GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 39.060
Y 88

GB/T 28021-2011

Adornment - Determination of Baneful Elements - Method of Spectrometry

饰品　有害元素的测定　光谱法

Issued on: October 31, 2011　　**Implemented on: February 01, 2012**

Issued by: **General Administration of Quality Supervision, Inspection and Quarantine of People's Republic of China;**

Standardization Administration of the People's Republic of China.

Table of Contents

Foreword

This Standard was drafted according to the rules specified in GB/T 1.1-2009.

This Standard was proposed by China National Light Industry Council.

This Standard shall be under the jurisdiction of National Standardization Technical Committee of Jewelry (SAC/TC 256).

Drafting organization of this Standard: National Jewelry Quality Supervision and Inspection Center.

Chief drafting staffs of this Standard: Li Suqing, Li Yuou and Li Wujun.

Adornment - Determination of Baneful Elements – Method of Spectrometry

1 Scope

This Standard specifies the method of spectrometry to determine content and dissolution quantity of baneful elements in adornment.

This Standard is applicable to determination of content and dissolution quantity of baneful elements in adornment.

2 Normative References

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any modification sheet) applies.

GB 28480 "Adornment - Provision for Limit of Baneful Elements"

3 Principles

As for adornments made of metal material, they are directly processed with conventional acid digestion method; as for adornments made of other materials, they are processed with enclosed high-temperature pressure tank - acid digestion method. The arsenic, lead, mercury and cadmium in the sample thus become soluble salts, and are dissolved in the acid digestion solution. After volumetrically scaling the digestion solution, determine them with flame atomic absorption spectrometric method and inductively-coupled plasma spectrometry.

IMMERSE the sample of dissolution quantities of stibium, arsenic, barium, cadmium, chromium, lead, mercury and selenium that are required to be tested INTO the hydrochloric acid solution of certain concentration for 2h. Simulate the conditions under which the sample, after being swallowed, contacts with the stomach-acid persistently for some time. Use flame atomic absorption spectrometry method and inductively-coupled plasma spectrometry to determine the ion concentration of stibium, arsenic, barium, cadmium, chromium, lead, mercury and selenium dissolved in the hydrochloric acid solution.

4 Reagents

Unless otherwise specified, only identified analytical reagent AND distilled water, or deionized water, or water with equivalent purity are to be used in the analysis.

4.1 Nitric acid, with a mass fraction of 65% ~ 68%, and density ρ of 1.40 g/mL.

4.2 Nitric acid, (1+1).

4.3 Hydrochloric acid, with a mass fraction of 36%~38%, and density ρ of 1.19g/mL.

4.4 Hydrochloric acid, (0.07 ± 0.005) mol/L, (1+169).

4.5 Aqua regia, nitric acid (4.1) + hydrochloric acid (4.4) = 1+3.

4.6 Aqua regia, aqua regia (4.5) + water = 1+19.

4.7 Hydrogen peroxide, with a mass fraction of 30%.

4.8 Hydrofluoric acid.

4.9 Stibium, arsenic, barium, cadmium, chromium, lead, mercury and selenium standard stock solution, 100 mg/L~1000 mg/L.

5 Apparatuses

Normal laboratory apparatuses and the following apparatuses.

5.1 Analytical spectrometer: The apparatus, after being optimized, may meet the following requirements: The precision is 1%, and the detection limit of tested element is superior to 0.2 mg/L. The flame atomic absorption spectrometer (AAS) or inductively coupled plasma emission spectrometer (ICP) is recommended.

5.2 Oven: 0°C~ 200°C, with a temperature control accuracy of ± 2°C.

5.3 Pressure tank: Polytetrafluoroethylene pressure tank or stainless steel pressure tank (polytetrafluoroethylene liner).

5.4 Analytical scale: With a sensibility weight of 0.1 mg and the accuracy grade of Grade 2.

5.5 Crushing equipment: Electric saw, grinder and metal cutter etc.

5.6 Capped container: Excluding baneful elements, the total volume is 1.6 times ~ 5.0 times of the extraction agent for hydrochloric acid solution.

6 Determination of Baneful Elements' Content

6.1 Sample preparation

6.1.1 Metal material

The sample is processed into the debris or strips of which the diameter is not greater than 1mm and the length is not greater than 5mm.

6.1.2 Other materials

The sample is grinded into powder, of which the diameter is not greater than 1mm, by crushing equipment such as electric saw and grinder.

6.2 Digestion of sample

6.2.1 Conventional acid digestion method

This method is applicable to samples specified in 6.1.1.

Weigh 0.3g~0.5g of samples for two portions, accurate to 0.1 mg; place them in the beaker or conical flask. Place gold, platinum and palladium samples into 10 ml of aqua regia (4.5); place other metal materials into 0.8ml of nitric acid (4.3); put them in electric hot plate or electric arc furnace and heat; cover with a watch glass; cool it after the sample is completely dissolved. Add 10ml of hydrochloric acid (4.3); place it on the electric hot plate or electric furnace; heat for 1h; then transfer into a 100ml volumetric flask; wash with water; fix the volume to the mark. Simultaneously do a reagent blank test.

ADD samples with the silver content greater than 5% INTO the hydrochloric acid; stand for 1h; filter the silver chloride; re-transfer it and fix the volume.

Note: As for non-precious metal material samples, the hydrochloric acid is not added during the test of their lead content.

6.2.2 Pressure tank digestion method

This method is applicable to samples specified in 6.1.2.

Weigh 0.3g~0.5g of samples for two portions, accurate to 0.1 mg; place them in pressure tank (5.3); add 8 ml of nitric acid (4.1) and 2mL of hydrogen peroxide (4.7); as for materials such as glass and ceramics with many silicon, re-add 3 ml of hydrofluoric acid (4.8); cover it with a watch glass; tighten up; place it in an oven (5.2); heat for 4h at 180°C ±5°C. Cool the pressure tank to room temperature; transfer the digestion solution into 100 ml volumetric flask; wash the pressure tank with water AND cover it with a watch glass for 3~4 times; merge the cleaning solution into the volumetric flask; dilute it with water to the scale. If the solution is not clear or the sediment is generated, filter it and keep the filtrate for determination. Simultaneously do a reagent blank test.

6.2.3 Other digestion methods

8 Test report

The test report shall at least include the following information:

— The identification of sample: including source, receiving date and shape of sample;

— The standards used (including issuance or publication year);

— Adopted methods;

— The content or dissolution quantity of baneful elements in sample, which is calculated according to the requirements of 6.4 and 7.3;

— If necessary, the difference to the analytical procedures specified in the standard method shall be provided;

— Records of abnormal conditions during the test;

— Test date;

— Signing and stealing of the laboratory that conducts the analysis;

— Signature of the responsible-person and operating personnel of the laboratory.

Appendix A

(Informative)

Recommended Analytical Test Line

The recommended wavelength of each element is detailed in Table A.1.

Table A.1 Recommended Wavelength of each Element in nm

Element	Wavelength	Other available wavelength
Lead	220.353	216.999
Cadmium	226.502	228.802
Chromium	283.563	284.325
Arsenic	193.696	189.042
Stibium	206.833	217.581
Selenium	196.090	203.985
Mercury	184.887	194.164
Barium	455.403	233.527

END

------ The End ------