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Method for determination of lead in food additives

食品添加剂中铅的测定方法

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This standard applies to the limit test and quantitative test of lead in food additives.

This standard refers to the determination method of lead issued by the Joint Expert Committee on Food Additives of the Food and Agriculture Organization of the United Nations and the World Health Organization (FAO/WHO) in 1983.

1 Principle

After the sample is treated, add ammonium citrate, potassium cyanide, hydroxylamine hydrochloride to eliminate the interference of iron, copper, zinc and other ions. At pH 8.5 ~ 9.0, lead ions and dithizone form a red complex, which is extracted with chloroform and compared with the standard series for limit test or quantitative test.

2 Reagents

Unless otherwise specified, the reagents used in this standard are analytically pure reagents; the water is deionized water or lead-free water.

2.1 Nitric acid (GB 626-78).

2.2 Sulfuric acid (GB 625-77).

2.3 Ammonia water (GB 631-77) (1 + 1): If it contains lead, it must be redissolved in a full glass distiller.

2.4 Hydrochloric acid (GB 622-77).

2.5 Chloroform (GB 682-78): It shall not contain oxides.

2.6 Phenol red indicator solution: 0.1% ethanol solution.

2.7 Diammonium hydrogen citrate (HGB 3294-60): 50% solution.

Weigh 100 g of diammonium hydrogen citrate. Dissolve it in 100 ml water. Add 2 drops of phenol red indicator solution. Add ammonia water (1 + 1) to adjust the pH to 8.5 ~ 9.0 (from yellow to red, add 2 more drops). Use dithizone chloroform solution for extraction several times, 10 ~ 20 ml each time, until the chloroform layer remains green. Discard the chloroform layer. Wash twice with chloroform, 5 ml each time. Discard the chloroform layer. Add water to dilute it to 200 ml.

2.8 Hydroxylamine hydrochloride (HG 3-967-76): 20% solution.

3.3 250 ml Kjeldahl flask or 250 ml conical flask.

4 Sample treatment

4.1 "Sample treatment" of inorganic samples can be carried out according to the methods specified in the standard texts.

4.2 "Sample treatment" of organic samples, in addition to the provisions of the standard texts, is generally carried out according to the following procedures.

4.2.1 Wet digestion: Weigh 5.0 g of sample. Place it in a 250 ml Kjeldahl flask or conical flask. Add 10 ml nitric acid to soak the sample. Place for a while (or overnight). Slowly heat. Wait for the effect to ease and cool slightly. Add 5 ml of sulfuric acid along the wall of the bottle. Then slowly heat, until the solution in the bottle begins to turn brown. Continue to add nitric acid (if necessary, add some perchloric acid; during the operation, pay attention to prevent explosion), until the organic matter is completely decomposed. Continue to heat until a large amount of sulfur dioxide white smoke is generated. The solution shall be colorless or slightly yellow. After cooling, transfer the solution to a 50 ml volumetric flask. Wash the Kjeldahl flask or conical flask with a small amount of water several times. Add the washing liquid to the volumetric flask. Add water to the scale. Mix well. Each 10 ml solution is equivalent to 1.0 g of sample.

Take the same amount of nitric acid and sulfuric acid. Do a reagent blank test according to the above method.

4.2.2 Dry digestion: This method is suitable for samples that are not suitable for wet digestion.

Weigh 5.0 g of sample into a porcelain crucible. Add appropriate amount of sulfuric acid to wet the sample. Carefully carbonize it. Then add 2 ml nitric acid and 5 drops of sulfuric acid. Carefully heat until the white smoke is gone. Transfer to a high temperature furnace. Completely ash it at 550 °C. Take it out after cooling. Add 1 ml of nitric acid (1 + 1) solution. Heat to dissolve the ash. Transfer the sample solution to a 50 ml volumetric flask (filter if necessary). Wash the crucible with a small amount of water. Transfer the washing liquid to the volumetric flask. Add water to the scale. Mix well and set aside. Each 10 ml solution is equivalent to 1.0 g of sample.

Take a crucible and perform a reagent blank test according to the above method.

5 Determination

5.1 Limit test

Take an appropriate amount of sample solution and lead limit standard solution (lead content not less than 5 μg). Place them in 125 ml separatory funnels, respectively. Add

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