

**PHARMACOPOEIAL DISCUSSION GROUP**

**SIGN-OFF DOCUMENT**

**CODE: E-44**

**NAME: Stearic Acid**

**REVISION 2**

**Harmonised attributes**

<b>Attribute</b>	<b>EP</b>	<b>JP</b>	<b>USP</b>
Definition*	+	+	+
Identification A	+	+	+
Identification B	+	+	+
Identification C	+	+	+
Acidity	+	+	+
Iodine value	+	+	+
Freezing point	+	+	+
Assay	+	+	+
Labelling (type of stearic acid)	+	+	+

\* USP stipulates the note [Note—Stearic Acid labeled solely for external use is exempt from the requirement that it be prepared from edible sources.]

**Legend**

+ will adopt and implement ; – will not stipulate

**Non-harmonised attributes**

Characters/description, Residue on ignition (JP/USP), Containers and storage/Packaging and storage (JP/USP).

**Local requirements**

<b>EP</b>	<b>JP</b>	<b>USP</b>
Appearance, Functionality-Related Characteristics (Particle-size distribution, Specific surface area)	Heavy metals	Color of Solution, Labeling (If it is for external use only, the labeling so indicates.)

**Reagents and reference materials**

Each pharmacopoeia will adapt the text to take account of local reference materials and reagent specifications.

Each pharmacopoeia will consider actual titrant concentration in equations according to their local rules of calculation for titration.

**European Pharmacopoeia**

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*KEVIN MORSE*

Date

*9-NOV-2021*

## E44 - STEARIC ACID

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### 3 DEFINITION

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

### 7 *Content:*

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

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### 9 IDENTIFICATION

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

11 B. Acid value: 194 to 212.

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

19

20

$$I_A = 5.611 n/m$$

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22 C. Examine the chromatograms obtained in the assay.

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

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

## 26 TESTS

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

29 **Iodine value.** See Table 1.

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

$$I_1 = 1.269 (n_2 - n_1) / m$$

42 **Freezing point.** See Table 1.

43 The apparatus consists of a test-tube about 25 mm in diameter and 150 mm long placed inside a  
44 test-tube about 40 mm in diameter and 160 mm long. The inner tube is closed by a stopper which  
45 carries a thermometer about 175 mm long and graduated in 0.2 °C fixed so that the bulb is about  
46 15 mm above the bottom of the tube. The stopper has a hole allowing the passage of the stem of a  
47 stirrer made from a glass rod or other suitable material formed at one end into a loop of about 18  
48 mm overall diameter at right angles to the rod. The inner tube with its jacket is supported  
49 centrally in a 1-litre beaker containing a suitable cooling liquid to within 20 mm of the top. A  
50 thermometer is supported in the cooling bath.

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

59 1) Apparatus

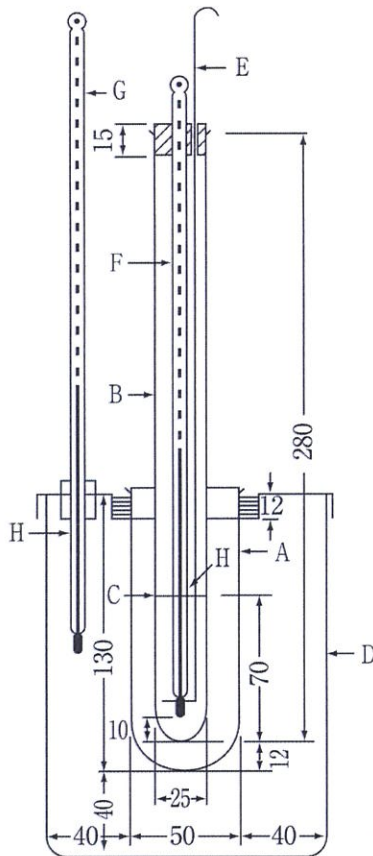
60 Use the apparatus illustrated in Figure 1.

61 2) Procedure

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

66 Place the sample container B in a bath about 5 °C above the approximate freezing point until all  
 67 but the last traces of crystals are melted. Fill the bath D with water or a saturated solution of  
 68 sodium chloride, at a temperature about 5 °C lower than the expected freezing point, insert the  
 69 sample container B into the cylinder A, ensuring that some seed crystals are present, and stir  
 70 thoroughly until solidification takes place. Note the highest temperature observed during  
 71 solidification.  
 72  
 73

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

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 KM PD

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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

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79 ASSAY

80 Gas chromatography: use the normalisation procedure.

81 *Test solution.* In a conical flask fitted with a reflux condenser, dissolve 0.100 g of the substance  
82 to be examined in 5 ml of *boron trifluoride-methanol solution*. Boil under reflux for 10 min. Add  
83 4.0 ml of *heptane* through the condenser and boil again under reflux for 10 min. Allow to cool.  
84 Add 20 ml of a saturated solution of *sodium chloride*. Shake and allow the layers to separate.  
85 Remove about 2 ml of the organic layer and dry it over 0.2 g of *anhydrous sodium sulphate*.  
86 Dilute 1.0 ml of this solution to 10.0 ml with *heptane*.

87 *Reference solution.* Prepare the reference solution in the same manner as the test solution using  
88 50 mg of *palmitic acid* and 50 mg of *stearic acid* instead of the substance to be examined.

89 *Column:*

90 — *material:* fused silica;

91 — *size:*  $l = 30$  m,  $\varnothing = 0.32$  mm;

92 — *stationary phase:* *macrogol 20 000* (film thickness 0.5  $\mu$ m).

93 *Carrier gas:* *helium for chromatography*.

94 *Flow rate:* 2.4 ml/min.

95

96 *Temperature:*

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

97 *Detection:* flame ionisation.

98 *Injection:* 1 µl.

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

100 *System suitability* : reference solution:

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

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

106 LABELLING

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

108

109 REAGENTS

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

111  
112 **Light petroleum.** A clear, colourless, flammable liquid without fluorescence, practically  
113 insoluble in water, miscible with ethanol (96 per cent).

114 d<sub>20,20</sub>: about 0.720.

115 Distillation range: 100 °C to 120 °C.

116 Water: maximum 0.03 per cent.

117

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