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The assessment method of the heavy metal Sb, As, Ba, Se and Cr (VI) in furniture

家具中重金属锑、砷、钡、硒、六价铬的评定方法

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Caution: Personnel, who use this Standard, shall have practical experience in laboratory work. This Standard does not address all safety problems. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with national regulations.

1 Scope

This Standard specifies the inspection and assessment methods of the soluble heavy metal Sb, As, Ba, Se, and Cr (VI) in furniture.

This Standard applies to furniture products whose surface is coating, veneer layer, and soft covering.

This Standard does not apply to furniture products with ceramic enamel or metal glaze on the surface, and all furniture products which are melted or fused in glass, glaze, or ceramic enamel, as well as furniture products with metallic electroplated coating.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the versions with the dates indicated are applicable to this document. For the undated references, the latest version (including all the amendments) are applicable to this document.

GB/T 602-2002 Chemical Reagent - Preparations of Standard Solutions for Impurity

GB/T 6682 Water for analytical laboratory use - Specification and test methods

3 Terms and definitions

The following terms and definitions are applicable to this document.

3.1 Base material

5.1.13 All glassware, sample containers, etc., before use, shall be immersed in nitric acid solution for 24 h, and washed with water and dried.

5.2 Cr (VI) in furniture

5.2.1 General laboratory instruments and equipment.

5.2.2 Spectrophotometer.

5.2.3 Crushing equipment: pulverizer, scissors, scraper, etc.

5.2.4 Stainless steel metallic screen: The pore size is 0.5 mm (32 mesh).

5.2.5 Water bath shaker: with temperature control function. The frequency is adjustable.

5.2.6 Acidity meter: The accuracy is 0.1 pH unit.

5.2.7 Balance: The accuracy is 0.1 mg.

5.2.8 Beaker: 500 mL, etc.

5.2.9 Pipette: 1 mL, 2 mL, 5 mL, 10 mL, 25 mL, etc.

5.2.10 Volumetric flask: 25 mL, 50 mL, 100 mL, 1000 mL, etc.

5.2.11 Extractor: 150 mL, 250 mL conical flask with cover or 150 mL, 250 mL beaker with a watch glass.

5.2.12 Filter membrane (applicable to aqueous solution): The pore size is 0.45 μm.

5.2.13 Measuring cylinder: 10 mL, 50 mL, 100 mL, etc.

5.2.14 High performance liquid chromatograph: equipped with constant flow pump, constant temperature column oven, variable wavelength ultraviolet detector.

5.2.15 Chromatographic data processor or workstation.

5.2.16 Chromatographic column: Agilent Zorbax Eclipse XDB-C18 25cm × 4.6mm (inner diameter), 5 μm stainless steel column; or equivalent chromatographic column.

5.2.17 Filter: The membrane pore size is about 0.22 μm.

5.2.18 All glassware, sample containers, etc., before use, shall be immersed in nitric acid solution for 24 h, and washed with water and dried.

6.3.1 Simulated gastric acid extraction

6.3.1.1 Diphenylcarbazide.

6.3.1.2 Acetone.

6.3.1.3 Methanol.

6.3.1.4 Phosphoric acid solution 1+1 (volume ratio).

6.3.1.5 Diphenylcarbazide developer: WEIGH 1.0 g of diphenylcarbazide (SEE 6.3.1.1) to dissolve it in 100 mL of acetone (SEE 6.3.1.2), and STORE in a brown bottle. The solution is stored at 4 °C and is valid for two weeks. When the solution fades, it shall be re-prepared.

6.3.1.6 Standard stock solution of Cr (VI) (purchased or prepared according to Method 2 in GB/T 602-2002): The concentration is 100 mg/L.

6.3.1.7 Standard solution of Cr (VI): The concentration is 5 mg/L. USE a pipette to PIPETTE 5.0 mL of standard stock solution of Cr (VI) (SEE 6.3.1.6) in a 100 mL volumetric flask; and USE water to dilute to volume. This solution shall be prepared before use on the same day.

6.3.1.8 According to the provisions of 6.2.1.1~6.2.1.5, this method shall be used for the inspection of furniture for infants and children.

6.3.2 Simulated acidic sweat extraction

6.3.2.1 It shall be in accordance with the provisions of 6.3.1.3.

6.3.2.2 It shall be in accordance with the provisions of 6.3.1.4.

6.3.2.3 It shall be in accordance with the provisions of 6.3.1.6.

6.3.2.4 It shall be in accordance with the provisions of 6.3.1.7.

6.3.2.5 It shall be in accordance with the provisions of 6.2.2.1~6.2.2.5.

6.3.3 Simulated alkaline sweat extraction

6.3.3.1 Disodium hydrogen phosphate dodecahydrate ($Na_2HPO_4 \cdot 12H_2O$).

6.3.3.2 Disodium hydrogen phosphate dihydrate ($Na_2HPO_4 \cdot 2H_2O$).

6.3.3.3 Alkaline sweat: WEIGH 0.5 g of L-histidine hydrochloride monohydrate, 5.0 g of sodium chloride, 5.0 g of disodium hydrogen phosphate dodecahydrate or 2.5 g of disodium hydrogen phosphate dihydrate in a 500 mL beaker; ADD distilled water to dissolve and dilute to 1 L; USE 0.1 mol/L sodium hydroxide

