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GB/T 5009.81-2003

Replacing GB/T 11681-1989

Method for Analysis of Hygienic Standard of Stainless Steel Food Containers and Table Wares

不锈钢食具容器卫生标准的分析方法

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Foreword

This Standard replaces GB/T 11681-1989 “Method for Analysis of Hygienic Standard of Stainless Steel Food containers and Table Wares”.

Compared with GB/T 11681-1989, the major changes of this Standard are as follows:

- According to GB/T 20001.4-2001 “Rules for Drafting Standards - Part 4: Methods for Chemical Analysis”, the structure of the previous standard is changed.

This Standard was proposed by and shall be under the jurisdiction of the Ministry of Health of the People's Republic of China.

This Standard was responsibly drafted by Shangxi Province Health and Anti-Epidemic Station, and Liaoning Province Food Hygiene Supervision and Inspection Institute.

The main drafters of this Standard: Li Wenyuan, Liu Guoxiang, Shi Chongyi, Lu Guihua, Xu Min, Li Min and Zhang Hua.

The previous standard was first-time released in 1989; and this is the first revision.

Method for Analysis of Hygienic Standard of Stainless Steel Food Containers and Table Wares

1 Scope

This Standard specifies the method for the analysis of the hygienic standard of stainless steel food containers and table wares.

This Standard applies to the determination of all hygienic indicators of all kinds of kitchen wares, table wares, food containers, as well as other containers, wares and devices in contact with foods, which are made of the material of stainless steel.

2 Normative References

The provisions in the following documents become the provisions of this Standard through the reference of this Standard. For the dated reference documents, none of their subsequent amendments (excluding corrigendum) or revisions apply to this Standard. However, all parties who reach an agreement according to this Standard are encouraged to study whether the latest editions of these documents can be applied. For the undated reference documents, their latest editions apply to this Standard.

GB/T 5009.12 Determination of Lead in Foods

GB/T 5009.62 Method for Analysis of Hygienic Standard of Ceramics for Food Containers

GB/T 5009.72 Method for Analysis of Hygienic Standard of Aluminum-Wares for Food Use

3 Preparation of Samples

3.1 Sampling Method

Take samples according to 0.1% of the product quantity; produce in small batches; take at least 6 samples each time; indicate respectively the product name, batch number, steel number and sampling date. One half of the samples are used for laboratory test and the other half are stored for two months to be used for arbitration analysis.

3.2 Visual inspection

generated during atomization absorbs certain radiation energy; the absorption amount is proportional to the content of the metallic elements; then the content of the samples can be quantified by comparing with the standard series.

4.1.2 Reagents

4.1.2.1 50 g/L ammonium dihydrogen phosphate solution: weigh 5 g of ammonium dihydrogen phosphate ($NH_4H_2PO_4$, guaranteed reagent); add water to dissolve and dilute to 100 mL.

4.1.2.2 Chromium standard solution: weigh precisely 2.828 9 g of potassium dichromate ($K_2Cr_2O_7$, reference reagent); dry to constant volume at 105 °C ~ 110 °C; add 50 mL of water to dissolve before transferring to a 1 000 mL volumetric flask; add 2 mL of nitric acid; shake and add water to dilute to the scale mark. The solution contains 1 mg of chromium per milliliter.

4.1.2.3 Lead standard solution: weigh precisely 1.000 0 g of metal lead (Pb, 99.99%); add 5 mL of $c(HNO_3)$ = 6 mol/L nitrate to dissolve; transfer to a 1 000 mL volumetric flask; add water to dilute to the scale mark. This solution contains 1 mg of lead per milliliter.

4.1.2.4 Nickel standard solution: weigh precisely 1.000 0 g of nickel (Ni, 99.99%); add 5 mL of $c(HNO_3)$ = 6 mol/L nitric acid to dissolve; transfer to a 1 000 mL volumetric flask; add water to dilute to the mark. This solution contains 1 mg of nickel per milliliter.

4.1.2.5 Standard solutions of chromium, nickel and lead: before use, dilute gradually the standard solutions of chromium, nickel and lead, respectively, to the metal standard working solutions equivalent to 1 μg per milliliter.

4.1.3 Instruments

4.1.3.1 Graphite furnace atomic absorption spectrophotometer.

4.1.3.2 Pyrolytic graphite tube and high purity argon.

4.1.3.3 Trace pipettor.

4.1.4 Analysis steps

4.1.4.1 Preparation of samples and mixed standard series

Draw 0.50 mL ~ 1.00 mL of sample soaking solution to place into a 10 mL volumetric flask; take another six 10 mL volumetric flasks; draw metal standard working solutions respectively. chromium: 0, 0.20, 0.40, 0.60, 0.80 and 1.00 mL, nickel: 0, 0.50, 1.00, 1.50, 2.00 and 2.50 mL; lead: 0, 0.30, 0.60, 0.90, 1.20 and 1.50 mL. Add 1.0 mL of 50 g/L ammonium dihydrogen phosphate solution to the samples and standard tubes; use water to dilute to the scale mark and mix evenly. The metal contents of the standard series prepared are - chromium: 0, 0.20, 0.40, 0.60, 0.80, 1.00 μg; nickel: 0, 0.50, 1.00, 1.50, 2.00

S -- area of sample in contact with soaking solution, in square centimeter (cm^2);

2 -- 2 mL of soaking solution per square centimeter, in milliliter per square centimeter (mL/cm^2).

