Third Commission Directive of 27 September 1983 on the approximation of the laws of the Member States relating to methods of analysis necessary for checking the composition of cosmetic products (83/514/EEC)

THIRD COMMISSION DIRECTIVE

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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

(83/514/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 last amended by Directive 83/341/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 laid down as quickly as possible; whereas two steps towards the attainment of this objective having already been taken through the definition of certain methods in Commission Directives 80/1335/EEC[(3)] and 82/434/EEC[(4)], the third step is to consist in the definition of methods for the determination of dichloromethane and 1,1,1-trichloroethane, the identification and determination of quinolin-8-ol and bis(8-hydroxyquino-linium) sulphate, the determination of ammonia, the identification and determination of nitromethane, the identification and determination of mercaptoacetic acid in hair-waving, hair-straightening and depilatory products, the identification and determination of hexachlorophene (INN), the tion of tosylchloramide sodium (INN), the determination of total fluorine in dental creams, the identification and determination of organomercury compounds, the determination of alkali and alkaline earth sulphides;

Whereas the measures provided for 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:

— determination of dichloromethane and 1,1,1-trichloroethane,

— identification and determination of quinolin-8-ol and bis(8-hydroxyquinolinium) sulphate,

— determination of ammonia,

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— identification and determination of nitro-methane,
— identification and determination of mercapto-acetic acid in hair-waving, hair-straightening and depilatory products,
— identification and determination of hexachlorophene (INN),
— determination of tosylchloramide sodium (INN),
— determination of total fluorine in dental creams,
— identification and determination of organo-mercury compounds,
— determination of alkali and alkaline earth sulphides,

are performed in accordance with the methods described 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 1984.

They shall forthwith inform the Commission thereof.

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 27 September 1983.

For the Commission

Frans ANDRIESSEN

Member of the Commission

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ANNEX

DETERMINATION OF DICHLOROMETHANE AND 1,1,1-TRICHLOROETHANE

1. SCOPE AND FIELD OF APPLICATION

This method describes the determination of dichloromethane (methylene chloride) and 1,1,1-trichloroethane (methyl chloroform) in all cosmetic products likely to contain these solvents.

2. DEFINITION

The dichloromethane and 1,1,1-trichloroethane content of the sample determined according to this method are expressed in percentage by mass.

3. PRINCIPLE

The method uses gas chromatography with chloroform as internal standard.

4. REAGENTS

All reagents must be of analytical quality.

4.1. Chloroform ($CHCl_3$).

4.2. Carbon tetrachloride (CCl_4).

4.3. Dichloromethane (CH_2Cl_2).

4.4. 1,1,1-trichloroethane (CH_3CCl_3).

4.5. Acetone.

4.6. Nitrogen.

5. APPARATUS

5.1. Usual laboratory apparatus.

5.2. Gas chromatograph fitted with a thermal conductivity detector.

5.3. Transfer bottle, 50 to 100 ml (see sampling method 5.3)[(5)].

5.4. Pressure gas-syringe, 25 or 50 µl (see sampling method 5.4.2.2)[(5)].

6. PROCEDURE

6.1. Non-pressurized sample: weigh the sample accurately in a stoppered conical flask. Introduce an accurately weighed quantity of chloroform (4.1) as internal standard equivalent to the presumed quantity of dichloromethane and 1,1,1-trichloroethane contained in the sample. Mix throroughly.

6.2. Pressurized sample: use the sampling method described in the sampling chapter, but with the following refinements:

6.2.1. After transferring a sample into a transfer bottle (5.3), further introduce into the transfer bottle a volume of chloroform (4.1) as internal standard equivalent to the presumed quantity of dichloromethane and/or 1,1,1-trichloroethane contained in the sample. Mix thoroughly. Rinse the dead volume of the valve with 0,5 ml of carbon tetrachloride (4.2). After drying, determine accurately the added mass of the internal standard by difference.

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6.2.2. After filling the syringe with the sample, the nozzle of the syringe should be purged with nitrogen (4.6) so that no residue remains before injection into the chromatograph.

6.2.3. After each sample is taken, the surface of the valve and of the transfer piece should be rinsed several times with acetone (4.5) (using as required a hypodermic syringe) and then dried thoroughly with nitrogen (4.6).

6.2.4. For each analysis, take measurements using two different transfer bottles and five measurements per bottle.

7. CHROMATOGRAPHIC CONDITIONS

7.1. ***Precolumn***

Tubing: stainless steel.

Length: 300 mm

Diameter: 3 or 6 mm.

Packing: same material as used for the analytical column packing.

7.2. ***Column***

The stationary phase is made of Hallcomid M 18 on chromosorb. The column must yield a resolution 'R' equal to, or better than, 1,5, where:

$$R = 2\frac{d'(r_2 - r_1)}{W_1 + W_2}$$

let:

r_1 and r_2 = retention times (in minutes),
W_1 and W_2 = peak widths at half height (in millimetres),
d' = the chart speed (in millimetres per minute).

7.3. As examples the following columns yield the results sought:

Column	***I***	***II***
Material:	Stainless steel tubing	Stainless steel tubing
Length:	350 cm	400 cm
Diameter:	3 mm	6 mm
Support:		
chromosorb:	WAW	WAW-DMCS-HP
sieve analysis	100 to 120 mesh	60 to 80 mesh
Stationary phase:	Hallcomid M 18, 10%	Hallcomid M 18, 20%

Temperature conditions may vary as a function of the apparatus. In the examples they have been set as follows:

Column	***I***	***II***
Temperatures:		
column:	65 °C	75 °C

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injector:	150 °C	125 °C
detector:	150 °C	200 °C
Carrier gas:		
helium flow rate:	45 ml/min	60 ml/min
inlet pressure	2,5 bar	2 bar
Injection:	15 µl	15µl

8. MIXTURE FOR ESTABLISHING THE RESPONSE FACTORS

Make up the following accurately weighed mixture in a stoppered conical flask:

Dichloromethane (4.3), 30 % (m/m).

1,1,1-trichloroethane (4.4), 35 % (m/m).

Chloroform (4.1), 35 % (m/m).

9. CALCULATIONS

9.1. ***Calculating a response factor of a substance 'p' relative to a substance 'a' selected as an internal standard***

Let the first substance be 'p', where:

k_p = its response factor,
m_p = its mass in the mixture,
A_p = its peak area.

Let the second substance be 'a', where:

k_a = its response factor (made equal to unity),
M_a = its mass in the mixture,
A_a = its peak area,

then:

$$k_p = \frac{m_p \times A_a}{M_a \times A_p}$$

As examples the following response factors have been obtained (for chloroform: k = l):

Dichloromethane: $k_1 = 0{,}78 \pm 0{,}03$

1,1,1-trichloroethane: $k_2 = 1{,}00 \pm 0{,}03$

9.2 ***Calculate the % (m/m) of dichloromethane and 1,1,1-trichloroethane present in the sample to be analyzed***

Let:

m_a = the mass (in grams) of chloroform introduced,
M_s = the mass (in grams) of the sample to be analyzed,
A_a = the area of the chloroform peak,
A_1 = the area of the d ichloromethane peak,
A_2 = the area of the 1 1,1-trichloroethane peak,