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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)

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ANNEX

DETERMINATION OF DICHLOROMETHANE AND 1,1,1-TRICHLOROETHANE QUANTITATIVE DETERMINATION OF TOSYLCHLORAMIDE SODIUM (INN) (CHLORAMINE-T)

1. SCOPE AND FIELD OF APPLICATION

This method relates to the quantitative thin-layer chromatographic determination of tosylchloramide sodium (chloramine-T) in cosmetic products.

2. DEFINITION

The chloramine-T content of the sample, as determined by this method, is expressed as a percentage by mass (m/m).

3. PRINCIPLE

Chloramine-T is completely hydrolyzed to 4-toluenesulphonamide by boiling with hydrochloric acid.

The amount of 4-toluenesulphonamide formed is determined photo-densitometrically by thin-layer chromatography.

4. REAGENTS

All reagents should be of analytical purity.

4.1. Tosylchloramide sodium (chloramine-T).

4.2. Standard solution of 4-toluenesulphonamide: 50 mg of 4-toluenesulphonamide in 100 ml of ethanol (4.5).

4.3. Hydrochloric acid, 37 % (m/m), d_4^{20} = 1,18 g/ml.

4.4. Diethyl ether.

4.5. Ethanol, 96 % (v/v).

4.6. ***Development solvent***

4.6.1. 1-butanol /ethanol (4.5) /water (40: 4: 9; v/v/v), or

4.6.2. Chloroform /acetone (6: 4; v/v).

4.7. Ready prepared thin-layer chromatography plates, silica gel 60, without fluorescent indicator.

4.8. Potassium permanganace.

4.9. Hydrochloric acid, 15 % (m/m).

4.10 Spray reagent: 2-toluidine, 1 % (m/v) solution in ethanol (4.5).

5. **APPARATUS**

5.1. Normal laboratory apparatus.

5.2. Usual thin-layer chromatography equipment.

5.3. Photodensitometer.

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6. PROCEDURE

6.1. ***Hydrolysis***

Weigh accurate y into a 50 ml round-bottom flask approximately 1 g of the sample (m). Add 5 ml of water and 5 ml of hydrochloric acid (4.3) and boil for one hour, using a reflux condenser. Immediately transfer the hot suspension with water into a 50 ml graduated flask. Allow to cool and make up to the mark with water. Centrifuge at at least 3000 rpm for five minutes and pass the supernatant liquid through a filter.

6.2. ***Extraction***

6.2.1. Take 30 ml of the filtrate and extract three times with 15 ml of diethyl ether (4.4). If necessary dry the ethereal phases and collect them in a 50 ml graduated flask. Make up with diethyl ether (4.4).

6.2.2. Take 25 ml of the dried ethereal extract and evaporate to dryness in a nitrogen stream. Redissolve the residue with 1 ml of ethanol (4.5).

6.3. ***Thin-layer chromatography***

6.3.1. Spot 20 µl of the ethanolic residue (6.2) on to a thin-layer chromatography plate (4.7).

At the same time and in the same manner, apply 8, 12, 16 and 20 µl of the standard solution of 4-toluenesulphonamide (4.2).

6.3.2. Then allow to develop approximately 150 mm in the development solvent (4.6.1 or 4.6.2).

6.3.3. After completely evaporating the development solvent, place the plate for two to three minutes in an atmosphere of chlorine vapour, which is produced by pouring about 100 ml of hydrochloric acid (4.9) over about 2 g of potassium permanganate (4.8) in a closed vessel. Remove the excess chlorine by heating the plate to 100 ^{o}C for five minutes. Then spray the plate with the reagent (4.10).

6.4. ***Measurement***

After approximately one hour, measure the violet spots by means of a photodensitometer at 525 nm.

6.5. ***Plotting the calibration curves***

Plot the maximum peak height values ascertained for the four 4-toluenesulphonamide spots against the corresponding quantities of 4-toluenesulphonamide (i.e. 4, 6, 8, 10 µg of 4-toluenesulphonamide per spot).

7. NOTE

The method may be controlled by using a solution of 0,1 or 0,2 % (m/v) of chloramine-T (4.1) treated in the same way as the sample (6).

8. CALCULATION

The chloramine-T content of the sample, expressed as a percentage by mass, is calculated as follows:

$$\% \text{ (m/m) Tosylchloramide sodium} = \frac{1.33 \times a}{60 \times m}$$

where:

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1,33 = the 4-toluenesulphonamide-chloramine-T conversion factor,
a = the quantity (in μg) of 4-toluenesulphonamide in the sample as read from the calibration curves,
m = the mass (in grams) of the sample taken.

9. REPEATABILITY[1]

For a chloramine-T content of about 0,2 % (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).

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(**1**) Norm ISO 5725.