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NATIONAL STANDARD OF THE

PEOPLE’S REPUBLIC OF CHINA

GB 1886.361-2022

National food safety standard - Food additive - Sodium copper chlorophyllin

食品安全国家标准 食品添加剂 叶绿素铜

Issued on: June 30, 2022 **Implemented on: December 30, 2022**

Issued by: National Health Commission of the People's Republic of China;
State Administration for Market Regulation.

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National food safety standard - Food additive - Sodium copper chlorophyllin

1 Scope

This document is applicable to sodium copper chlorophyllin, a food additive, that chlorophyll is extracted from silkworm sand or grass (such as tall fescue, lucerne, nettle), spirulina and other plants as raw materials, or directly uses chlorophyll as raw material, and then is prepare by copper substitution reaction. The solvents used are acetone, diazomethane, methanol, ethanol, isopropanol, n-hexane and (or) petroleum ether (boiling range is 90°C~120°C).

2 Molecular formula and relative molecular mass

2.1 Molecular formula

Copper chlorophyllin a ($C_{55}H_{72}CuN_4O_5$)

Copper chlorophyllin b ($C_{55}H_{70}CuN_4O_6$)

2.2 Relative molecular mass

Copper chlorophyllin a: 932.75 (according to 2018 international relative atomic mass)

Copper chlorophyllin b: 946.73 (according to 2018 international relative atomic mass)

3 Technical requirements

3.1 Sensory requirements

Sensory requirements shall meet the requirements of Table 1.

3.2 Physical and chemical indicators

Physical and chemical indicators shall meet the requirements of Table 2.

Annex A

Inspection methods

A.1 General

Unless otherwise specified, the reagents and water used in this Standard refer to analytically pure reagents and grade three water specified in GB/T 6682. The standard solutions, standard solutions used for determination of impurities, preparations and products shall be prepared in accordance with 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 is not specified.

A.2 Identification test

A.2.1 Maximum absorption peak and absorbance ratio range

Take the specimen solution in A.3.3.1 Determination of chlorophyll content. There are maximum absorption peaks in the two wavelength ranges of 405nm ± 3nm and 635nm ± 5nm. The absorbance ratio of the two measurements is between 3.2~4.0.

A.2.2 Copper ion test

A.2.2.1 Reagents and materials

A.2.2.1.1 Sulfuric acid.

A.2.2.1.2 Hydrochloric acid solution: 1+3.

A.2.2.1.3 Sodium diethyldithiocarbamate solution: 1g/L.

A.2.2.2 Analysis steps

Take 1g of specimen. Place in a crucible that has been burnt to constant weight at 800°C ± 25°C. Slowly heat until the specimen is completely carbonized. Cool the carbonized specimen. Use 0.5mL~1mL of sulfuric acid to wet the residue. Continue to heat until the sulfuric acid vapor escapes. Burn the residue to a heavy weight in a high temperature furnace at 800°C ± 25°C. Add 10mL of hydrochloric acid solution to the residue. Heat on the water bath to dissolve. After filtration, add water to 10mL. Take 5mL of the above solution. Add 0.5mL of sodium diethyldithiocarbamate solution. Produce a reddish-brown precipitate.

A.3 Determination of chlorophyll content

A.3.1 Reagents and materials

A.3.1.1 Anhydrous ether.

A.3.1.2 Disodium hydrogen phosphate solution (0.15mol/L): Weigh 53.7g of disodium hydrogen phosphate ($Na_2HPO_4 \cdot 12H_2O$). Add water to dissolve. Dilute and set volume to 1000mL.

A.3.1.3 Potassium dihydrogen phosphate solution (0.15mol/L): Weigh 20.4g of potassium dihydrogen phosphate (KH_2PO_4). Add water to dissolve. Dilute and set volume to 1000mL.

A.3.1.4 Phosphate buffer (pH 7.5): Measure disodium hydrogen phosphate solution (0.15mol/L) and potassium dihydrogen phosphate solution (0.15mol/L) respectively. Mix by volume ratio of 21:4.

A.3.1.5 Solution A: copper chloride-methanol solution (5%).

A.3.1.6 Solution B: sodium hydroxide-methanol solution (10%).

A.3.2 Instruments and equipment

A.3.2.1 Spectrophotometer.

A.3.2.2 Constant temperature water bath.

A.3.2.3 Separatory funnel (250mL).

A.3.3 Analysis steps

A.3.3.1 Preparation of specimen solution

Weigh 1g of specimen, to the nearest of 0.0002g. Place in an Erlenmeyer flask. Add 50mL~60mL of anhydrous ether to dissolve. Add 1mL of solution A. Heat to reflux for 15min in a constant temperature water bath at 50°C ± 2°C. Remove. Add 2mL of solution B. Shake well. Continue to heat to reflux for 20min in a constant temperature water bath at 50°C ± 2°C. Shake intermittently throughout the process. Make sure the reaction is complete. After reaction, remove the Erlenmeyer flask. Cool. Add a little water. Shake well. Transfer to a separatory funnel. Then use an appropriate amount of water to wash the Erlenmeyer flask in several times. The washing liquid is transferred to the separatory funnel. Shake well and let stand for at least 1h.

