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FIRST COMMISSION DIRECTIVE

of 22 December 1980

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

(80/1335/EEC)

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

(80/1335/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 prescribed pursant to Community provisions concerning the composition of the cosmetic products are satisfied;

Whereas all the necessary methods of analysis must be established as soon as possible; whereas the laying down of methods for the sampling, laboratory preparation, identification and determination of free sodium and potassium hydroxides, the identification and determination of oxalic acid and alkaline salts thereof in hair care products, the determination of chloroform in toothpastes and of zinc, and the identification and determination of phenolsulfonic acid constitutes a first step in this direction;

Whereas the measures laid down in the present Directive are in conformity 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, in the official testing of cosmetic products:

— the sampling,
— the laboratory preparation of test samples,
— the identification and determination of free sodium and potassium hydroxides,
— the identification and determination of oxalic acid and alkaline salts in hair-care products,
— the determination of chloroform in toothpastes,
— the determination of zinc,
— the identification and determination of phenolsulfonic acid

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 1982.

They shall forthwith inform the Commission thereof.

Article 3

This Directive is addressed to the Member States.

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[1] OJ No L 262, 27. 9. 1976, p. 169.
[2] OJ No L 192, 31. 7. 1979, p. 35.

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ANNEX

I. SAMPLING OF COSMETIC PRODUCTS

1. SCOPE AND FIELD OF APPLICATION

The procedure for the sampling of cosmetic products is described with a view to their analysis in the various laboratories.

2. DEFINITIONS

2.1. *Basic sample:*

a unit taken from a batch offered for sale.

2.2. *Total sample:*

the sum of all the basic samples having the same batch number.

2.3. *Laboratory sample:*

a representative fraction of the total sample that is to be analyzed in the individual laboratories.

2.4. *Test portion:*

a representative portion of the laboratory sample that is required for one analysis.

2.5. *Container:*

the article that contains the product and is in continuous direct contact with it.

3. SAMPLING PROCEDURE

3.1. Cosmetic products shall be sampled in their original containers and forwarded to the analytical laboratory unopened.

3.2. For cosmetic products which are placed on the market in bulk or retailed in a container different from the original manufacturer's pack, appropriate instructions for sampling at the point of use or sale should be issued.

3.3. The number of basic samples required for the preparation of the laboratory sample shall be determined by the analytical method and the number of analyses to be performed by each laboratory.

4. SAMPLE IDENTIFICATION

4.1. Samples shall be both sealed where taken and identified, in accordance with the rules in force in the relevant Member State.

4.2. Each basic sample taken shall be labelled with the following information:

— name of the cosmetic product,
— date, time and place of sampling,
— name of the person responsible for taking the sample,
— name of the inspectorate.

4.3. A report on the sampling shall be drawn up in accordance with the rules in force in the relevant Member State.

5. STORAGE OF SAMPLES

5.1. Basic samples must be stored in accordance with the manufacturer's instructions appearing on the label if any.

5.2. Unless other conditions are specified, laboratory samples shall be stored in the dark at between 10 and 25 ºC.

5.3. Basic samples must not be opened until the analysis is about to begin.

II. LABORATORY PREPARATION OF TEST PORTIONS

1. GENERAL

1.1. Where possible the analysis shall be carried out on each basic sample. If the basic sample is too small, the minimum number of basic samples

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should be used. They should first be mixed together thoroughly before taking the test portion.

1.2. Open the container, under an inert gas if so specified in the analytical method and withdraw the number of test portions required as quickly as possible. The analysis should then proceed with the least possible delay. If the sample has to be preserved the container should be resealed under an inert gas.

1.3. Cosmetic products may be prepared in liquid or solid forms or in a semi-solid form. If separation of an initially homogeneous product occurs it should be re-homogenized before taking the test portion.

1.4. If the cosmetic product is put up for sale in a special way, as a result of which it cannot be treated in accordance with these instructions, and if no provision is made for the relevant methods of examination an original procedure may be adopted, provided that it is set out in writing as part of the analysis report.

2. LIQUIDS

2.1. These may occur in the form of products such as solutions in oil, in alcohol, and in water, toilet waters, lotions or milks, and may be packed in flasks, bottles, ampoules or tubes.

2.2. **Withdrawal of the test portion:**

— shake the container vigorously before opening,
— open the container,
— pour a few millilitres of the liquid into a test-tube for visual examination of its character for the purpose of taking the test-portion,
— reseal the container, or
— withdraw the required test portions,
— reseal the container carefully.

3. SEMI-SOLIDS

3.1. These may occur in the form of products such as pastes, creams, stiff emulsions and gels and may be packed in tubes, plastic bottles or jars.

