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**Determination of Lead, Cadmium, Chromium and Mercury
Content in the Finishing Material of Wood-based Panel**

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Determination of Lead, Cadmium, Chromium and Mercury Content in the Finishing Material of Wood-based Panel

1 Scope

This document describes the method of determining the content of heavy metal elements (lead, cadmium, chromium and mercury) in the finishing material and finishing coat of wood-based panel using inductively coupled (ICP) plasma emission spectrometer, atomic absorption spectrophotometer and atomic fluorescence spectrometer, etc.

This document is applicable to the determination of the content of heavy metal elements in the finishing material and finishing coat of wood-based panel.

2 Normative References

Through the normative references in this text, the contents of the following documents constitute indispensable clauses of this document. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 21191 Atomic Fluorescence Spectrometer

JJG 015 Verification Regulation for Inductively Coupled Plasma - Atomic Emission Spectrometer

JJG 023 Verification Regulation of Atomic Absorption Spectrometer

JJG 694 Atomic Absorption Spectrophotometer

3 Method Summary

3.1 Determination of Heavy Metal Elements Content

The specimen is digested through the wet method or microwave; the heavy metal elements are transferred to the digestion solution. Adopt the ICP plasma emission spectrometry to determine the spectral intensity of lead, cadmium, chromium and mercury in the digestion solution. Alternatively, adopt the flame atomic absorption spectrometry to determine the spectral intensity of lead, cadmium and chromium in the digestion solution; adopt the hydride generator

- cold atomic absorption spectrometry or atomic fluorescence spectrometry to determine the spectral intensity of mercury in the digestion solution. In accordance with the spectral intensity, conduct the quantitative determination.

3.2 Determination of Soluble Heavy Metal Elements Content

Under the conditions of specified temperature and time, use hydrochloric acid of a certain acidity to extract the soluble heavy metal elements in the specimen. Adopt the ICP plasma emission spectrometry to determine the spectral intensity of lead, cadmium, chromium and mercury in the extracting solution. Alternatively, adopt the flame atomic absorption spectrometry to determine the spectral intensity of lead, cadmium and chromium in the extracting solution; adopt the hydride generator - cold atomic absorption spectrometry or atomic fluorescence spectrometry to determine the spectral intensity of mercury in the extracting solution. In accordance with the spectral intensity, conduct the quantitative determination.

4 Reagents and Reference Materials

4.1 Test Water

The water used shall reach the requirements of distilled water or deionized water with above Grade-2 purity specified in GB/T 6682.

4.2 Hydrofluoric Acid

With premium purity.

4.3 Hydrogen Peroxide (30%)

With premium purity.

4.4 Hydrogen Peroxide Solution (0.5%)

Hydrogen peroxide 0.5% (volume fraction).

4.5 Hydrochloric Acid

Density $\rho = 1.19$ g/mL, with premium purity.

4.6 Hydrochloric Acid Solution (1 mol/L)

Concentration $c(\text{HCl}) = 1$ mol/L.

4.7 Hydrochloric Acid Solution (0.07 mol/L)

Concentration $c(\text{HCl}) = (0.07 \pm 0.005)$ mol/L.

4.8 Hydrochloric Acid Solution (5%)

brown volumetric flask. Use the hydrochloric acid solution (5%) (4.8) to dilute to the scale and shake it well. In this mixed solution, the mass concentration of mercury is 100 µg/L. Then, use the hydrochloric acid solution (5%) (4.8) to stepwise dilute it to obtain a mixed standard working solution with the mass concentrations of 25.0 µg/L, 10.0 µg/L, 5.0 µg/L, 2.5 µg/L, 1.0 µg/L and 0.0 µg/L.

5 Instruments and Materials

5.1 ICP plasma emission spectrometer: the instrument shall be able to provide a stable and clear plasma torch; the stability of the instrument shall comply with the stipulations of JJG 015.

5.2 Atomic absorption spectrophotometer: with a continuous flow injection hydride generator, equipped with quartz tube and lead, cadmium, chromium, mercury hollow cathode lamps; the stability of the instrument shall comply with the stipulations of JJG 023 and JJG 694.

5.3 Atomic fluorescence spectrometer: equipped with a high-intensity mercury hollow cathode lamp; the stability of the instrument shall comply with the stipulations of GB/T 21191.

5.4 Microwave digestion apparatus: equipped with PTFE digestion tank. Before use, the digestion tank needs to be soaked in the nitric acid solution (4.10) for 24 h, then, washed with water and dried.

