

CORN STARCH

Corn Starch consists of the starch granules separated from the mature grain of corn [*Zea mays* Linnè (Fam. Gramineae)].

Identification—

A: Under a microscope, using a mixture of glycerin and water (1:1) as a mounting agent, it appears as angular polyhedral, rounded, or spheroidal granules of irregular sizes up to about 35 μm in diameter. The central hilum consists of a distinct cavity or two- to five-rayed cleft, and there are no concentric striations. Between orthogonally oriented polarizing plates or prisms, the starch granules show a distinct black cross intersecting at the hilum.

B: Suspend 1 g of it in 50 ml of water, boil for 1 minute, 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 color is produced, which disappears on heating.

TESTS

pH— Prepare a slurry by weighing 5.0 g of Corn Starch, transferring to a suitable nonmetallic container, and adding 25.0 ml of freshly boiled and cooled water. Agitate continuously at a moderate rate for 1 min. Stop the agitation and allow to stand for 15 minutes. Determine the pH to the nearest 0.1 unit: the pH, determined potentiometrically, is between 4.0 and 7.0.

Loss on drying— Dry about 1 g, accurately weighed, at 130° for 90 min: it loses not more than 15.0% of its weight.

Residue on ignition— not more than 0.6%, determined on a 1.0 g test specimen.

Limit of iron— Shake 1.5 g of Corn Starch with 15 ml of 2 N hydrochloric acid, and filter. Transfer 10 ml of the filtrate to a test tube, add 2 ml of citric acid solution (2 in 10), 0.1 ml of thioglycolic acid, and mix. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, dilute with water to 20 ml, and mix (*Test Solution*). Immediately before use, dilute an accurately measured volume of a *Standard Iron*

37 *Solution* quantitatively with water to obtain a *Diluted Standard Iron Solution* containing
38 the equivalent of 1 µg of iron per ml. Prepare the *Standard Solution* by transferring 10
39 ml of the *Diluted Standard Iron Solution* to a test tube and proceeding in the same
40 manner as directed for the preparation of the *Test Solution*, beginning with “add 2 ml of
41 citric acid solution (2 in 10).” After 5 minutes, any pink color in the *Test Solution* is not
42 more intense than that in the *Standard Solution*, corresponding to a limit of 10 µg of iron
43 per g.

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45 **Limit of oxidizing substances**— Transfer 4.0 g to a glass-stoppered, 125-ml conical flask,
46 and add 50.0 ml of water. Insert the stopper, and swirl for 5 minutes. Transfer to a glass-
47 stoppered, 50-ml centrifuge tube, and centrifuge to clarify. Transfer 30.0 ml of the clear
48 supernatant liquid to a glass-stoppered, 125-ml conical flask. Add 1 ml of glacial acetic
49 acid and 0.5 g to 1.0 g of potassium iodide. Insert the stopper, swirl, and allow to stand
50 for 25 to 30 minutes in the dark. Add 1 ml of *starch solution*, and titrate with 0.002 N
51 sodium thiosulfate to the disappearance of the starch-iodine color. Perform a blank
52 determination, and make any necessary correction. Each ml of 0.002 N sodium thiosulfate
53 is equivalent to 34 µg of oxidant, calculated as hydrogen peroxide. Not more than 1.4 ml
54 of 0.002 N sodium thiosulfate is required (20 µg per g, calculated as H₂O₂).

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56 **Limit of sulfur dioxide**— Not more than 50 µg per g.

57 *Reagents*—

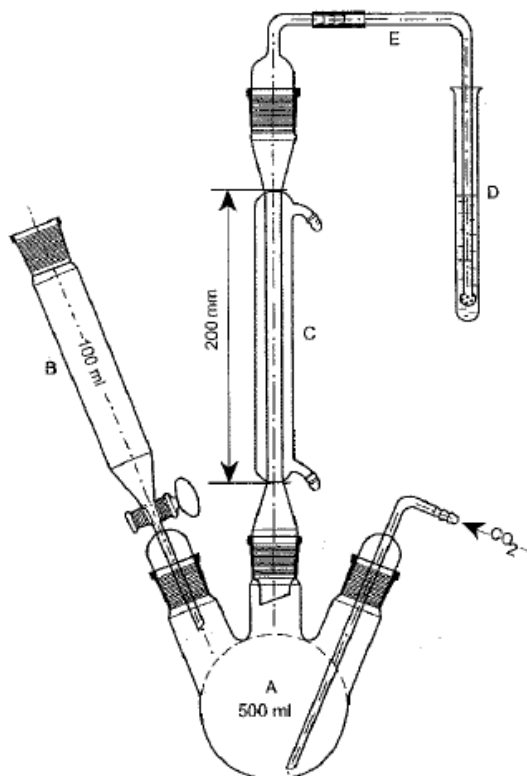
58 *Carbon dioxide*— Use carbon dioxide, with a flow regulator that will maintain a flow
59 of 100 ± 5 ml per minute.