After the liquid in the separatory funnel is separated, filter the bottom liquid. Note that the interfacial layer remains in the separatory funnel. Do not filter down to avoid clogging filter paper. After filtration, rinse the upper acetyl layer and interface with water until the washed water layer is colorless. The washing liquid is merged into the lower filtrate. Discard the upper ether layer and interface. Use water to wash the filter paper several times until almost it is almost colorless. The washing liquid is combined into the lower filtrate. Keep all filtrates.

Transfer the filtrate to another separatory funnel. Use 40mL of anhydrous ether to shake and wash. Place still for 15min. Transfer the lower liquid to a 250mL volumetric flask.

5mL of nitric acid. Digest the specimen according to the operation steps of microwave digestion. Refer to B.1 for digestion conditions. Cool. Take out the digestion tank. Acid to about 1mL at 120°C~140°C. After the digestion tank is cooled, transfer the digest to a 25mL volumetric flask. Use a small amount of grade two water to wash the digestion tank 2~3 times. Combine washing solution in the volumetric flask. Use grade two water to set volume to the scale. Mix well for future use. At the same time, conduct reagent blank test.

A.4.3.2 Dry ashing

Weigh 0.1g of specimen, to the nearest of 0.0002g. Heat in a crucible over low heat. Carbonize to it is smokeless. Transfer to a 550°C muffle furnace. Conduct ashing for 3h~4h. Cool and take it out. For incompletely-ashed specimens, add several drops of nitric acid. Heat with small fire. Steam dry carefully. Then transfer to the 550°C muffle furnace. Continue ashing 1h~2h till the specimen is white grey. Cool and take out. Use an appropriate amount of nitric acid solution (1+1) to dissolve. Use water to set volume to 10mL. Mix well for future use. At the same time, conduct the blank test.

A.4.4 Analysis steps

Except for the specimen processing, other steps are determined according to the methods specified in GB 5009.13.

A.5 Determination of free copper (Cu)

A.5.1 Reagents and materials

Same with GB 5009.13.

A.5.2 Instruments and equipment

Same with GB 5009.13.

A.5.3 Specimen processing

Weigh 0.1g of specimen, to the nearest of 0.0002g. Place in an Erlenmeyer flask. Add 20mL of anhydrous ether to dissolve. Then add 100mL of water. Cover with the stopper. Shake 1min. Transfer to a 125mL separatory funnel. Place still for 30min. Use double-layer filter paper to filter the lower liquid in the separatory funnel. If the filtrate is colored, filter again with double-layer filter paper until the filtrate is colorless. The filtrate shall be the specimen solution.

A.5.4 Analysis steps

Except for the specimen processing, other steps are determined according to the methods specified in GB 5009.13.

A.6 Determination of solvent residues (dichloromethane, methanol, isopropanol,

n-hexane, n-heptane)

A.6.1 Reagents and materials

A.6.1.1 Water: Grade one water specified in GB/T 6682.

A.6.1.2 Standard product of test component: Dichloromethane, methanol, isopropanol, n-hexane, n-heptane. Chromatographically pure.

A.6.1.3 Internal standard product: 3-methyl-2-pentanone. Chromatographically pure.

A.6.1.4 N-methylpyrrolidone.

A.6.2 Instruments and equipment

Gas chromatograph: Equipped with hydrogen flame ionization detector (FID) and headspace specimen injector.

A.6.3 Reference chromatographic conditions

A.6.3.1 Chromatographic column: Quartz capillary column (Φ 0.53mm×30m). Coating is dimethyl polysiloxane. Thickness is 5μm. Or chromatographic columns with equivalent performance.

A.6.3.2 Carrier gas: Nitrogen.

A.6.3.3 Carrier gas flow rate: 5 mL/min.

A.6.3.4 Column temperature: Maintain at 35°C for 5min. Raise to 90°C at 5°C/min. Maintain for 6min.

A.6.3.5 Inlet temperature: 140°C.

A.6.3.6 Detector temperature: 300°C.

A.6.3.7 Injection volume: 1.0mL.

A.6.4 Reference headspace injection conditions

A.6.4.1 Specimen heating temperature: 60°C.

A.6.4.2 Specimen heating time: 10min.

A.6.4.3 Syringe temperature: 90°C.

A.6.4.4 Mass transfer temperature: 100°C.

A.6.5 Analysis steps

A.6.5.1 Preparation of internal standard solution

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