5.5 Balance: with a division value of 0.1 mg.

5.6 Constant-temperature oscillating water bath: can maintain the temperature at $(37 \pm 2) ^\circ\text{C}$; the oscillation frequency is 60 times/min.

5.7 pH tester: with an accuracy of ± 0.1 pH unit.

5.8 Metal mesh sieve: with an aperture of 0.5 mm.

5.9 Scraper: a tool with a sharp blade, suitable for scraping the surface material of the specimen.

5.10 Mortar: before use, it needs to be washed with the nitric acid solution (4.10) and dried.

5.11 Pipette: 1 mL, 2 mL, 5 mL, 10 mL and 25 mL.

5.12 Brown volumetric flask: 25 mL, 50 mL, 100 mL and 1,000 mL. Before use, it needs to be soaked in the nitric acid solution (4.10) for 24 h, then, washed with water and dried.

5.13 Triangular flask with a stopper: 50 mL and 100 mL. Before use, it needs to be soaked in the nitric acid solution (4.10) for 24 h, then, washed with water and dried.

5.14 Aqueous membrane filter: with an aperture of 0.45 µm.

6 Preparation of Specimens

6.1 Preparation of Finishing Material Specimens

Before specimen preparation, the impregnated adhesive film paper shall be suspended and placed in a blast drying oven at 170 °C for 15 min, then, cooled at room temperature for 1 h. Make the impregnated adhesive film paper, thermosetting resin-impregnated paper high-pressure decorative laminate (HPL) and polyvinyl chloride (PVC) film and other finishing materials into a fragment with an area of less than 25 mm². Take this fragment as a specimen; after mixing, divide it into two equal parts; the mass of each specimen is not less than 0.5 g.

6.2 Preparation of Finishing Coat Specimen of Wood-based Panel

6.2.1 General Requirements

Dry and pollution-free finishing coat of wood-based panel shall be selected. At a distance of at least 25 mm away from the edge, scrape the specimen from the surface.

6.2.2 Preparation of coat specimen of wood-based panel

Use a scraper to evenly scrape the surface coating of the panel and avoid scraping the fibers of the substrate. The scraped specimen is ground with a mortar and passes through a metal sieve with an aperture of 0.5 mm. After mixing, the specimen is divided into two equal parts; the mass of each specimen is not less than 0.5 g.

6.2.3 Preparation of finishing coat specimen of impregnated paper wood-based panel

Use a scraper to evenly scrape the surface film adhesive layer and avoid scraping the fibers of the substrate. The scraped specimen is ground with a mortar and passes through a metal sieve with an aperture of 0.5 mm. After mixing, the specimen is divided into two equal parts; the mass of each specimen is not less than 0.5 g.

6.2.4 Preparation of finishing coat specimen of polyvinyl chloride (PVC) film of wood-based panel

Tear or use a blade to assist in removing the PVC film on the surface of the panel. Then, use the blade to scrape off the wood fiber and colloid on the back to make a fragment with an area of less than 25 mm². Take this fragment as a specimen; after mixing, divide it into two equal parts; the mass of each specimen is not less than 0.5 g.

7 Extraction

7.1 Extraction of Total Heavy Metal Elements

7.1.1 Digestion with wet method

In accordance with the requirements of 8.1.1, determine the spectral intensity of lead, cadmium, chromium and mercury in the standard working solution (4.18.1).

In accordance with the spectral intensity of each element and the concentration parameters of the standard working solution, respectively draw the concentration - spectral intensity linear regression standard curve of lead, cadmium, chromium and mercury elements. The linear correlation coefficient R^2 of the standard curve shall be greater than 0.995.

8.1.3 Determination of solution

Adopt the method of 8.1.2 to respectively measure the spectral intensity of lead, cadmium, chromium and mercury in the blank solution and the extracting solution.

8.2 Determination of Lead, Cadmium and Chromium Content in Extracting Solution through Atomic Absorption Spectrometry

8.2.1 Determination conditions of instrument

In accordance with the operation manual of the atomic absorption spectrometer, set the analysis parameters. The typical analysis parameters are shown in Appendix C.

8.2.2 Drawing of standard curve

In accordance with the requirements of 8.2.1, determine the absorbance of lead, cadmium and chromium in the standard working solution (4.18.2).

In accordance with the absorbance of each element and the concentration parameters of the standard solution, respectively draw the concentration - absorbance linear regression standard curve of lead, cadmium and chromium elements. The linear correlation coefficient R^2 of the standard curve shall be greater than 0.995.