4.2 Diphenylcarbazide colorimetry (determination of chromium)

4.2.1 Principle

Use potassium permanganate oxidate low-valent chromium to high-valent chromium (Cr^{6+}); add sodium pyrophosphate to hide the residual iron; use diphenylcarbazide to produce red complex with chromium; then quantify by comparing with the standard series.

4.2.2 Reagents

4.2.2.1 Sulfuric acid: c (H_2SO_4) = 2.5 mol/L. Take 70 mL of guaranteed reagent sulfuric acid to add to water while stirring; add water to 500 mL after placing aside for cooling.

4.2.2.2 3 g/L potassium permanganate solution: weigh 0.3 g of potassium permanganate and add water to dissolve to 100 mL.

4.2.2.3 200 g/L urea solution: weigh 20 g of urea and add water to dissolve to 100 mL.

4.2.2.4 100 g/L sodium nitrite solution: weigh 10 g of sodium nitrite and add water to dissolve to 100 mL.

4.2.2.5 Saturated sodium hydroxide solution.

4.2.2.6 50 g/L sodium pyrophosphate solution: weigh 5 g of sodium pyrophosphate ($Na_4P_2O_7 \cdot 10H_2O$) and add water to dissolve to 100 mL.

4.2.2.7 Diphenylcarbazide solution: weigh 0.5 g of diphenylcarbazide to dissolve in 50 mL of acetone; add 50 mL of water; prepare immediately prior to use; store in a brown bottle. If the color of the solution becomes dark, it shall not be used.

4.2.2.8 Chromium standard solution: the preparation method is the same as 4.1.2.2; the concentration of the working solution is 10 μg of chromium per milliliter.

4.2.3 Instruments

4.2.3.1 Spectrophotometer and 3 cm cuvettes.

4.2.3.2 25 mL stoppered cuvettes.

4.2.4 Analysis steps

4.2.4.1 Draw standard curves

Draw 0, 0.25, 0.50, 1.00, 1.50, 2.00, 2.50 and 3.00mL of chromium standard working

4.3.2.1 100 g/L ammonium citrate dibasic solution: weigh 10 g of ammonium citrate dibasic [$(NH_4)_2HC_6H_5O_7$] to dissolve in water to 100 mL.

4.3.2.2 10 g/L dimethylglyoxime-ethanol solution: weigh 1 g of dimethylglyoxime [$(CH_3)_2C_2(NOH)_2$] to dissolve in ethanol [95% (volume fraction)] to 100 mL; filter if any insoluble matter; prepare the filtrate for use.

4.3.2.3 10 g/L alkaline dimethylglyoxime solution: weigh 1 g of dimethylglyoxime to dissolve in c (NaOH) = 0.2 mol/L sodium hydroxide solution to 100 mL.

4.3.2.4 Hydrochloric acid: c (HCl) = 0.5 mol/L.

4.3.2.5 4% (volume fraction) acetic acid.

4.3.2.6 Ammonia: c (NH_4OH) = 5 mol/L.

4.3.2.7 Ammonia: c (NH_4OH) = 2 mol/L.

4.3.2.8 Ammonia: c (NH_4OH) = 0.3 mol/L.

4.3.2.9 20% (volume fraction) sodium hydroxide solution.

4.3.2.10 Nickel standard solution: the preparation method is the same as 4.1.2.4; the concentration of the working solution is equivalent to 10 μg of nickel per milliliter.

4.3.3 Instruments

4.3.3.1 Spectrophotometer and 1 cm cuvettes.

4.3.3.2 25 mL stoppered cuvettes.

4.3.3.3 125 mL and 60 mL separating funnels.

4.3.4 Analysis steps

4.3.4.1 Draw standard curves

Draw 0, 0.25, 0.50, 1.00, 2.00, 3.00, 4.00 and 5.00 mL of nickel standard working solution; add 4% acetic acid to 100 mL; transfer to 125 mL separating funnels; operate hereunder same as the operation of the samples; draw standard curves by taking the absorbance as ordinate and the standard concentration as abscissa.

4.3.4.2 Determination

Take 100 mL of sample soaking solution; add 20% sodium hydroxide solution to adjust to neutral or weak alkalinity; place aside for 2 h; filter then transfer the filtrate to a 125 mL separating funnel; add 2 mL of ammonium citrate dibasic; add several drops of 2 mol/L ammonia to adjust the pH value of the solution to 8~9. Add 2 mL of dimethylglyoxime ethanol solution; add 10 mL of chloroform; shake vigorously for 1 min; stand then separate

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