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Determination of total lead content in material of toys and children's products

玩具及儿童用品材料中总铅含量的测定

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Determination of total lead content in material of toys and children's products

Warning -- The personnel using this document shall have practical experience in formal laboratory work. This document does not indicate all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated by relevant national laws and regulations.

1 Scope

This standard specifies the determination methods of total lead content in materials of toys and children's products.

This standard is applicable to the determination of total lead content in surface coatings, metallic and non-metallic materials of toys and children's products.

2 Normative references

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

GB/T 602 Chemical reagent -- Preparations of standard solutions for impurity

GB/T 6379.2 Measurement methods and results -- Accuracy (trueness and precision) -- Part 2: Determine the standard methods of measurement repeatability and reproducibility of the basic method

GB 6675.4 Safety of toys -- Part 4: Migration of certain elements

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

3 Terms and definitions

Terms and definitions defined in GB 6675.4 are applicable to this document.

of hydrogen peroxide (5.3), and continue heating; repeat the step 1~2 times until the digestion of the sample is complete. When the solution is left about 1 mL, remove the beaker and let it cool to room temperature. Dilute the solution with about 10 mL of water, filter the solution into a 25 mL volumetric flask, and then use 5 mL of nitric acid (5.6) in total to rinse the beaker and filter paper three times. Transfer the washing solution into the 25 mL volumetric flask. The mixed solution will be determined later.

In the process of adding hydrogen peroxide, special attention shall be paid to the possible risk of solution bumping or overflow.

7.2.1.2 Microwave digestion method

Weigh about 0.2 g of the sample, and the weight shall be accurate to 1 mg; place it in a microwave digestion vessel, respectively add 5 mL of nitric acid (5.2) and 2 mL of hydrogen peroxide (5.3), and seal the digestion vessel. Carry out the digestion according to the following temperature program: raise the temperature to (180 ± 5) °C in about 10 minutes, and keep the temperature for 30 minutes; then, let the temperature go down. After the digestion tank is cooled to room temperature, open the digestion tank, and transfer the digestion solution to a 25 mL volumetric flask; wash the inner tank and inner lid of the microwave digestion vessel three times with a small amount of nitric acid (5.6), and incorporate the washing solution into the volumetric flask. If the digestion solution is not clear or has precipitation, the solution shall be filtered, and the residual solid substances shall be washed 3 times with 5 mL of nitric acid (5.6) in total; then, transfer the washing solution into the 25 mL volumetric flask. The mixed solution will be determined later.

7.2.2 Metallic materials

Note: This standard provides two digestion methods for metallic materials, which can be selected by the laboratory technicians according to the conditions.

7.2.2.1 Electric hot plate heating digestion method

Weigh about 0.2 g of the sample, and the weight shall be accurate to 1 mg; put it into a 50 mL beaker, add 6 mL of hydrochloric acid (5.4) and 2 mL of nitric acid (5.2), and heat it on an electric hot plate to keep the solution slightly boiling; digest it for about 15 minutes; if the dissolution of the sample is not complete, remove the beaker and cool it down, add 0.5 mL~2 mL of nitric acid (5.2) and a few drops of hydrochloric acid (5.4), and then continue heating; repeat the step 1~2 times to complete the digestion of the sample; when the solution is left about 1mL, remove the beaker and let it cool to room temperature. Dilute with about 10 mL of water, filter the solution into a 25 mL volumetric flask, and then rinse the beaker and filter paper three times with 5 mL of nitric acid (5.6) in total. Transfer the washing solution into the 25 mL volumetric flask. The mixed solution will be determined later.

7.2.2.2 Microwave digestion method

Weigh about 0.2 g of the sample, and the weight shall be accurate to 1 mg; place it in a microwave digestion vessel of polytetrafluoroethylene, add 6 mL of nitric acid (5.2) and 2 mL of hydrochloric acid (5.4), and seal the digestion vessel after the reaction subsides at room temperature. Carry out the digestion according to the following temperature program: raise the temperature to (180 ± 5) °C in about 6 minutes, and keep the temperature for 5 minutes before cooling down. After the vessel is cooled to room temperature, open the vessel, transfer the solution to a 25 mL volumetric flask; wash the inner tank and inner lid of the microwave digestion vessel three times with a small amount of nitric acid (5.6), and incorporate the washing solution into the volumetric flask. If the digestion solution is not clear or has precipitation, the solution shall be filtered, and the residual solid substances shall be washed three times with 5 mL of nitric acid (5.6) in total; then, transfer the washing solution into the 25 mL volumetric flask. The mixed solution will be determined later.

