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National Food Safety Standard - Food additive - Sodium starch octenyl succinate

食品安全国家标准

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Annex A

Test methods

A.1 General

Unless otherwise specified, the reagents and water used in this Standard refer to analytical reagents and grade 3 water specified in GB/T 6682. Unless otherwise specified, the standard titration solutions used in the test, the standard solutions, preparations and products used in the determination of impurities are prepared according to the provisions of GB/T 601, GB/T 602 and GB/T 603. When the solution used in the test does not indicate which solvent is used for preparation, it refers to aqueous solution.

A.2 Identification test

A.2.1 Iodine staining

ADD 1 g of sample to 20 mL of water, to make a suspension; ADD a few drops of iodine solution. The color is dark blue to brown red.

A.2.2 Copper reduction

WEIGH 2.5 g of sample; PLACE it in a long-necked flask; ADD 10 mL of dilute hydrochloric acid (0.82 mol/L) and 70 mL of water; MIX evenly; REFLUX for 3 h; COOL. TAKE 0.5 mL of the cooled solution; ADD 5 mL of hot alkaline copper tartrate test solution; it produces a large amount of red precipitate.

To prepare alkaline copper tartrate test solution, proceed as follows:

a) Solution A: TAKE 34.66 g of copper sulfate crystals ($CuSO_4 \cdot 5H_2O$). The crystals shall show no signs of weathering or moisture absorption. ADD water to dissolve and dilute to 500 mL. Keep this solution in a small sealed container;

b) Solution B: TAKE 173 g of potassium sodium tartrate crystals ($KNaC_4H_4O_6 \cdot 4H_2O$) and 50 g of sodium hydroxide (NaOH); ADD water to dissolve and dilute to 500 mL. Keep this solution in a small alkali-resistant container;

c) MIX the same volume of solution A and solution B to obtain alkaline copper tartrate test solution.

A.3 Determination of sulfur dioxide residue

A - Built-in adapter;
B - Separating funnel;
C - Round-bottom flask;
D - Gas internal takeover;
E - Condenser;
F - Connecting ball;
G - Receiver.

Figure A.1 -- Diagram of sulfur dioxide test apparatus

The apparatus in Figure A.1 is used to selectively transfer sulfur dioxide from sample to 3 % hydrogen peroxide solution in boiling aqueous hydrochloric acid solution. The apparatus is easier to connect than conventional apparatus. Since the height of the 3 % hydrogen peroxide solution is above the ball tip, the back pressure in the apparatus is unavoidable, but part F can reduce the back pressure to the lowest possible level, thereby reducing the possibility of sulfur dioxide loss due to leakage.

NOTE: In Figure A.1, part D needs to be equipped with a hose for connection. If polyethylene and quartz hoses are used, they shall be pre-cooked before use in this procedure.

The entire apparatus shall be connected as shown in Figure A.1. Except for the connection between the separatory funnel and the flask, the sealing surfaces of all other connections shall be coated with a thin layer of piston lubricant. All connectors shall be tightly clamped to ensure tightness during the analysis. The separatory funnel B, of which the volume shall be greater than or equal to 100 mL. It shall be equipped with a built-in adapter A equipped with hose connections to ensure that a certain pressure is maintained above the internal solution. (It is not recommended to use a constant pressure titration funnel, because the condensate may contain sulfur dioxide, which will adhere to the inner wall of the funnel or the tube wall). The round-bottom flask C, of which the volume is 1000 mL, with three 24/40 mm conical ports. The gas internal takeover D shall have a sufficient length to ensure that the introduced nitrogen can reach 2.5 cm at the bottom of the flask. The condenser E, of which the jacket length shall be 300 mm. The connecting ball F is a glass piece made according to the requirements of Figure A.2, which is the same size as the 50 mL measuring barrel. The 3 % hydrogen peroxide solution is placed in the receiver G, of which the inner diameter is 2.5 cm and the length is 18 cm.

A.3.1.3.2 Preparation for determination

CONNECT the apparatus according to the requirements of Figure A.1. The flask shall be connected to a heater with adjustable power. ADD 400 mL of distilled water to the flask. CLOSE the valve of the separatory funnel; ADD 90 mL of 4 mol/L hydrochloric acid to the funnel. INTRODUCE nitrogen at a rate of 200 mL/min ± 10 mL/min. ACTIVATE the coolant in the condenser at the same time. ADD 30 mL of 3 % hydrogen peroxide solution that has been calibrated with standard solution TO receiver G. After 15 min, the apparatus and water will be completely deoxygenated, and the apparatus can be used for test of samples.

A.3.1.3.3 Distillation

REMOVE the separatory funnel; quantitatively ADD the ethanol solution of the sample to the flask. WIPE the conical connection clean with laboratory paper towels. APPLY piston lubricant to the external connection of the separatory funnel. REINSTALL the separatory funnel. After the connection is made, the nitrogen flow through the 3 % hydrogen peroxide solution shall be restored immediately, and the connection shall be checked to ensure that it is sealed.

The rubber ball above the separatory funnel is equipped with a valve to ensure that there is sufficient pressure above the hydrochloric acid solution. OPEN the separatory funnel valve and let the hydrochloric acid solution flow into the flask. Continue to ensure that there is sufficient pressure above the solution. If necessary, the valve can be temporarily closed to supplement the pressure. To prevent the loss of sulfur dioxide into the separatory funnel, the valve shall be closed before the last few milliliters flow out of the separatory funnel.

CONNECT the power supply for heating; CONTROL the heating speed so that the reflux liquid is 80 drops to 90 drops per minute. After 1.75 h of distillation, the content of the 1000 mL flask is cooled at the above reflux rate. And TRANSFER the content of the receiver G.

A.3.1.3.4 Titration

ADD 3 drops of methyl red indicator, TITRATE the above solution with standard titration until the yellow end point, ensuring that it does not fade within 20 s.

A.3.1.4 Calculation of results

The sulfur dioxide residue is calculated as the mass fraction w_1 of sulfur dioxide, expressed in milligrams per kilogram (mg/kg), and calculated according to formula (A.1):

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