3.2. **Withdrawal of the test portion, *either:***

3.2.1. narrow-necked containers. Expel at least the first centimetre of the product. Extrude the test portion and reseal the container immediately.

3.2.2. wide-necked containers. Scrape the surface evenly to remove the top layer. Take out the test portion and reseal the container immediately.

4. SOLIDS

4.1. These may occur in the form of products such as loose powders, compacted powders, sticks and may be packed in a wide variety of containers.

4.2. Withdrawal of the test portion, either:

4.2.1. loose powder — shake vigorously before unstoppering or opening. Open and remove the test portion.

4.2.2. Compact powder or stick — remove the surface layer by even scraping. Take the test portion from underneath.

5. PRODUCTS IN PRESSURIZED PACKAGES *('aerosol dispensers')*

5.1. These products are defined in Article 2 of Council Directive 75/324/EEC of 20 May 1975 [1].

5.2. **Test portion:**

After vigorous shaking, a representative quantity of the contents of the aerosol dispenser are transferred with the aid of a suitable connector (see for example Figure 1: in specific cases the analytical method may require the use of other connectors) into a plastic-coated glass bottle (Figure 4) fitted with an aerosol valve but not fitted with a dip tube. During the transfer the bottle is held valve downwards. This transfer renders the contents clearly visible corresponding to one of the following four cases:

[1] OJ No L 147, 9. 6. 1975, p. 40.

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5.2.1. An aerosol product in the form of a homogeneous solution for direct analysis.

5.2.2. An aerosol product consisting of two liquid phases. Each phase can be analyzed after the lower phase has been separated into a second transfer bottle. In this case the first transfer bottle is held valve downwards. In such a case this lower phase is often aqueous and devoid of propellent (e.g. butane/water formulation).

5.2.3. An aerosol product containing a powder in suspension. The liquid phase can be analyzed after removal of the powder.

5.2.4. A foam or cream product. First weigh exactly into the transfer bottle 5 to 10 g of 2-methoxyethanol. This substance prevents foam from forming during the degassing operation and it is then possible to expel the propellent gases without loss of liquid.

5.3. **Accessories**

The connector (Figure 1) is made of duralumin or brass. It is designed to fit to different valve systems via a polyethylene adaptor. It is given as an example: other connectors may be used. (See Figures 2 and 3).

The transfer bottle (Figure 4) is made of white glass coated on the outside with a protective layer of transparent plastic material. It holds 50 to 100 ml. It is fitted with an aerosol valve without a dip tube.

5.4. **Method**

In order that enough of the sample may be transferred, the transfer bottle must be purged of air. For this purpose, introduce through the connector about 10 ml of dichlorodifluoromethane or butane (depending on the aerosol product to be examined) and then degas completely until the liquid phase disappears, holding the transfer bottle with the valve uppermost. Remove the connector. Weigh the transfer bottle ('a' grams). Vigorously shake the aerosol dispenser from which the sample is to be taken. Attach the connector to the valve on the sample aerosol container (valve upwards), fit the transfer flask (neck downwards) to the connector and press. Fill the transfer bottle to about two thirds full. If the transfer ceases prematurely owing to pressure equalization, it can be resumed by chilling the transfer bottle. Remove the connector, weigh the filled bottle ('b' grams) and determine the weight of aerosol sample transferred, m_1 (m_1 = b — a).

The sample thus obtained can be used:

1. for a normal chemical analysis;

2. for an analysis of the volatile constituents by gas chromatography.

5.4.1. *Chemical analysis*

Holding the transfer bottle valve upwards, proceed as follows:

— degas. If the degassing operation gives rise to foaming, use a transfer bottle into which an exactly-weighed quantity (5 to 10 g) of 2-methoxyethanol has previously been introduced with a syringe through the connector,

— complete the removal of the volatile constituents without loss by shaking in a waterbath maintained at 40 ºC. Detach the connector,

— reweigh the transfer bottle ('c' grams) in order to determine the weight of the residue, m_2 (m_2 = c — a).

(*NB:*

When calculating the weight of the residue, deduct the weight of any 2-methoxyethanol used.)

— open the transfer bottle by removing the valve,

— dissolve the residue completely in a known quantity of an appropriate solvent,

— perform the desired determination on an aliquot.

Formulas for the calculation are:

$$R = \frac{r \times m_2}{m_1} \text{ and } Q = \frac{R \times P}{100},$$

where:

m_1 = mass of aerosol taken into the transfer bottle;

m_2 = mass of residue after heating at 40 ºC;

r = percentage of the particular substance in m_2 (determined according to the appropriate method);