60 *Bromophenol blue indicator solution*— Dissolve 100 mg of bromophenol blue in 100
61 ml of dilute alcohol (1 in 5), and filter if necessary.

62 *Hydrogen peroxide solution*— Dilute 30% hydrogen peroxide with water to obtain a
63 3% solution. Just before use, add 3 drops of *Bromophenol blue indicator solution*, and
64 neutralize to a violet-blue endpoint with 0.01 N sodium hydroxide. Do not exceed the
65 endpoint.

66 *Apparatus*— In this test, the sulfur dioxide is released from the test specimen in a
67 boiling acid medium and is removed by a stream of carbon dioxide. The separated gas is
68 collected in a dilute hydrogen peroxide solution where the sulfur dioxide is oxidized to
69 sulfuric acid and titrated with standard alkali. A suitable apparatus for sulfur dioxide
70 determination is shown in the accompanying diagram. The apparatus consists essentially
71 of a 500-ml three-neck, round-bottom, boiling flask, *A*, a separatory funnel, *B*, having a
72 capacity of 100 ml or greater, a gas inlet tube of sufficient length to permit introduction

73 of the carbon dioxide within 2.5 cm of the bottom of the boiling flask, a reflux condenser,
 74 C, having a jacket length of 200 mm, and a delivery tube, E, connecting the upper end of
 75 the reflux condenser to the bottom of a receiving test tube, D. Apply a thin film of
 76 stopcock grease to the sealing surfaces of all of the joints except the joint between the
 77 separatory funnel and the boiling flask, and clamp the joints to ensure tightness.
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Figure — *Apparatus for the determination of sulfur dioxide*

82 *Procedure*— Add 150 ml of water to the boiling flask. Close the stopcock of the
 83 separatory funnel, and begin the flow of carbon dioxide at a rate of 100 ± 5 ml per minute
 84 through the *Apparatus*. Start the condenser coolant flow. Add 10 ml of *Hydrogen peroxide*
 85 *solution* to the receiving test tube. After 15 minutes, without interrupting the flow of
 86 carbon dioxide, remove the separatory funnel from the boiling flask and transfer 25.0 g
 87 of test specimen into the boiling flask with the aid of 100 ml of water. Apply stopcock
 88 grease to the outer joint of the separatory funnel, and replace the separatory funnel in the
 89 boiling flask. Close the stopcock of the separatory funnel, and add 80 ml of 2 N
 90 hydrochloric acid to the separatory funnel. Open the stopcock of the separatory funnel to
 91 permit the hydrochloric acid solution to flow into the boiling flask, guarding against the
 92 escape of sulfur dioxide into the separatory funnel by closing the stopcock before the last

93 few ml of hydrochloric acid drain out. Boil the mixture for 1 hour. Remove the receiving
94 test tube, and transfer its contents to a 200-ml wide-necked, conical flask. Rinse the
95 receiving test tube with a small portion of water, add the rinsing to the 200-ml conical
96 flask, and mix. Heat on a water bath for 15 minutes and allow to cool. Add 0.1 ml of
97 *Bromophenol blue indicator solution*, and titrate the contents with 0.1 N sodium
98 hydroxide until the color changes from yellow to violet-blue, with the color change lasting
99 for at least 20 seconds. Perform a blank determination and make any necessary correction.
100 Calculate the content, in μg per g, of sulfur dioxide in the test specimen taken by the
101 formula:

$$102 \quad 1000(32.03)VN/W,$$

103 in which 32.03 is the milliequivalent weight of sulfur dioxide, V is the volume, in mL, of
104 titrant consumed, N is the normality of the titrant, and W is the weight, in g, of test
105 specimen taken.

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

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111 REAGENTS

112 *Iodine solution*— Dissolve 12.7 g of iodine and 20 g of potassium iodide in water and
113 dilute to 1000.0 ml with water. To 10.0 ml of this solution, add 0.6 g of potassium iodide
114 and dilute to 100.0 ml with water. Prepare immediately before use.

115 *Standard Iron Solution*— Dissolve 863.4 mg of ferric ammonium sulfate [$\text{FeNH}_4(\text{SO}_4)_2$
116 $\cdot 12\text{H}_2\text{O}$] in water, add 10 ml of 2 N sulfuric acid, and dilute with water to 100.0 ml. Pipet
117 10 ml of this solution into a 1000-ml volumetric flask, add 10 ml of 2 N sulfuric acid,
118 dilute with water to volume, and mix. This solution contains the equivalent of 0.01 mg
119 (10 μg) of iron per ml.

120 *Starch solution*— Mix 1 g of soluble starch with 10 mg of red mercuric iodide and
121 sufficient cold water to make a thin paste. Add 200 ml of boiling water, and boil for 1
122 minute with continuous stirring. Cool, and use only the clear solution. [NOTE—
123 Commercially available, stabilized starch indicator solutions may be used.]