E44 - STEARIC ACID

DEFINITION

Mixture consisting mainly of stearic (octadecanoic) acid ($C_{18}H_{36}O_2$; M_r 284.5) and palmitic (hexadecanoic) acid ($C_{16}H_{32}O_2$; M_r 256.4) obtained from fats or oils of vegetable or animal origin.

Content:

Stearic acid 50	*Stearic acid*: 40.0 per cent to 60.0 per cent. *Sum of the contents of stearic and palmitic acids*: minimum 90.0 per cent.
Stearic acid 70	*Stearic acid*: 60.0 per cent to 80.0 per cent. *Sum of the contents of stearic and palmitic acids*: minimum 90.0 per cent.
Stearic acid 95	*Stearic acid*: minimum 90.0 per cent. *Sum of the contents of stearic and palmitic acids*: minimum 96.0 per cent.

IDENTIFICATION

A. It complies with the test for freezing point (see Tests).

B. Acid value: 194 to 212.

Dissolve 0.5 g of the substance to be examined (*m* g), in 50 ml of a mixture of equal volumes of ethanol (96 per cent) and light petroleum, previously neutralised with 0.1 M potassium hydroxide or 0.1 M sodium hydroxide, using 0.5 ml of phenolphthalein solution as indicator. If necessary, heat to about 90 °C to dissolve the substance to be examined. When the substance to be examined has dissolved, titrate with 0.1 M potassium hydroxide or 0.1 M sodium hydroxide until the pink colour persists for at least 15 s (*n* ml of titrant). When heating has been applied to aid dissolution, maintain the temperature at about 90 °C during the titration.

$$I_A = 5.611\ n/m$$

C. Examine the chromatograms obtained in the assay.

Results : the retention times of the principal peaks in the chromatogram obtained with the test

solution are approximately the same as those of the principal peaks in the chromatogram obtained with the reference solution.

TESTS

Acidity. Melt 5.0 g, shake for 2 min with 10 ml of hot carbon dioxide-free water, cool slowly and filter. To the filtrate add 0.05 ml of methyl orange solution. No red colour develops.

Iodine value. See Table 1.

Introduce 1.0 g of the substance to be examined (m g) into a 250 ml flask fitted with a ground-glass stopper and previously dried or rinsed with glacial acetic acid, and dissolve it in 15 ml of chloroform unless otherwise prescribed. Add very slowly 25.0 ml of iodine bromide solution. Close the flask and keep it in the dark for 30 min unless otherwise prescribed, shaking frequently. Add 10 ml of a 100 g/l solution of potassium iodide and 100 ml of water. Titrate with 0.1 M sodium thiosulphate, shaking vigorously until the yellow colour is almost discharged. Add 5 ml of starch solution and continue the titration adding the 0.1 M sodium thiosulphate dropwise until the colour is discharged (n_1 ml of 0.1 M sodium thiosulphate). Carry out a blank test under the same conditions (n_2 ml of 0.1 M sodium thiosulphate).

$$I_I = 1.269\ (n_2 - n_1) / m$$

Freezing point. See Table 1.

The apparatus consists of a test-tube about 25 mm in diameter and 150 mm long placed inside a test-tube about 40 mm in diameter and 160 mm long. The inner tube is closed by a stopper which carries a thermometer about 175 mm long and graduated in 0.2 °C fixed so that the bulb is about 15 mm above the bottom of the tube. The stopper has a hole allowing the passage of the stem of a stirrer made from a glass rod or other suitable material formed at one end into a loop of about 18 mm overall diameter at right angles to the rod. The inner tube with its jacket is supported centrally in a 1-litre beaker containing a suitable cooling liquid to within 20 mm of the top. A thermometer is supported in the cooling bath.

Place in the inner tube sufficient quantity of the liquid or previously melted substance to be examined, to cover the thermometer bulb and determine the approximate freezing point by cooling rapidly. Place the inner tube in a bath about 5 °C above the approximate freezing point until all but the last traces of crystals are melted. Fill the beaker with water or a saturated solution of sodium chloride, at a temperature about 5 °C lower than the expected freezing point, insert the inner tube into the outer tube, ensuring that some seed crystals are present, and stir thoroughly until solidification takes place. Note the highest temperature observed during solidification. Alternatively, the Freezing point is measured by the following method.

