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Method for limit test of heavy metals in food additives

食品添加剂中重金属限量试验法

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1 Scope

This standard applies to the limit test of heavy metals in food additives.

This standard refers to the determination method of heavy metals 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.

2 Principle

Under weak acidic conditions (pH 3 ~ 4), heavy metal ions in the sample react with hydrogen sulfide to produce brown-black, which is compared with the lead standard solution treated in the same way, to carry out the limit test.

3 Reagents

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

3.1 Nitric acid (GB 626-78).

3.2 Sulfuric acid (GB 625-77).

3.3 Hydrochloric acid (GB 622-77).

3.3.1 6 mol/L hydrochloric acid: Measure 50 mL of hydrochloric acid; dilute to 100 mL with water.

3.3.2 1 mol/L hydrochloric acid: Measure 8.3 mL of hydrochloric acid; dilute to 100 mL with water.

3.4 Ammonia water (GB 631-77).

3.4.1 6 mol/L ammonia water: Measure 40 mL of ammonia water; dilute to 100 mL with water.

3.4.2 1 mol/L ammonia water: Measure 6.7 mL of ammonia water; dilute to 100 mL with water.

3.5 pH3.5 acetate buffer: Weigh 25.0 g of ammonium acetate (GB 1292-77); dissolve

it in 25 mL of water; add 45 mL of 6 mol/L hydrochloric acid; adjust the pH to 3.5 with dilute hydrochloric acid or dilute ammonia water; dilute to 100 mL with water.

3.6 Phenolphthalein indicator solution: 1% ethanol solution.

3.7 Saturated hydrogen sulfide water: Pass hydrogen sulfide gas into water without carbon dioxide until saturated (this solution is prepared before use).

3.8 Lead standard solution: Weigh 0.1598 g of high-purity lead nitrate (HG 3-1309-80); dissolve in 10 mL of 1% nitric acid; quantitatively transfer to a 100 mL volumetric flask; dilute with water to the mark. 1 mL of this solution is equivalent to 1.0 mg of lead. Dilute 100 times with water before use, so that 1.0 mL is equivalent to 10 μg lead.

3.9 1% nitric acid: Take 1 mL of nitric acid (GB 626-78); dilute to 100 mL with water.

4 Instruments

The glassware used needs to be soaked in 10 ~ 20% nitric acid for more than 24 hours, rinsed repeatedly with tap water, finally rinsed with water.

4.1 50 mL Nessler colorimetric tube.

5 Sample treatment

Generally, samples can be directly measured according to Chapter 6 "Measurement". However, if the color of tube A is darker than that of tube C, the samples shall be treated first. Organic samples must be digested by dry method; then proceed according to 6.1, 6.2, 6.4.

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

5.2 "Sample treatment" of organic samples can generally be carried out according to the following procedures, in addition to those as specified in the standard texts.

5.2.1 Wet digestion: Weigh 5.0 g of sample; place it in a 250 mL Kjeldahl flask or conical flask; add 10 ~ 15 mL of nitric acid to soak the sample; leave it for a while (or overnight); heat it slowly; cool it slightly after the effect is relieved; add 5 mL of sulfuric acid along the wall of the bottle; then heat it slowly until the solution in the bottle begins to turn brown; continue to add nitric acid (if necessary, add some perchloric acid; be careful to prevent explosion during the operation). Continue heating until the organic matter is completely decomposed, until the largest white smoke of sulfur dioxide is generated; at last the solution shall be dark brown or slightly yellow. After cooling, add 20 mL of water and boil to remove the residual nitric acid, until white smoke is produced. Do this twice and let it cool. Pipette the solution to a 50 mL volumetric flask;

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