Fourth Commission Directive of 11 October 1985 on the approximation of the laws of the Member States relating to methods of analysis necessary for checking the composition of cosmetic products (85/490/EEC)

ANNEX

DETERMINATION OF CHLOROBUTANOL

1. SCOPE AND FIELD OF APPLICATION

This method is suitable for the determination of chlorobutanol (INN) up to a maximum concentration of 0,5 % (m/m) in any cosmetic product, except aerosols.

2. DEFINITION

The content of chlorobutanol measured by this method is expressed as percentage by mass (% m/m) of product.

3. PRINCIPLE

After appropriate treatment of the product to be analyzed the determination is done by gas chromatography using 2,2,2-trichloroethanol as the internal standard.

4. REAGENTS

All the reagents should be of analytical purity.

4.1. Chlorobutanol (1,1,1-trichloro-2-methylpropan-2-ol).

4.2. 2,2,2-Trichloroethanol.

4.3. Absolute ethanol.

4.4. Standard solution of chlorobutanol: 0,025 g in 100 ml ethanol (4.3) (m/v).

4.5. Standard solution of 2,2,2-trichloroethanol: 4 mg in 100 ml ethanol (4.3) (m/v).

5. APPARATUS

5.1. Normal laboratory equipment.

5.2. Gas chromatograph with electron detector, Ni 63.

6. PROCEDURE

6.1. **Preparation of sample**

Weigh accurately between 0,1 and 0,3 g (p g) of the sample. Place in 100 ml volumetric flask. Dissolve it in ethanol (4.3), add 1 ml of the internal standard solution (4.5) and make up to the mark with ethanol (4.3).

6.2. Gas chromatography conditions

6.2.1. The operating conditions must yield a resolution factor $R \geq 1,5$.

$$R = 2\frac{d'R_2 - d'R_2}{W_1 + W_2}$$

Where

R_1 and R_2 = retention times, in minutes, of the peaks,
W_1 and W_2 = peak widths at half height, in millimetres,
d' = the chart speed, in millimetres per minute.

6.2.2. As examples, the following operating conditions provide the required resolution:

Column	**I**	**II**
Material	Glass	Stainless steel
Length	1,80 m	3 m
Diameter	3 mm	3 mm
Stationary phase	10 % Carbowax 20 M TPA on Gaschrom Q 80-100 mesh	5 % OV 17 on Chromosorb WAW DMCS 80-100 mesh
Conditioning	2 to 3 days at 190 °C	
Temperature:		
— injector	200 °C	150 °C
— column	150 °C	100 °C
— detector	200 °C	150 °C
Carrier gas	Nitrogen	Argon/methane (95/5 v/v)
Flowrate	35 ml/min	35 ml/min

6.3. **Standard curve**

Using five 100 ml volumetric flasks, add 1 ml of the standard solution (4.5) and 0,2, 0,3, 0,4, 0,5, and 0,6 ml of solution 4.4 respectively, and make up to the mark with ethanol (4.3) and mix. Inject 1 µl of each of these solutions into the chromatograph in accordance with the operating conditions described in 6.2.2 and construct a calibration curve by plotting as the abscissa the ratio of the mass of chlorobutanol to that of 2,2,2-trichloroethanol and as the ordinate the ratio of the corresponding peak areas.

6.4. Inject 1 µl of solution obtained in 6.1 and proceed according to the conditions described in 6.2.2

7. CALCULATION

7.1. Calculate from the standard curve (6.3) the quantity ‘a’ expressed as µg of chlorobutanol, in the solution 6.1.

7.2. The content of chlorobutanol in the sample is calculated according to the formula:

$$\% \text{ chlorobutanol (m/m)} = \frac{a \times 10^2}{p \times 10^6} = \frac{a}{p \times 10^4}$$

8. REPEATABILITY[1]

For a chlorobutanol content of 0,5 % (m/m) the difference between the results of two determinations in parallel carried out on the same sample should not exceed 0,01 %.
Note

If the result is equal to or exceeds the maximum permitted concentration it is necessary to check the absence of interferences.

(**1**) ISO 5725.