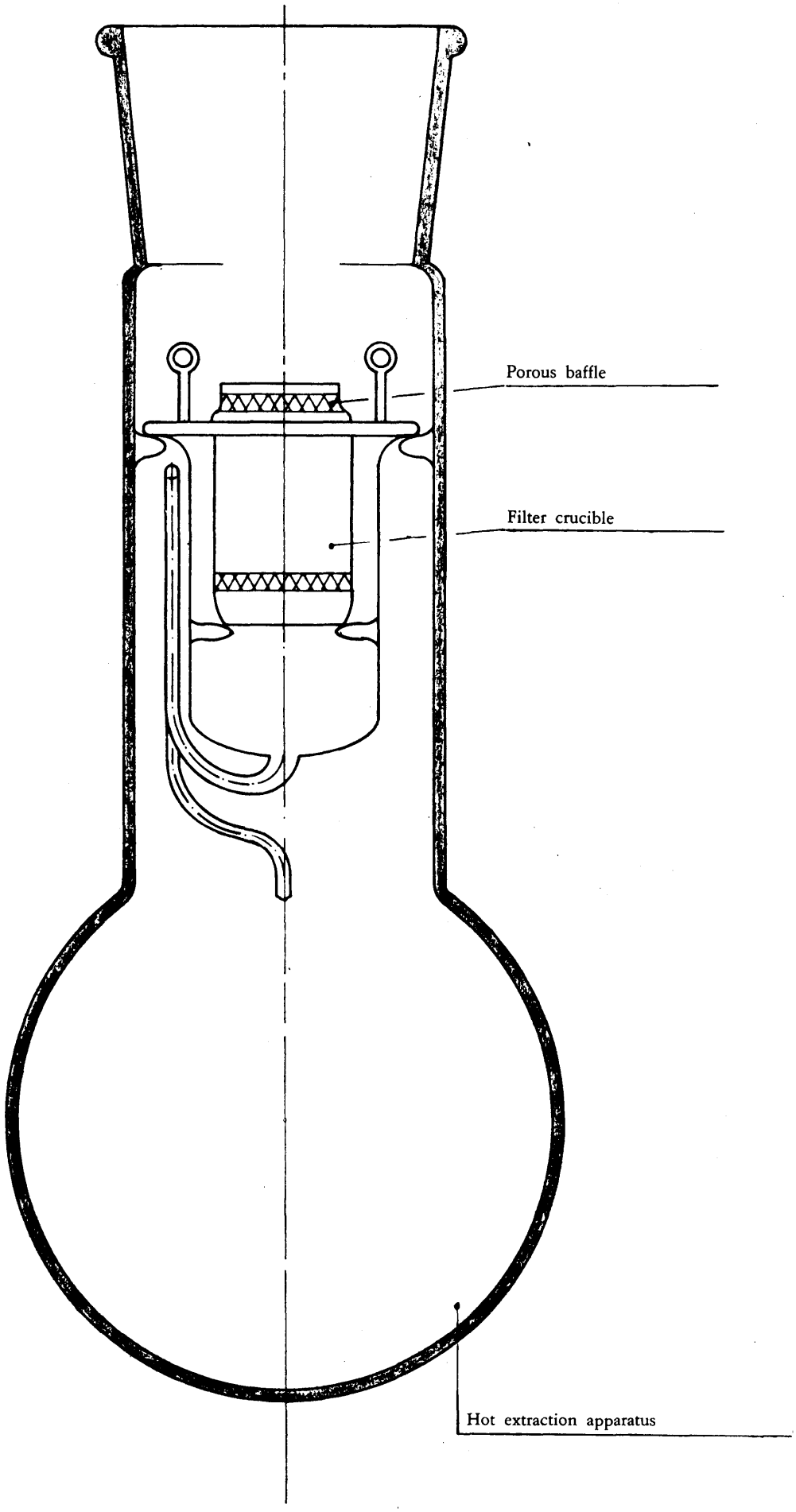
Calculate the results as described in the general instructions. The value of 'd' is 1,00 with the following exceptions :

silk 1,01

acrylic 0,98.

6. PRECISION

On homogeneous mixtures of textile fibres, the confidence limits of results obtained by this method are not greater than ± 1 for a confidence level of 95 %.


Porous baffle
Filter crucible
Hot extraction apparatus