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
DETERMINATION OF AMMONIA

1.   SCOPE AND FIELD OF APPLICATION

This method describes the determination of free ammonia in cosmetic products.

2.   DEFINITION

The ammonia content of the sample determined in accordance with this method is expressed in percentage by mass of ammonia.

3.   PRINCIPLE

Barium chloride solution is added to a test portion of the cosmetic product diluted in an aqueous methanol medium. Any precipitate which may form is filtered or centrifuged off. This procedure avoids the loss of ammonia, during steam distillation, from certain ammonium salts such as the carbonate and hydrogencarbonate and those of the fatty acids, with the exception of ammonium acetate.

The ammonia is steam distilled from the filtrate or supernatant and is determined by potentiometric or other titration.

4.   REAGENTS

All reagents should be of analytical purity.

4.1.   Methanol.

4.2.   Barium chloride dihydrate, 25 % (m/v) solution.

4.3.   Orthoboric acid, 4 % (m/v) solution.

4.4.   Sulphuric acid, 0,25 M standard solution.

4.5.   Anti-foam liquid.

4.6.   Sodium hydroxide, 0,5 M standard solution.

4.7.   Indicator, if required: mix 5 ml of a 0,1 % (m/v) methyl red solution in ethanol with 2 ml of 0,1 % (m/v) methylene blue solution in water.

5.   APPARATUS

5.1.   Usual laboratory apparatus.

5.2.   Centrifuge with stoppered 100 ml bottles.

5.3.   Steam distillation apparatus.

5.4.   Potentiometer.

5.5.   Indicating glass electrode and dimercury dichloride (calomel) reference electrode.

6.   PROCEDURE

6.1.   Weigh into a 100 ml standard flask a mass (m) of the sample corresponding to 150 mg maximum of ammonia.

Third Commission Directive of 27 September 1983 on the approximation of the laws...
ANNEX
Document Generated: 2023-12-15

3

6.2.   Add 10 ml of water, 10 ml of methanol (4.1) and 10 ml of barium chloride solution (4.2). Make up to 100 ml with methanol (4.1).

6.3.   Mix and leave overnighs in the refrigerator (5 ^{o}C).

6.4.   Then filter, or centrifuge the still cold solution in closed tubes for 10 minutes, so as to obtain a clear filtrate or supernatant layer.

6.5.   Pipette 40 ml of this clear solution into the steam distillation apparatus (5.3), followed by 0,5 ml of antifoam liquid (4.5), where appropriate.

6.6.   Distil and collect 200 ml of distillate in a 250 ml beaker containing 10 ml of standard sulphuric acid (4.4) and 0,1 ml of indicator (4.7).

6.7.   Back titrate the excess acid with standard sodium hydroxide solution (4.6).

6.8.   NB: For potentiometric determination, collect 200 ml of distillate in a 250 ml beaker containing 25 ml of orthoboric acid solution (4.3) and titrate with standard sulphuric acid (4.4), recording the neutralization curve.

7.   CALCULATIONS

7.1.   *Calculation in the case of back titration*

Let:

V_1	=	the volume (in inillilitres) of the sodium hydroxide solution (4.6) used,
M_1	=	its actual molarity (4.6),
M_2	=	the actual molarity factor of the sulphuric acid solution (4.4),
m	=	the mass (in milligrams) of the test portion (6.1) taken,

then:

$$\text{ammonia \% (m/m)} = \frac{(20\,M_2 - V_1 M_1) \times 17 \times 100}{0,4\,m} = \frac{(20\,M_2 - V_1 M_1) \times 4250}{m}$$

7.2.   *Calculation in the case of direct potentiometric titration*

Let:

V_2	=	the volume (in millilitres) of the sulphuric acid solution (4.4) used,
M_2	=	its actual molarity (4.4),
m	=	the mass (in milligrams) of the test portion (6.1) taken,

then:

$$\text{ammonia \% (m/m)} = \frac{V_2 \times M_2 \times 17 \times 100}{0,4\,m} = \frac{4250\,V_2 M_2}{m}$$

8.   REPEATABILIT Y[1]

For a content of about 6 % ammonia, the difference between the results of two determinations carried out in parallel on the same sample should not exceed an absolute value of 0,6 %.

(**1**)   Norm ISO 5725.