8.2.3 Determination of solution

Respectively introduce the blank solution and the extracting solution into the atomic absorption spectrometer. Adopt the method of 8.2.2 to respectively determine the absorbance of lead, cadmium and chromium in the blank solution and the extracting solution.

8.3 Determination of Mercury Content in Extracting Solution through Hydride Generator - Cold Atomic Absorption Spectrometry

8.3.1 Drawing of standard curve

In accordance with the operation manual of the hydride generator - cold atomic absorption spectrometer, set the analysis parameters. Continuously introduce the mercury element standard working solution into the hydride generator system. Use hydrochloric acid solution (5%) (4.8) as the carrier liquid and 20 g/L potassium borohydride mixed solution (4.15) as the reducing agent. At 253.7 nm, use the mercury hollow cathode lamp to determine the absorbance of mercury element.

In accordance with the absorbance of mercury element and the concentration parameter of the mercury element standard working solution, draw the concentration - absorbance linear regression standard curve of mercury. The linear correlation coefficient R^2 of the standard curve shall be greater than 0.995.

8.3.2 Determination of solution

Respectively introduce the blank solution and extracting solution into the hydride generator system. Adopt the method of 8.3.1 to respectively determine the absorbance of mercury element in the blank solution and the extracting solution.

8.4 Determination of Mercury Content in Extracting Solution through Atomic Fluorescence Spectrometry

8.4.1 Determination conditions of instrument

In accordance with the operation manual of the atomic fluorescence spectrometer, set the analysis parameters. The typical analysis parameters are shown in Appendix D.

8.4.2 Drawing of standard curve

In accordance with the requirements of 8.4.1, continuously introduce the mercury element standard working solution into the atomic fluorescence spectrometer. Use hydrochloric acid solution (5%) (4.8) as the carrier liquid and 20 g/L potassium borohydride mixed solution (4.15) as the reducing agent; determine the fluorescence intensity of mercury element in the standard working solution.

In accordance with the fluorescence intensity of mercury element and the concentration parameter of the mercury element standard working solution, draw the concentration - fluorescence intensity linear regression standard curve of mercury. The linear correlation coefficient R^2 of the standard curve shall be greater than 0.995.

8.4.3 Determination of solution

Adopt the method of 8.4.2 to respectively determine the fluorescence intensity of mercury element in the blank solution and the extracting solution.

9 Result Calculation

9.1 Result Expression of Content of Heavy Metal Elements

9.1.1 The content of lead, cadmium, chromium and mercury in the extracting solution and the blank solution shall be calculated in accordance with Formula (1), and accurate to 0.01 mg/L:

$$\rho_i = (K_i A_i + B_i) / 1\ 000 \quad \dots\dots\dots (1)$$

A_i ---the absorbance, or fluorescence intensity, or spectral intensity of soluble heavy metal lead, cadmium, chromium or mercury;

B_i ---the standard curve intercept of lead, cadmium, chromium or mercury, expressed in ($\mu\text{g/L}$).

9.2.2 The content of soluble heavy metal lead, cadmium, chromium and mercury in the specimen shall be calculated in accordance with Formula (4). Take the average value of the results of two parallel tests as the determined value, which shall be accurate to 0.01 mg/kg:

$$m_i = \frac{(\rho_i - \rho_{i0}) \times V \times F}{M} \dots\dots\dots (4)$$

Where,

m_i ---the content of soluble heavy metal lead, cadmium, chromium and mercury elements in the specimen, expressed in (mg/kg);

ρ_i ---the mass concentration of lead, cadmium, chromium or mercury in the extracting solution, expressed in (mg/L);

ρ_{i0} ---the mass concentration of lead, cadmium, chromium or mercury in the blank solution (0.07 mol/L or 1 mol/L hydrochloric acid solution), expressed in (mg/L);

V ---the total volume of hydrochloric acid solution added to the 50 mL conical flask, expressed in (mL);

F ---the dilution ratio of the extracting solution;

M ---the mass of the specimen taken, expressed in (g).

9.2.3 Result representation

The content of soluble elements in the finishing material shall take the corrected value of the analysis result as the final test result. The corrected value shall be calculated in accordance with Formula (5), and accurate to 0.01 mg/kg:

$$m = m_i \times (1 - F_i) \dots\dots\dots (5)$$

Where,

m ---the content of a certain soluble heavy metal element in the sample, expressed in (mg/kg);

m_i ---the content of soluble heavy metal elements lead, cadmium, chromium and mercury in the specimen, expressed in (mg/kg);

F_i ---the analysis correction factor, see Table 3.

Table 3 -- Analysis Correction Factor of Soluble Elements

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