0.5 g (accurate to 0.1 mg) of the prepared sample and 25 mL of 0.07 mol/L hydrochloric acid solution (SEE 6.2.1.2), at (37±2)°C, are uniformly mixed in a suitably sized container. SHAKE for 1 min, and DETECT the acidity of this mixture. If the pH value of the mixture is greater than 1.2, while shaking the mixture, ADD about 2 mol/L hydrochloric acid solution (SEE 6.2.1.3) dropwise until the pH value of the mixture is 1.2. The mixture shall be protected from light. At a temperature of (37±2)°C, USE a temperature-controlled water bath shaker at an oscillation frequency of 100 r/min ~ 200 r/min to continuously shake for 1 h. At a temperature of (37±2)°C, LET stand for 1 h. (If necessary, SEPARATE them using a centrifuge with a centrifugal capacity of 5000g. After the standing time, the filtration separation treatment shall be carried out as quickly as possible. If centrifugation separation treatment is used, the time shall not exceed 10 min.) Before the elemental analysis is carried out, when the sample solution is stored for more than 1 d, it shall add hydrochloric acid solution to stabilize, so that the concentration c(HCl) of the stored sample solution is about 1 mol/L.

7.2.1.2 Method 2: simulated acidic sweat treatment

0.5 g (accurate to 0.1 mg) of the prepared sample and 25 mL of acidic sweat (SEE 6.2.2.5), at (37±2)°C, are uniformly mixed in a suitably sized container. SHAKE for 1 min, and DETECT the acidity of this mixture. If the pH value of the mixture is greater than 5.5, while shaking the mixture, ADD about 2 mol/L hydrochloric acid solution (SEE 6.2.1.3) dropwise until the pH value of the mixture is 5.5. The mixture shall be protected from light. At a temperature of (37±2)°C, USE a temperature-controlled water bath shaker at an oscillation frequency of 100 r/min ~ 200 r/min to continuously shake for 1 h. At a temperature of (37±2)°C, LET stand for 1 h. (If necessary, SEPARATE them using a centrifuge with a centrifugal capacity of 5000g. After the standing time, the filtration separation treatment shall be carried out as quickly as possible. If centrifugation separation treatment is used, the time shall not exceed 10 min.) The sample solution shall be tested on the same day.

7.2.2 Treatment of sample for the determination of Cr (VI) in furniture

7.2.2.1 Method 1: simulated gastric acid treatment

2.0 g (accurate to 0.1 mg) of the prepared sample and 50 mL of 0.07 mol/L hydrochloric acid solution (SEE 6.2.1.2), at (37±2)°C, are uniformly mixed in a suitably sized extractor. SHAKE for 1 min, and DETECT the acidity of this mixture. If the pH value of the mixture is greater than 1.2, while shaking the mixture, ADD about 2 mol/L hydrochloric acid solution (SEE 6.2.1.3) dropwise until the pH value of the mixture is 1.2. COVER the extractor with a stopper or a watch glass. The mixture shall be protected from light. At a temperature of (37±2)°C, USE a temperature-controlled water bath shaker at an oscillation

Mobile phase: methanol (SEE 6.3.1.3)+phosphoric acid solution (SEE 6.3.1.4) =30+70 (volume ratio);

Flow velocity: 1.0 mL/min;

Column temperature: room temperature (Temperature difference shall be no more than 2 °C);

Test wavelength: 540 nm;

Injection volume: 20 μL;

The above operating conditions are typical operating parameters. The analyst can, according to the characteristics of the instrument, appropriately adjust the above operating parameters, to obtain the best separation effect.

8.2.2.2 Preparation of a series of standard working solutions

PIPETTE 0.0 mL, 0.4 mL, 1.0 mL, 2.0 mL, 3.0 mL, 4.0 mL, 5.0 mL, 6.0 mL, 7.0 mL, 8.0 mL, 10.0 mL of standard solution of Cr (VI) (SEE 6.3.1.7) to 100 mL volumetric flasks, respectively; DILUTE to volume, and SHAKE well. The concentration of Cr (VI) in this standard solution series is 0.0 mg/L, 0.02 mg/L, 0.05 mg/L, 0.10 mg/L, 0.15 mg/L, 0.20 mg/L, 0.25 mg/L, 0.30 mg/L, 0.35 mg/L, 0.40 mg/L, 0.5 mg/L, respectively. PIPETTE 20 mL of the above standard sample solution; and ADD 1.0 mL of developer (SEE 6.3.1.5), 1.0 mL of phosphoric acid solution (SEE 6.3.1.4), SHAKE well. After standing for 5 min to 10 min, it is determined by liquid chromatography.

The series of standard working solutions shall be prepared on the same day of use.

8.2.2.3 Preparation of sample solution

PIPETTE 20 mL of the extract treated according to the provisions of 7.2.2; ADD 1.0 mL of developer (SEE 6.3.1.5), 1.0 mL of phosphoric acid solution (SEE 6.3.1.4); and SHAKE well. PIPETTE another 20 mL of the extract; ADD 2.0 mL of distilled water, and SHAKE well. Blank determination is carried out by liquid chromatography.

8.2.2.4 Determination of the content of Cr (VI) in sample

After the baseline of the instrument is stabilized, under the above test conditions, TAKE an appropriate amount of the series of standard working solutions; USE a liquid chromatograph, at a wavelength of 540 nm, to determine the peak area. With the peak area as the ordinate and the corresponding Cr (VI) content as the abscissa, the calibration curve is plotted. The calibration curve shall include

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