PHARMACOPOEIAL DISCUSSION GROUP

Revision 1 Stage 5B

E-42 RICE STARCH

Harmonised attributes

Attribute	EP	JP	USP
Definition	+	+	+
Identification			
A	+	+	+
B	+	+	+
C	+	+	+
pH	+	+	+
Iron	+	+	+
Oxidising substances	+	+	+
Sulfur dioxide	+	+	+
Loss on drying	+	+	+
Sulphated ash	+	+	+
Microbial contamination	+	-	+

Legend
+ will adopt and implement ; – will not stipulate

Non-harmonised attributes

Characters/Description, Foreign matter, Storage/Containers and storage/Packaging and storage

Local requirements

Absence of *Salmonella* (EP)

Reagents and reference materials

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

Date : Nov. 6, 2013 Signatures :

European Pharmacopoeia

for Takeyuki Satou
Japanese Pharmacopoeia

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United States Pharmacopeia

E-42 RICE STARCH

Oryzae amylum

DEFINITION

Rice starch is obtained from the caryopsis of *Oryza sativa* L.

IDENTIFICATION

A. Examined under a microscope using a mixture of equal volumes of glycerol and water, it presents polyhedral, simple grains 1 µm to 10 µm, mostly 4 µm to 6 µm, in size. These simple grains often gather in ellipsoidal, compound grains 50 µm to 100 µm in diameter. The granules have a poorly visible central hilum and there are no concentric striations. Between orthogonally orientated polarising plates or prisms, the starch granules show a distinct black cross intersecting at the hilum.

B. Suspend 1 g in 50 mL of water, boil for 1 min and cool. A thin, cloudy mucilage is formed.

C. To 1 mL of the mucilage obtained in identification test B add 0.05 mL of iodine solution. An orange-red to dark-blue colour is produced which disappears on heating.

TESTS

pH. Shake 5.0 g with 25.0 mL of freshly boiled and cooled water for 60 s. Allow to stand for 15 min. The pH of the solution is 5.0 to 8.0.

Iron. Shake 1.5 g with 15 ml of dilute hydrochloric acid (1 in 5). Filter. The filtrate complies with the limit test for iron (10 ppm).

Procedure — To 10 ml of the filtrate, add 2 ml of a 200 g/l solution of citric acid and 0.1 ml of thioglycollic acid. Mix, make alkaline with ammonia and dilute to 20 ml with water. Prepare a standard in the same manner, using 10 ml of iron standard solution (1 ppm Fe). After 5 min, any pink colour in the test solution is not more intense than in the standard.

Oxidising substances. Transfer 4.0 g to a glass-stoppered, 125 ml conical flask and add 50.0 ml of water. Insert the stopper and swirl for 5 min. Transfer to a glass-stoppered 50 ml centrifuge tube and centrifuge. Transfer 30.0 ml of the clear supernatant liquid to a glass-stoppered 125 ml conical flask. Add 1 ml of glacial acetic acid and 0.5 g to 1.0 g of potassium iodide. Insert the stopper, swirl, and allow to stand for 25 min to 30 min in the dark. Add 1 ml of starch solution and titrate with 0.002 M sodium thiosulphate until the starch-iodine colour

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disappears. Carry out a blank determination. Not more than 1.4 ml of 0.002 M sodium thiosulphate is required (0.002 per cent, calculated as H_2O_2).

1 ml of 0.002 M sodium thiosulphate is equivalent to 34 µg of oxidising substances, calculated as hydrogen peroxide.

Sulphur dioxide: Not more than 50 ppm.

Procedure — Introduce 150 ml of water into the flask (*A*) (see Figure) and pass carbon dioxide through the whole system for 15 min at a rate of 100 ± 5 ml/min. To 10 ml of dilute hydrogen peroxide solution (1 in 10) add 0.15 ml of a 1 g/l solution of bromophenol blue in alcohol (20 per cent *V/V*). Add 0.1 M sodium hydroxide until a violet-blue colour is obtained, without exceeding the end-point. Place the solution in the test-tube (D). Without interrupting the stream of carbon dioxide, remove the funnel (B) and introduce through the opening into the flask (*A*) 25.0 g (*m* g) of the substance to be examined with the aid of 100 ml of water. Close the tap of the funnel and add 80 ml of dilute hydrochloric acid (1 in 5) to the funnel. Open the tap of the funnel to permit the hydrochloric acid solution to flow into the flask, guarding against the escape of sulfur dioxide into the funnel by closing the tap before the last few mL of hydrochloric acid solution drain out. Boil for 1 h. Open the tap of the funnel and stop the flow of carbon dioxide and also the heating and the cooling water. Transfer the contents of the test-tube with the aid of a little water to a 200 ml wide-necked, conical flask. Heat on a water-bath for 15 min and allow to cool. Add 0.1 ml of a 1 g/l solution of bromophenol blue in alcohol (20 per cent *V/V*) and titrate with 0.1 M sodium hydroxide until the colour changes from yellow to violet-blue (V_1 ml). Carry out a blank titration (V_2 ml). Calculate the content of sulphur dioxide in parts per million from the expression:

$$32030 \times (V_1 - V_2) \times n / m$$

n = molarity of the sodium hydroxide solution used as titrant.

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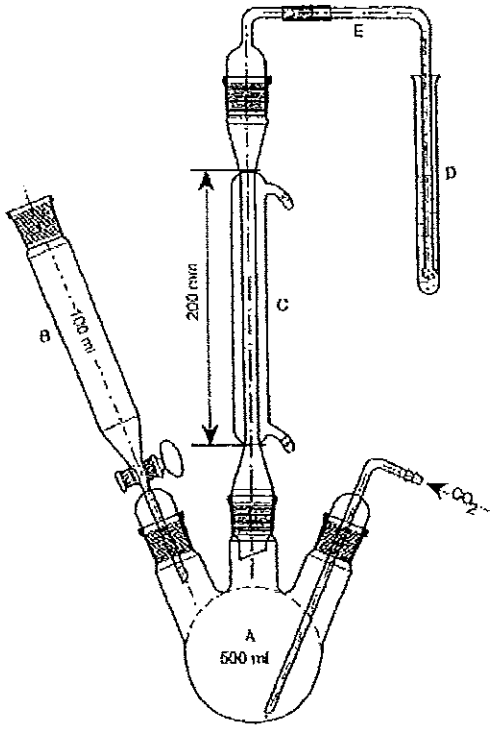

Figure — *Apparatus for the determination of sulphur dioxide*

Loss on drying. Not more than 15.0 per cent, determined on 1.00 g by drying in an oven at 130 °C for 90 min.

Sulfated ash. Not more than 0.6 per cent, determined on 1.0 g.

Microbial contamination (internationally harmonized methods) - TAMC: acceptance criterion 10^3 CFU/g. TYMC: acceptance criterion 10^2 CFU/g. Absence of *Escherichia coli*.

REAGENTS

Iodine solution — To 10.0 ml of 0.05 M iodine solution, add 0.6 g of potassium iodide and dilute to 100.0 ml with water. Prepare immediately before use.

Starch solution — Triturate 1.0 g of soluble starch with 5 ml of water and whilst stirring pour the mixture into 100 ml of boiling water containing 10 mg of mercuric iodide.
Carry out the test for sensitivity each time the reagent is used.
Test for sensitivity. To a mixture of 1 ml of the *starch solution* and 20 ml of water, add about 50 mg of potassium iodide and 0.05 ml of *iodine solution*; the solution is blue.

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