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SECOND COMMISSION DIRECTIVE

of 14 May 1982

on the approximation of the laws of the Member States relating to methods of analysis necessary for checking the composition of cosmetic products

(82/434/EEC)

(OJ L 185, 30.6.1982, p. 1)

Amended by:

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SECOND COMMISSION DIRECTIVE

of 14 May 1982

on the approximation of the laws of the Member States relating to methods of analysis necessary for checking the composition of cosmetic products

(82/434/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 76/768/EEC of 27 July 1976 on the approximation of the laws of the Member States relating to cosmetic products [1], as amended by Directive 79/661/EEC [2], and in particular Article 8 (1) thereof,

Whereas Directive 76/768/EEC provides for the official testing of cosmetic products with the aim of ensuring that the conditions laid down by Community provisions concerning the composition of cosmetic products are satisfied;

Whereas all the necessary methods of analysis should be drawn up as quickly as possible; whereas, the first step towards the attainment of this objective having already been taken by the definition of certain methods in Commission Directive 80/1335/EEC [3], the second step is to consist in the definition of methods for identification of some oxidizing agents and determination of hydrogen peroxide in cosmetic hair-care products, identification and semi-quantitative determination of certain oxidation colorants in hair dyes, identification and determination of nitrite, identification and determination of free formaldehyde, determination of resorcinol in shampoos and hair lotions, and determination of methanol in relation to ethanol or propan-2-ol;

Whereas the measures laid down in this Directive are in accordance with the opinion of the Committee on the adaptation of Directive 76/768/EEC to technical progress,

HAS ADOPTED THIS DIRECTIVE:

Article 1

Member States shall take all necessary steps to ensure that, during official testing of cosmetic products:

— identification of oxidizing agents and determination of hydrogen peroxide in hair-care products,
— identification and semi-quantitative determination of certain oxidation colorants in hair dyes,
— identification and determination of nitrite,
— identification and determination of free formaldehyde,
— determination of resorcinol in shampoos and hair lotion,
— determination of methanol in relation to ethanol or propan-2-ol

are performed in accordance with the methods described in the Annex.

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 1983.

They shall forthwith inform the Commission thereof.

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(1) OJ No L 262, 27. 9. 1976, p. 169.
(2) OJ No L 192, 31. 7. 1979, p. 35.
(3) OJ No L 383, 31. 12. 1980, p. 27.

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Article 3

This Directive is addressed to the Member States.

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ANNEX

I. IDENTIFICATION OF OXIDIZING AGENTS AND DETERMINATION OF HYDROGEN PEROXIDE IN HAIR-CARE PRODUCTS

PURPOSE AND SCOPE

The iodometric determination of hydrogen peroxide in cosmetics is only possible in the absence of other oxidizing agents that form iodine from iodides. Consequently, before the iodometric determination of hydrogen peroxide it is necessary to detect and identify any other oxidizing agents present. This identification breaks down into two stages; the first covers the persulphates, the bromates and hydrogen peroxide and the second covers barium peroxide.

A. IDENTIFICATION OF PERSULPHATES, BROMATES AND HYDROGEN PEROXIDE

1. PRINCIPLE

Sodium persulphate, potassium persulphate and ammonium persulphate; potassium bromate, sodium bromate and hydrogen peroxide — whether or not originating from barium peroxide — are identified by means of descending paper chromatography, use being made of two developing solvent.

2. REAGENTS

All reagents should be of analytical purity.

2.1. 0·5 % (m/v) aqueous reference solutions of the following compounds:

2.1.1. Sodium persulphate

2.1.2. Potassium persulphate

2.1.3. Ammonium persulphate

2.1.4. Potassium bromate

2.1.5. Sodium bromate

2.1.6. Hydrogen peroxide

2.2. Developing solvent A, 80 % (v/v) ethanol

2.3. Developing solvent B, benzene — methanol — 3-methyl butan-1-ol — water (34:38:18:10 by vol)

2.4. Detecting agent A, 10 % (m/v) aqueous solution of potassium iodide

2.5. Detecting agent B, 1 % (m/v) aqueous solution of starch

2.6. Detecting agent C, 10 % (m/m) hydrochloric acid

2.7. 4N hydrochloric acid

3. APPARATUS AND EQUIPMENT

3.1. Chromatography paper (Whatman paper No 3 and No 4 or their equivalents)

3.2. Micropipette, 1 µl

3.3. Standard flasks, 100 ml

3.4. Fluted filters

3.5. Apparatus for descending paper chromatography

4. SAMPLE PREPARATION

4.1. **Water soluble products**

Make two solutions of each sample by dissolving 1 g and 5 g of the product respectively in 100 ml of water. Use 1 µl of each of these solutions for carrying out the paper chromatography described in Section 5.

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4.2. **Products sparingly soluble in water**

4.2.1. Weigh out 1 g and 5 g of the sample and disperse in 50 ml of water, make up to 100 ml with water in each case and mix. Filter the two dispersions over a fluted filter (3.4) and use 1 µl of each of the filtrates in order to carry out the paper chromatography described in Section 5.

4.2.2. Prepare once again two dispersions of each sample by dispersing 1 g and 5 g in 50 ml of water, acidify with dilute hydrochloric acid (2.7), make up with water to 100 ml and mix. Filter the dispersions through a fluted filter (3.4) and use 1 µl of the two filtrates in order to carry out the paper chromatography described in Section 5.

4.3. **Creams**

Disperse 5 g and 20 g of each product in 100 ml of water and use the dispersions to carry out the paper chromatography described in Section 5.

5. METHOD

5.1. Put an appropriate quantity of solvents A (2.2) and B (2.3) into two separate chromatography tanks in order to carry out the descending paper chromatography. Saturate the chromatography tanks for at least 24 hours with solvent vapour.

5.2. Apply 1 µl of one sample solution and of one reference solution prepared according to Sections 4 and 2.1 to each starting point on a strip of chromatography paper (Whatman No 3 or equivalent) 40 cm long and 20 cm wide (3.1) or another suitable format and evaporate the solution in air.

5.3. Place the chromatography strip (5.2) in the chromatography tank filled with developing solvent A (5.1) and develop until the solvent front has advanced 35 cm (about 15 hours).

5.4. Repeat the procedure described in Sections 5.2 and 5.3, using chromatography paper (Whatman No 4 or equivalent) (3.1) and developing solvent B. Chromatograph until the solvent front has advanced 35 cm (about five hours).

5.5. After development remove the chromatograms and dry them in air.

5.6. Reveal the spots in the chromatogram by spraying it successively with:

5.6.1. detecting agent A (2.4) followed shortly thereafter by detecting agent B (2.5). The spots of the persulphates will now appear first in the chromatogram and will be followed by the hydrogen peroxide spots. Mark the spots with a pencil;

5.6.2. detecting agent C (2.6) on the chromatograms obtained in accordance with Section 5.6.1; the presence of bromates will now be indicated by greyish blue spots in the chromatogram.

5.7. Under the abovementioned conditions pertaining to developing solvents A (2.2) and B (2.3), the Rf values of the reference substances (2.1) are approximately as follows:

	Developing solvent A (2.2)	*Developing solvent B (2.3)*
Sodium persulphate	0·40	0·10
Potassium persulphate	0·40	0·02 + 0·05
Ammonium persulphate	0·50	0·10 + 0·20
Sodium bromate	0·40	0·20
Potassium bromate	0·40	0·10 + 0·20
Hydrogen peroxide	0·80	0·80