1) Apparatus

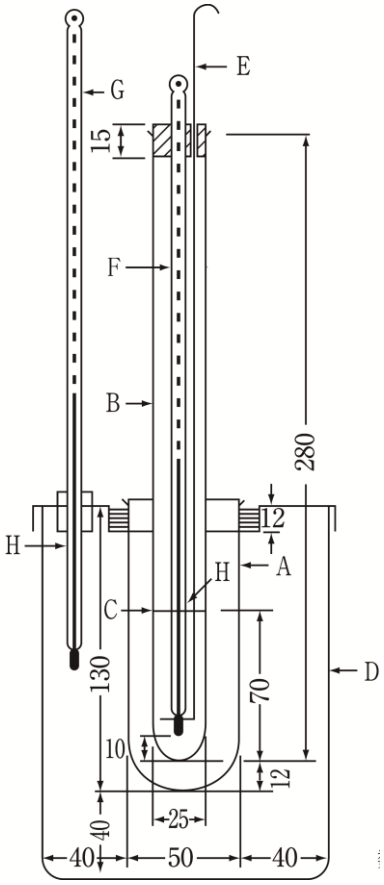
Use the apparatus illustrated in Figure 1.

2) Procedure

Place in the sample container B sufficient quantity of the liquid or previously melted substance to be examined, to the marked line C. Adjust the immersion line H of thermometer F to the same level of the meniscus of the sample and determine the approximate freezing point by cooling rapidly.

Place the sample container B in a bath about 5 °C above the approximate freezing point until all but the last traces of crystals are melted. Fill the bath D with water or a saturated solution of sodium chloride, at a temperature about 5 °C lower than the expected freezing point, insert the sample container B into the cylinder A, ensuring that some seed crystals are present, and stir thoroughly until solidification takes place. Note the highest temperature observed during solidification.

Figure 1.



A: Cylinder made of glass (the tube is painted with silicone oil on both sides of the wall to prevent clouding).
B: Sample container (a hard glass test tube, which is painted with silicone oil to prevent clouding, except at the region of the wall in contact with the sample; insert it into cylinder A, and fix with cork stopper).
C: A marked line.
D: Bath made of glass or plastics.
E: Stirring rod made of glass or stainless steel (3 mm in diameter, the lower end part of it is bent to make a loop, about 18 mm in diameter).
F: Thermometer with an immersion line.
G: Thermometer with an immersion line or a total immersion thermometer.
H: Immersion line

Table 1.

Type	**Iodine value**	**Freezing point (°C)**
Stearic acid 50	maximum 4.0	53 - 59
Stearic acid 70	maximum 4.0	57 - 64
Stearic acid 95	maximum 1.5	64 - 69

ASSAY

Gas chromatography: use the normalisation procedure.

Test solution. In a conical flask fitted with a reflux condenser, dissolve 0.100 g of the substance to be examined in 5 ml of *boron trifluoride-methanol solution*. Boil under reflux for 10 min. Add 4.0 ml of *heptane* through the condenser and boil again under reflux for 10 min. Allow to cool. Add 20 ml of a saturated solution of *sodium chloride*. Shake and allow the layers to separate. Remove about 2 ml of the organic layer and dry it over 0.2 g of *anhydrous sodium sulphate*. Dilute 1.0 ml of this solution to 10.0 ml with *heptane*.

Reference solution. Prepare the reference solution in the same manner as the test solution using 50 mg of *palmitic acid CRS* and 50 mg of *stearic acid CRS* instead of the substance to be examined.

Column:

— *material*: fused silica;

— *size*: *l* = 30 m, Ø = 0.32 mm;

— *stationary phase*: *macrogol 20 000* (film thickness 0.5 µm).

Carrier gas: *helium for chromatography*.

Flow rate: 2.4 ml/min.

Temperature:

	Time (min)	**Temperature (°C)**
Column	0 – 2 2 – 36 36 - 41	70 70 → 240 240
Injection port		220
Detector		260

Detection: flame ionisation.

Injection: 1 µl.

Relative retention with reference to methyl stearate : methyl palmitate = about 0.9.

System suitability : reference solution:

— *resolution*: minimum 5.0 between the peaks due to methyl stearate and methyl palmitate;

— *relative standard deviation*: maximum 3.0 per cent for the areas of the peaks due to methyl palmitate and methyl stearate, determined on 6 injections; maximum 1.0 per cent for the ratio of the areas of the peaks due to methyl palmitate to the areas of the peaks due to methyl stearate, determined on 6 injections.

LABELLING

The label states the type of stearic acid (50, 70, 95).

REAGENTS

Phenolphthalein solution. A 10 g/l solution of phenolphthalein in ethanol (96 per cent).

Light petroleum. A clear, colourless, flammable liquid without fluorescence, practically insoluble in water, miscible with ethanol (96 per cent).
d20,20: about 0.720.
Distillation range: 100 °C to 120 °C.
Water: maximum 0.03 per cent.

Boron trifluoride-methanol solution. A 140 g/l solution of boron trifluoride in methanol.