II

(Acts whose publication is not obligatory)

COMMISSION

COMMISSION DIRECTIVE

of 4 April 1990

amending the Second Directive 82/434/EEC on the approximation of the laws of the Member State relating to methods of analysis necessary for checking the composition of cosmetic products

(90/207/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Whereas Second Commission Directive 82/434/EEC of 14 May 1982 on the approximation of the laws of the Member States relating to methods of analysis for checking the composition of cosmetic products (1) lays down a common method of analysis for the identification and determination of free formaladehyde ;

Whereas, in the light of new scientific and technical data, it has proved to be necessary to amend this method of analysis ;

Whereas the measures provided for in this Directive are in conformity with the opinion of the Committee on the Adaptation to Technical Progress of the Directives on the Removal of Technical Barriers to Trade in the Cosmetic Products sector,

HAS ADOPTED THIS DECISION :

Article 1

Chapter IV of the Annex to Directive 82/434/EEC shall be replaced by the text set out in the Annex hereto.

Article 2

Member States shall bring into force the laws, regulations or administrative provisions necessary to comply with this Directive not later than 31 December 1990. They shall forthwith inform the Commission thereof.

The provisions adopted under the first paragraph shall refer expressly to this Directive.

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 4 April 1990.

For the Commission

Karel VAN MIERT

Member of the Commission

(1) OJ No L 185, 30. 6. 1982, p. 1.

ANNEX

'IV. IDENTIFICATION AND DETERMINATION OF FREE FORMALDEHYDE

1. PURPOSE AND SCOPE

This method describes the identification and two determination according to the presence or not of formaldehyde donors. It is applicable to all cosmetic products.

1.1. **Identification**

1.2. **General determination by pentane-2,4-dione colorimetry**

This method applies when formaldehyde is used alone or with other preservatives that are not formaldehyde donors.

Where this is not the case, and if the result exceeds the maximum permitted concentration, the following method of confirmation must be used.

1.3. **Determination in the presence of formaldehyde donors**

In the method mentioned above (1.2), during the derivization, the formaldehyde donors split and lead to results that are too high (combined and polymerized formaldehyde).

It is necessary to separate the free formaldehyde by liquid chromatography.

2. DEFINITION

The free formaldehyde content of the sample determined according to this method is expressed as percentage by mass.

3. IDENTIFICATION

3.1. **Principle**

Free and combined formaldehyde in a sulphuric acid medium turns Schiff's reagent pink or mauve.

3.2. **Reagents**

All reagents should be of analytical purity and the water has to be demineralized.

3.2.1. Fuchsin;

3.2.2. Sodium sulphite hydrated at $7H_2O$;

3.2.3. Concentrated hydrochloric acid (d = 1,19);

3.2.4. Sulphuric acid, about 1M;

3.2.5. *Schiff's reagent:*

100 mg of fuchsin (3.2.1) is weighed into a beaker and dissolved in 75 ml of water at 80 °C. After cooling, add 2,5 g of sodium sulphite (3.2.2). Make up to 100 ml.

Use within two weeks.

3.3. **Procedure**

3.3.1. Weigh 2 g of the sample in a 10-ml beaker.

3.3.2. Add two drops of sulphuric acid (3.2.4) and 2 ml of Schiff's reagent (3.2.5). This reagent must be absolutely colourless when it is used.

Shake and leave to stand for five minutes.

3.3.3. If a pink or mauve tint is observed within the five minutes, the formaldehyde is present in excess of 0,01 % and is to be determined by the free and combined method (4) and, if necessary, by procedure (5).

4. GENERAL DETERMINATION BY PENTANE-2,4-DIONE COLORIMETRY

4.1. **Principle**

Formaldehyde reacts with pentane-2,4-dione in the presence of ammonium acetate to form 3,5-diacetyl-1,4-dihydrolutidine. This is extracted with butan-1-ol and the absorbance of the extract is measured at 410 nm.

4.2. **Reagents**

All reagents should be of analytical purity and the water has to be demineralized.

4.2.1. Anhydrous ammonium, acetate ;

4.2.2. Concentrated acetic acid, $d^{20}_4 = 1,05$;

4.2.3. Pentane-2,4-dione freshly distilled under reduced pressure 25 mm Hg 25° — it should not exhibit any absorption at 410 nm.

4.2.4. Butan-1-ol ;

4.2.5. Hydrochloric acid, 1 M ;

4.2.6. Hydrochloric acid, approximately 0,1 M ;

4.2.7. Sodium hydroxide, 1 M ;

4.2.8. Starch solution freshly prepared according to the European Pharmacopoeia (1 g/50 ml water), 2nd edition 1980, part I-VII-1-1 ;

4.2.9. 37 to 40 % w/v formaldehyde ;

4.2.10 Standard iodine solution, 0,05 M ;

4.2.11. Standard sodium thiosulphate solution, 0,1 M ;

4.2.12 *Pentane-2,4-dione reagent :*

In a 1 000 ml volumetric flask dissolve :

— 150 g ammonium acetate (4.2.1),

— 2 ml pentane-2,4-dione (4.2.3),

— 3 ml acetic acid (4.2.2).

Make up to 1 000 ml with water (pH of solution about 6,4).
This reagent must be freshly prepared ;

4.2.13. Reagent (4.2.12) without pentane-2,4-dione ;

4.2.14 *Formaldehyde-standard : stock solution*

Pour 5 g of formaldehyde (4.2.9) into a 1 000-ml volumetric flask and make up to 1 000 ml with water.

Determine the strength of this solution as follows :

Remove 10,00 ml ; add 25,00 ml of a standard iodine solution (4.2.10) and 10,00 ml of sodium hydroxide solution (4.2.7).

Allow to stand for five minutes.

Acidify with 11,00 ml of HCl (4.2.5) and determine the excess iodine with a standard sodium thiosulphate solution (4.2.11), using starch solution (4.2.8) as indicator.

1 ml of 0,05 M iodine (4.2.10) consumed is equivalent to 1,5 mg formaldehyde ;

4.2.15. *Formaldehyde-standard : diluted solution*

Dilute the formaldehyde stock solution successively 1/20 and then 1/100 with water.

1 ml of this solution contains about 1 µg of formaldehyde.

Calculate the exact content.

4.3. **Apparatus**

4.3.1. Standard laboratory apparatus ;

4.3.2. Phase separation filter, Whatman 1 PS (or equivalent) ;

4.3.3. Centrifuge ;

4.3.4. Water-bath set at 60 °C ;

4.3.5. Spectrophotometer ;

4.3.6. Glass cells with an optical path of 1 cm.

4.4. **Procedure**

4.4.1. *Sample solution*

Into a 100-ml volumetric flask weigh to within 0,001 g a quantity (in g) of the test sample corresponding to a presumed quantity of formaldehyde of about 150 µg.

Make up to 100 ml with water and mix (solution S).

(Check that the pH is close to 6 ; if not, dilute in the hydrochloric acid solution (4.2.6).)