7.2.3 Ceramics, glass, crystal, and other non-metallic materials containing silicon

Weigh about 0.2 g of the sample, and the weight shall be accurate to 1 mg; place it in a microwave digestion vessel of polytetrafluoroethylene, add 6 mL of nitric acid (5.2) and 1 mL of hydrofluoric acid (5.5), and seal the vessel after the reaction subsides; place the vessel in a microwave digestion apparatus, and carry out the digestion according to the following temperature program: raise the temperature to (185±5) °C in about 6 minutes, and maintain the temperature for 10 minutes before cooling down. After the vessel is cooled to room temperature, open the vessel, add 8 mL of boric acid solution (5.9) to the digestion tank, and then transfer the solution to a 25 mL plastic volumetric flask; wash the inner tank and the inner lid of the microwave digestion vessel three times with a small amount of nitric acid (5.6), and incorporate the washing solution into the plastic volumetric flask. If the digestion solution is not clear or has precipitation, the solution shall be filtered, and the remaining solid substances shall be washed 3 times with 5 mL of nitric acid (5.6) in total; transfer the washing solution to the 25 mL plastic volumetric flask. The mixed solution will be determined later.

Wear acid and alkali-resistant gloves and gas masks during operation to prevent inhalation of hydrofluoric acid or skin contact.

7.2.4 Plastics, polymers, and other silicon-free non-metallic materials

Weigh about 0.2 g of the sample, and the weight shall be accurate to 1 mg; put it into a microwave digestion vessel of polytetrafluoroethylene, add 7 mL of nitric acid (5.2), and seal the vessel after the reaction subsides; place the vessel in a microwave digestion apparatus, and carry out the digestion according to the following temperature program: raise the temperature to (205±5) °C in about 20min, and maintain the temperature for 10min before cooling down. After the vessel is cooled to room temperature, open the vessel, and transfer the solution to a 25 mL volumetric flask; wash the inner tank and

inner lid of the microwave digestion vessel three times with a small amount of nitric acid (5.6), and incorporate the washing solution into the volumetric flask. If the digestion solution is not clear or has precipitation, the solution shall be filtered, and the residual solid substances shall be washed three times with 5 mL of nitric acid (5.6) in total. Transfer the washing solution into the 25 mL volumetric flask. The mixed solution will be determined later.

7.2.5 Blank test

Carry out a blank test, and the blank test shall be carried out in parallel; adopt the same analysis steps, and use the same amount of all reagents; however, no sample is added.

7.3 Determination

7.3.1 General rule

Use a flame atomic absorption spectrometer to determine. Let the test solution be sucked into the acetylene-air flame, and determine the absorption value of the selected spectral lines emitted from a lead hollow cathode lamp. Calculate the measured value according to the standard curve.

Note: The testing laboratory technicians can also use other validated detection methods, such as graphite furnace atomic absorption spectrometry (GFAAS), inductively coupled plasma optical emission spectrometry (ICP-OES), or inductively coupled plasma mass spectrometry (ICP-MS); special attention should be paid to the matrix interference during the analysis by ICP-MS or ICP-OES.

7.3.2 Selection of instrument working conditions

See Appendix A for the working conditions of the flame atomic absorption spectrometer.

7.3.3 Standard curve

When digesting the sample by the electric hot plate heating digestion method, determine the absorbance values of the lead series standard working solutions 1 (5.11) in order of concentration from low to high. When digesting the sample by the microwave digestion method, determine the absorbance values of the lead series standard working solutions 2 (5.12) in order of concentration from low to high. Take the absorbance as the ordinate and the concentration as the abscissa to draw the standard curve, and calculate the correlation coefficient. The linear correlation coefficient shall be ≥0.995.

7.3.4 Determination of lead in sample solutions

Under the same conditions as drawing the standard curve, determine the concentration values of the blank test solution and the sample solution. If the content of lead in the sample solution exceeds the maximum concentration value of the standard curve, then before the determination, the solution shall be properly diluted with nitric acid that is

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