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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 5009.62-2003

**Method for analysis of hygienic standard of ceramics for food
contact**

陶瓷制食具容器卫生标准的分析方法

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Foreword

This Standard replaces GB/T 5009.62-1996 "Method for Analysis of Hygienic Standard of Ceramics for Food Containers". Compared with GB/T 5009.62-1996, main changes of this Standard are as follows: - The framework of the former standard is reformatted in accordance with GB/T 20001.4-2001 "Rules for Drafting Standards - Part 4. Methods of Chemical Analysis". 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 drafted by Guangdong Foshan Municipal Sanitation and Anti-epidemic Station. This Standard was first-time issued in 1985, and first-time revised in 1996. This is the second-time revision. Method for Analysis of Hygienic Standard of Ceramics for Food Containers

1 Scope

China's national analysis method for the hygienic standard of ceramic tableware, containers and utensils in direct contact with food. Ceramic itself is inert; the glaze is not. Glazes and the decorations over them have historically been coloured with lead and cadmium compounds, which give reds, yellows and oranges that are hard to obtain any other way and which produce a brilliant, durable finish. If the glaze is properly formulated and fired those metals are locked in the glass, and if it is not - underfired, or with an overglaze decoration applied on top rather than fired in - they leach, and acidic food accelerates it. A

single badly made mug used daily for fruit juice is a genuine source of lead exposure, and lead has no safe level in children. So the test is an extraction with dilute acetic acid, which stands in for acidic food, followed by determination of the metals released. The standard specifies the analysis for each hygienic index of such ware.

This Standard specifies the analysis methods for each hygienic index of various tableware, container and utensils which are made of ceramic and directly contact food. This Standard is applicable to the analysis for each hygienic index of various tableware, container and utensils which are made of ceramic and directly contact food.

2 Normative References

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. For dated reference, subsequent amendments (excluding corrigendum) or revisions of these publications do not apply. However, the parties who enter into agreement based on this Standard are encouraged to investigate the possibility of applying the most recent editions of the standards. For undated references, the latest edition of the normative document referred to applies.

GB/T 5009.12-2003 Determination of Lead in Foods

GB 13121 Hygienic Standard of Ceramics for Food Containers

3 Sampling Method

Sample from each batch of the products with glaze colour floration allocated. Generally, the sampling quantity of small-batch shall not be less than 6. Product name, batch number, and sampling date shall be indicated. If the sample is in small shape, the sampling quantity may be increased in accordance with inspection requirements. Half of the samples are used for assay, and the other half are preserved for two months for arbitrary analysis. Where, X - The lead content in the soak solution, in mg/L; A_s - Absorbance of lead standard solution; m - Mass of lead standard solution, in μg ; A_t - absorbance of soak solution; V - Volume of soak solution adopted, in millilitre (mL); Expression of results. report that it is less than or larger than 7 mg/L.

7.1 Atomic absorption spectrometry

7.1.1 Principle The cadmium ion in soak solution shall be led into the atomic absorption spectrophotometer and atomized; absorb 228.8 nm resonance line; the absorbed dose is proportional to the cadmium content; it shall be quantified through comparison with the standard series.

7.1.2 Reagent

7.1.2.1 Cadmium standard solution. accurately weigh out 0.1142 g of cadmium oxide; add 4

mL of glacial acetic acid; dissolve them by slowly heating; cool it down; transfer the solution into a 100mL volumetric flask; dilute with water to the scale; 1mL of this solution is equivalent to

1.00 mg of cadmium.

7.1.2.2 Standard cadmium working solution. suck

1.0 mL of standard cadmium solution; pour it into a 100 mL volumetric flask; dilute it with acetic acid (4%) to the scale. Per millilitre of this solution is equivalent to 10.0 µg of cadmium.

7.1.3 Instrument Atomic absorption spectrophotometer.

7.1.4 Analysis procedure

7.1.4.1 Preparation of standard curve. suck 0mL, 0.50mL, 1.00mL, 3.00mL, 5.00mL,

10.00 mL of standard cadmium working solution respectively; pour them into 100ml volumetric flasks respectively; dilute with acetic acid (4%) to the scale; mix uniformly; per millilitre of these solutions are equivalent to 0µg, 0.05µg, 0.10µg, 0.30µg, 0.50µg, 0.70µg and 1.00 µg of cadmium respectively. Calibrate the atomic

7.2.2.2 Sodium hydroxide-potassium cyanide solution (No. 1). Weigh out 400 g of sodium hydroxide and 10 g of potassium cyanide; dissolve them in water; dilute to 1000ml.

7.2.2.3 Sodium hydroxide-potassium cyanide solution (No. 2). Weigh out 400 g of sodium hydroxide and

0.5 g of potassium cyanide; dissolve them in water, dilute to 1000ml.

7.2.2.4 Dithizone-trichloromethane solution (0.1g/L).

7.2.2.5 Dithizone-trichloromethane solution (

0.02 g/L).

7.2.2.6 Sodium potassium tartrate solution (250 g/L).

7.2.2.7 Hydroxylamine hydrochloride solution (200 g/L).

7.2.2.8 Tartaric acid solution (20 g/L). store it in refrigerator.

7.2.2.9 Standard cadmium working solution. the same as 7.1.2.2.

7.2.3 Instrument Visible spectrophotometer.

7.2.4 Analysis procedure Take two piece of 125 mL separating funnels; add

0.5 mL of standard cadmium working solution (equivalent to 5 µg of cadmium) and

9.5 mL of acetic acid (4%) into one separating funnel; add 10mL of sample soak solution into the other one. Add 1 mL of sodium potassium tartrate solution, 5 mL of sodium hydroxide-potassium cyanide solution (No. 1), and 1 mL of hydroxylamine hydrochloride solution into the two separating funnels respectively. Shake the solution uniformly after adding all reagents above. Add 15mL of dithizone-trichloromethane solution (

0.1 g/L); shake for 2min (this step shall be rapidly conducted). Take another set of separating funnels; add 25mL of tartaric acid solution respectively; pour the dithizone-trichloromethane solution in the former set of separating funnels into the latter set of separating funnels; wash the former set of separating funnels with 10mL of trichloromethane; pour the trichloromethane cleaning solution into the latter set of separating funnels. Shake the latter set of separating funnels for 2min; discard the dithizone-trichloromethane solution; add 6 mL of trichloromethane respectively; shake and discard the trichloromethane layer. Add

1.0 mL of hydroxylamine hydrochloride solution;

15.0 mL of dithizone-trichloromethane solution (

0.02 g/L) and 5 mL of sodium hydroxide-potassium cyanide solution (No.2) into the aqueous solution in separating funnels respectively; shake immediately for 2min. Wipe and dry the internal wall of the lower pipe of separating funnels; insert a small amount of absorbent cotton in order to filter the globule. Pour the dithizone-trichloromethane solution into the 25mL colorimeter tubes-with-plugs, and conduct colour comparison. The degree of red of sample tube must not be larger than that of the standard tube. Otherwise, calibrate the zero point of 3 cm cuvette with trichloromethane; measure the absorbance at point where the wave length is 518 nm; and conduct quantitation.

7.2.5 Result Calculation It shall be the same as 6.2.3.