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NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 1903.56-2022

National food safety standard - Food nutritional fortification substance - Sodium selenate

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1 Scope

This document applies to sodium selenite, a food nutritional fortification substance, of which sodium selenite is prepared from selenous acid and sodium hydroxide, and then it is obtained by oxidizing sodium selenite with hydrogen peroxide.

2 Chemical name, molecular formula, structural formula and

relative molecular mass

2.1 Chemical name

Sodium selenate.

2.2 Molecular formula

Na2SeO4

2.4 Relative molecular mass

188.94 (according to the international relative atomic mass in 2018)

3.1 Sensory requirements

Sensory requirements shall meet the requirements in Table 1.

Annex A Inspection methods

A.1 Warning According to the provisions of Chapter 6 of GB 12268-2012, sodium selenate

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belongs to item 6.1 toxic substance. Handle with care. Some reagents used in the inspection methods of this Standard are toxic or corrosive. Operate according to relevant national regulations. Use it with caution. If splashed on skin, rinse with water immediately.

Severe cases shall seek medical attention immediately. When using volatile acids, it shall be done in a fume hood.

A.2 General provisions The reagents and water used in this Standard refer to analytically pure reagents and grade three water specified in GB/T 6682 unless other requirements are specified. The standard solution, standard solution for impurity determination, preparations and products used in the test shall be prepared according to the provisions of GB/T 601,

GB/T 602 and GB/T 603 unless other requirements are specified. The solution used in the test refers to the aqueous solution when the solvent used for preparation is not indicated.

A.3 Identification test A.3.1 Reagents and materials A.3.1.1 Hydrochloric acid (rho1.19g/mL).

A.3.1.2 Sulfuric acid (rho1.80g/mL).

A.3.1.3 Sodium hydroxide.

A.3.1.4 Sodium thiosulfate solution (100g/L): Weigh 10g of sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$). Use water to dissolve. Set volume constant to 100mL.

A.3.1.5 Hydrochloric acid solution: 1+1.

A.3.1.6 Sulfuric acid solution: 1+1.

A.3.1.7 Sodium hydroxide solution (200g/L): Weigh 20g of sodium hydroxide. Use water to dissolve. Set volume constant to 100mL.

A.3.1.8 Hydrazine hydrochloride.

A.3.1.9 Platinum wire.

A.4.2.1 Selenium: Selenium (Se) content is not less than 99.99%.

A.4.2.2 Hydrochloric acid.

A.4.2.3 Nitric acid.

A.4.2.4 Sulfuric acid solution: 1+1.

A.4.2.5 Hydrochloric acid solution: 1+1.

A.4.2.6 Potassium iodide solution: 100g/L.

Weigh 10g of potassium iodide. Dissolve in water. Transfer all to a 100mL volumetric flask. Dilute to the scale. Shake well. Store in a brown bottle. This solution is prepared when it is needed.

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A.4.2.7 Starch indicator solution: 10g/L.

A.4.2.8 Sodium thiosulfate standard titration solution: c(Na2S2O3)~0.1mol/L.

A.4.2.9 Sodium thiosulfate standard titration solution: c(Na2S2O3)~0.01mol/L. Pipette 10.00mL of sodium thiosulfate standard titration solution (A.4.2.8) into a 100mL volumetric flask. Use water to set volume constant to the scale. Shake well.

A.4.3 Instruments and equipment A.4.3.1 Electronic balance: the resolution is 0.01g and 0.0001g.

A.4.3.2 Electric heating plate.

A.4.4 Analysis steps A.4.4.1 Determination of sodium selenite Weigh 5.0g of the specimen after determining the drying loss, accurate to 0.0001g.

Place in a 250mL Erlenmeyer flask. Add 50mL of water to dissolve. Add 10mL of sulfuric acid solution (A.4.2.4). Shake well. Add 2mL of potassium iodide solution (A.4.2.6). Shake well. Then add 5mL of starch indicator solution (A.4.2.7). Titrate with sodium thiosulfate standard titration solution (A.4.2.9) until the blue color disappears, which shall be the end point. If the consumed titration volume is above 10mL, the weight of the specimen to be weighed shall be appropriately reduced.

Conduct blank test at the same time. Except that the specimen is not added for the blank test, the types and amounts of other added reagents (except the standard titration solution) are the same as the test solution.

A.4.4.2 Determination of sodium selenate Weigh 1.2g of the specimen after determining the drying loss, accurate to 0.0001g.

Place in a 250mL beaker. After slowly adding 20mL of hydrochloric acid, cover with a watch glass or small beaker. Heat at low temperature for 30min. Do not boil the solution.

After the yellow-green upper layer of the solution dissipates, remove it and cool it down.

Transfer the solution to a 100mL volumetric flask. Use water to dilute to the scale.

Shake well.

Pipette 10.00mL of the solution. Place in a 250mL Erlenmeyer flask. Add 20mL of water to dissolve. Add 10mL of hydrochloric acid solution (A.4.2.5). Shake well. Use sodium thiosulfate standard titration solution (A.4.2.8) to titrate. Add 2mL of potassium iodide solution (A.4.2.6) near the end point and shake well. Then add 5mL of starch indicator solution (A.4.2.7). Continue to titrate till the blue color disappears, which shall be the end point.

Conduct the blank test at the same time. Except that the specimen is not added for the blank test, the type and amount of other added reagents (except the standard titration solution) are the same as the test solution.

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A.4.5 Result calculation The content of sodium selenite is calculated according to the mass fraction w1 of sodium selenite (Na2SeO3) according to the formula (A.1).

Where, V1 - The volume of sodium thiosulfate standard titration solution consumed by the specimen solution in A.4.4.1, in milliliters (mL);

V2 - The volume of sodium thiosulfate standard titration solution consumed by the blank solution in A.4.4.1, in milliliters (mL);

c1 - The concentration of sodium thiosulfate standard titration solution, in moles per liter (mol/L);

m1 - The weighted mass of the specimen after determining the drying loss in A.4.4.1, in grams (g);

M1 - The molar mass of sodium selenite (1/4Na2SeO3), in grams per mole (g/mol) (M1 =43.23).

The calculation result retains three significant figures. Take the arithmetic mean of the parallel determination results as the determination result. The absolute difference between two parallel determination results is not more than 0.005%.

The content of sodium selenate is calculated according to the mass fraction w2 of a multi-element standard solution certified by the state and awarded a standard substance certificate is used.

A.5.2.5 Standard solution (100mg/L): use a single-element standard stock solution that has been certified by the state and granted a standard substance certificate.

A.5.2.6 Sodium chloride.

A.5.2.7 Water: meet the specifications for the grade one water specified in GB/T 6682.

A.5.3 Instruments A.5.3.1 Inductively coupled plasma optical emission spectrometer (ICP-OES).

A.5.3.2 Electronic balance: the resolution is 0.0001g.

A.5.4 Analysis steps A.5.4.1 Preparation of specimen solution Weigh about 2g of specimen, accurate to 0.0001g. Place in a 25mL colorimetric tube.

Add 15mL of water to dissolve. Add 0.5mL of nitric acid. Then use water to set volume constant to the scale. Shake well.

Conduct blank test at the same time. Except that the specimen is not added for the blank test, the types and amounts of other reagents added are the same as the specimen solution.

A.5.4.2 Sodium salt matrix solution Weigh about 2g of sodium chloride, accurate to 0.0001g. Place in a 25mL colorimetric tube. Add 15mL of water to dissolve. Add 0.5mL of

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