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)

ANNEX

DETERMINATION OF DICHLOROMETHANE AND 1,1,1-TRICHLOROETHANE
IDENTIFICATION AND DETERMINATION OF NITROMETHANE

1. SCOPE AND FIELD OF APPLICATION

This method is suitable for the identification and determination of nitromethane at up to about 0,3 % in cosmetic products packed in aerosol dispensers.

2. DEFINITION

The nitromethane content of the sample determined according to this method is expressed in percentage by mass of nitromethane, in the total aerosol dispenser content.

3. PRINCIPLE

The nitromethane is identified by colour reaction. Nitromethane is determined gas chromatographically after addition of an internal standard.

4. IDENTIFICATION

4.1. ***Reagents***

All reagents should be of analytical purity.

4.1.1. Sodium hydroxide, 0,5 M solution.

4.1.2. *Folin's reagent*

Dissolve 0,1 g of sodium 3,4-dihydro-3,4-dioxonaphthalene-l-sulphonate in water and dilute to 100 ml.

4.2. ***Procedure***

To 1 ml of sample add 10 ml of 4.1.1 and 1 ml of 4.1.2. A violet coloration indicates the presence of nitromethane.

5. DETERMINATION

5.1. *Reagents*

All reagents must be of analytical quality.

5.1.1. Chloroform (internal standard 1).

5.1.2. 2,4-dimethylheptane (internal standard 2).

5.1.3. Ethanol, 95 %.

5.1.4. Nitromethane.

5.1.5. *Chloroform reference solution*

Into a tared 25 ml volumetric flask, introduce about 650 mg of chloroform (5.1.1). Accurately reweigh the flask and contents. Make up to 25 ml with 95 % ethanol (5.1.3). Weigh and calculate the percentage by mass of chloroform in this solution.

5.1.6. *2,4-dimethylheptane reference solution*

Make up in a similar manner to the chloroform reference solution but weigh 270 mg of 2,4-dimethylheptane (5.1.2) into the 25 ml volumetric flask.

5.2. ***Apparatus***

5.2.1. Gas chromatograph with flame ionization detector.

5.2.2. Apparatus for sampling of aerosols (transfer bottle, microsyringe connectors, etc.) as described in Chapter II of the Annex to Commission Directive 80/1335/EEC of 22 December 1980[(1)].

5.2.3. Usual laboratory apparatus.

5.3. **Procedure**

5.3.1. *Preparation of the sample*

Into a 100 ml tared transfer bottle, purged or evacuated according to the procedure described in 5.4 of Chapter II of the abovementioned Directive, introduce about 5 ml of either of the internal standard solutions (5.1.5 or 5.1.6). Use a 10 or 20 ml glass syringe, without needle, adapted to the transfer piece following the technique described in paragraph 5 of Chapter II of the above-mentioned Commission Directive. Reweigh to determine the quantity introduced. Using the same technique, transfer into this bottle about 50 g of the contents of the aerosol dispenser sample. Again reweigh to determine the quantity of sample transferred. Mix well.

Inject about 10 µl using the specified microsyringe (5.2.2). Make five injections.

5.3.2. *Preparation of the standard*

Into a 50 ml volumetric flask, accurately weigh about 500 mg of nitromethane (5.1.4) and either 500 mg of chloroform (5.1.1) or 210 mg of 2,4-dimethylheptane (5.1.2). Make up to volume with 95 % ethanol (5.1.3). Mix well. Place 5 ml of this solution into a 20 mg volumetric flask. Make up to volume with 95 % ethanol (5.1.3).

Inject about 10 µl using the specified microsyringe (5.2.2). Make five injections.

5.3.3. *Gas chromatographic conditions*

5.3.3.1. *Column*

This is in two parts, the first containing didecyl phthalate on Gas Chrom Q as packing, the second having Ucon 50 HB 280X on Gas Chrom Q as packing. The prepared combined 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).

As examples the following two parts yield the required resolution:

Column A:

Material: stainless steel.

Length: 1,5 m.

Diameter: 3 mm.

Packing: 20 % didecyl phthalate on Gas Chrom Q (100 to 120 mesh).

Column B:

Materia: stainless steel.

Length: 1,5 m.

Diameter: 3 mm.

Packing: 20 % Ucon 50 HB 280X on Gas Chrom Q (100 to 120 mesh).

5.3.3.2. *Detector*

A suitable sensitivity setting for the electrometer of the flame ionization detector is 8×10^{-10}A.

5.3.3.3. *Temperature conditions*

The following have been found suitable:

Injection port: 150 °C,

Detector: 150 °C,

Column: between 50 and 80 °C depending upon individual columns and apparatus.

5.3.3.4. *Suitable gas supplies*

Carrier gas: nitrogen.

Pressure: 2,1 bar.

Flow: 40 ml/min

Detector supplies: as specified by the makers of the detector.

6. CALCULATIONS

6.1. ***Response factor of nitromethane, calculated with reference to the internal standard used***

If ‘n’ represents nitromethane:

let:

k_n = its response factor,
m'_n = its mass (in grams) in the mixture,
S'_n = its peak area.

If ‘c’ represents the internal standard, chloroform or 2,4-dimathylheptane:

let:

m'_c = its mass (in grams) in the mixture,
S'_c = its peak area,

then:

$$K_n = \frac{m'_n}{m'_c} \times \frac{S'_c}{S'_n}$$

(k_n is a function o[2] the apparatus).

6.2. ***Concentration of nkromethane in the sample***

If 'n' represents nitromethane:

let:

k_n = its response factor,
S_n = its peak area.

If 'c' represents the internal standard, chloroform or 2,4-dimethylheptane:

let:

m_c = its mass (in grans) in the mixture,
S_c = its peak area,
M = the mass (in grams) of the aerosol transferred,

then the % (m/m) nitromethane in the sample is:

$$\frac{m_c}{M} \times \frac{K_n \times S_n}{S_c} \times 100$$

7. REPEATABILITY[(2)]

For a nitromethane content of about 0,3 % (m/m), the difference between the results of two determinations carried out in parallel on the same sample should not exceed an absolute value of 0,03 % (m/m).

(**1**) OJ No L383, 31.12.1980, p. 27.

(**2**) Norm ISO 5725.