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 ALKALI AND ALKALINE EARTH SULPHIDES

1. SCOPE AND FIELD OF APPLICATION

This method describes the determination of sulphides present in cosmetic products. The presence of thiols or other reducing agents (including sulphites) does not interfere.

2. DEFINITION

The concentration of sulphides determined by this method is expressed as a percentage of sulphur by mass.

3. PRINCIPLE

After acidification of the medium, hydrogen sulphide is entrained by a stream of nitrogen and then fixed in the form of cadmium sulphide. The latter is filtered and rinsed and then determined by iodometry.

4. REAGENTS

All reagents should be of analytical purity.

4.1. Concentrated hydrochloric acid, d_4^{20} = 1,19 g/ml.

4.2. Sodium thiosulphate, 0,1 M standard solution.

4.3. Iodine, 0,05 M standard solution.

4.4. Disodium sulphide.

4.5. Cadmium di(acetate).

4.6. Concentrated ammonia, $d_4>^{20}$ = 0,90 g/ml.

4.7. Ammoniacal solution of cadmium di(acetate): dissolve 10 g of cadmium di(acetate) (4.5) in approximately 50 ml of water. Add ammonia (4.6) until the precipitate redissolves (i.e. approximately 20 ml). Make up to the 100 ml mark with water.

4.8. Nitrogen.

4.9. Solution of ammonia M.

5. APPARATUS

5.1. Usual laboratory equipment.

5.2. 100 ml round-bottom flask with three standard ground-glass necks.

5.3. Two 150 ml conical flasks with ground-glass necks, fitted with a device comprising a dip tube and a side outlet tube for releasing the entraining gas.

5.4. One long-stem tunnel.

6. PROCEDURE

6.1. Entrainment of the sulphides

6.1.1. Take a package which has not been previously opened. Weigh accurately a mass (m) (expressed in grams) of the product corresponding to not more than 30 mg of sulphide ions in the round-bottom flask (5.2). Add 60 ml of water and two drops of an anti-foaming liquid.

6.1.2. Transfer 50 ml of solution (4.7) to each of the two conical flasks (5.3).

6.1.3. Fit a dropping funnel, the dip tube and the outlet tube on to the round-bottom flask (5.2). Connect the outlet tube to the conical flasks (5.3) set up in series by means of PVC tubing.

NB: The entraining apparatus must pass the following leak-tightness test: simulating the test conditions, replace the product to be determined by 10 ml of a sulphide solution (prepared from 4.4) containing 'X mg' of sulphide (iodometrically determined). Let 'Y' be the number of milligrams of sulphide found at the end of this operation. The difference between quantity 'X' and quantity 'Y' must not exceed 3 %.

6.1.4. Pass nitrogen (4.8) through for 15 minutes, at a rate of two bubbles per second, in order to expel the air contained in the round-bottom flask (5.2).

6.1.5. Heat the round-bottom flask to 85 ± 5 ^{o}C.

6.1.6. Stop the nitrogen (4.8) stream and add 40 ml of hydrochloric acid (4.1) drop by drop.

6.1.7. Turn the nitrogen (4.8) stream on again when nearly all the acid has been transferred, leaving a minimum liquid seal to prevent leakage of hydrogen sulphide.

6.1.8. Cease heating after 30 minutes. Allow the flask (5.2) to cool and continue to pass the nitrogen (4.8) stream through for at least one-and-a-half hours.

6.2. Titration

6.2.1. Filter the cadmium sulphide through a long-stem funnel (5.4).

6.2.2. Rinse the conical flasks (5.3) first with the ammonia solution (4.9) and pour on the filter. Then rinse with distilled water and use the water to wash the precipitate retained by the filter.

6.2.3. Complete the washing of the precipitate with 100 ml of water.

6.2.4. Place the paper filter in the first conical flask that contained the precipitate. Add 25 ml (n_1) of the iodine solution (4.3), approximately 20 ml of hydrochloric acid (4.1) and 50 ml of distilled water.

6.2.5. Determine the excess iodine using the sodium thiosulphate solution (n_2) (4.2).

7. CALCULATON

The sulphide content of the sample, expressed as sulphur, as percentage by mass, is calculated by the following formula:

$$\% \text{ sulphur} = \frac{32(n_1 x_1 - n_2 x_2)}{20\,m}$$

where:

n_1 = the number (in millilitres) of iodine standard solution (4.3) used,
x_1 = the molarity of this solution,

n_2 = the number (in millilitres) of the sodium thiosulphate standard solution (4.2),
x_2 = the molarity of this solution,
m = the mass (in grams) of the test sample.

8. REPEATABILITY[1]

For a sulphide content of about 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 0,2 % (m/m).

(**1**) Norm ISO 5725.