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Limits and determination of lead, cadmium, mercury, arsenic and hexavalent chromium in glass fiber

玻璃纤维中铅、汞、镉、砷及六价铬的限量指标与测定方法

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1 Scope

This Standard specifies limits and determination of lead (Pb), mercury (Hg), cadmium (Cd), arsenic (As) and hexavalent chromium (Cr^{6+}) in glass fiber.

This Standard is applicable to glass fiber as well as its products.

For coated glass fiber products, glass fiber in glass fiber reinforced plastics, after removing the coated or attached components, this Standard also applies.

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 amendments) applies.

GB/T 601, *Chemical Reagent - Preparations of Standard Volumetric Solutions*

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

GB/T 4842, *Argon*

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

3 Limits

See Table 1 for requirements for limits of lead (Pb), mercury (Hg), cadmium (Cd), arsenic (As) and hexavalent chromium (Cr^{6+}) in glass fiber.

Table 1 -- Limits of lead (Pb), mercury (Hg), cadmium (Cd), arsenic (As) and hexavalent chromium (Cr^{6+}) in glass fiber

Item	Lead (Pb)	Mercury (Hg)	Cadmium (Cd)	Arsenic (As)	Hexavalent chromium (Cr^{6+})

solution is 0.02mg/mL each.

4.2.2.10 Lead, mercury, cadmium, arsenic mixed working curve series solution: In a set of 100mL volumetric flasks, respectively add 0.00mL, 0.50mL, 1.25mL, 2.50mL, 5.00mL, 10.00mL of lead, mercury, cadmium, arsenic mixed standard solution. Add 5mL of nitric acid. Dilute to the marking line. Shake well. Transfer into plastic bottles. The concentrations of lead, mercury, cadmium, and arsenic in this series of solutions are 0.00μg/mL, 0.10μg/mL, 0.25μg/mL, 0.50μg/mL, 1.00μg/mL, 2.00μg/mL, respectively.

4.2.3 Instruments

4.2.3.1 Inductively coupled plasma emission spectrometer.

4.2.3.2 Microwave digestion furnace.

4.2.3.3 100mL high pressure digestion tank.

4.2.4 Test solution preparation

4.2.4.1 Weigh 0.2g of sample, to the nearest of 0.0001g. Put in the digestion tank. Add 5mL of nitric acid, 5mL of hydrofluoric acid, 1mL~2mL of hydrogen peroxide. Cover the safety valve. Put the digestion tank into the microwave digestion system. Digest the sample according to the digestion conditions recommended by the instrument manufacturer. After cooling, transfer and dilute to 100mL. Mix well. It shall be test solution A, used for determination of lead, mercury, cadmium and arsenic.

4.2.4.2 Weigh about 0.2g of sample, to the nearest of 0.0001g. Put in a platinum dish. Add 3mL of nitric acid, 2mL of perchloric acid and 10mL of hydrofluoric acid. Place it on a low-temperature electric furnace to heat and evaporate until white smoke of perchloric acid begins to emit. Cool. Then add 5mL of hydrofluoric acid. Continue heating to evaporate till it is dry. Cool. Add 5mL of nitric acid and 30mL of water. Heat until the solution is clear. Transfer to a 100mL volumetric flask. Dilute to the marking line. Shake well. It shall be test solution B, used for determination of lead, cadmium.

4.2.4.3 Weigh about 0.2g of sample, to the nearest of 0.0001g. Put in a platinum dish. Use water to moisture. Add 5mL of nitric acid, 8~10 drops of potassium permanganate solution, 2mL of sulfuric acid and 10mL of hydrofluoric acid. Heat at low temperature on an electric stove to evaporate to a thick syrup. Immediately remove it. Cool. Then add 5mL of hydrofluoric acid. Continue to heat at low temperature to evaporate to a thick syrup. Remove and cool. Add 5mL of nitric acid and 30mL of water. Heat until the solution is clear. After cooling, transfer into a 100mL volumetric flask. Dilute to the marking line. Shake well. It shall be test solution C, used for determination of arsenic.

4.3.2.1 Nitric acid, ρ=1.42g/cm^3.

4.3.2.2 Hydrochloric acid, ρ=1.19g/cm^3.

4.3.2.3 Hydrogen peroxide: 30%.

4.3.2.4 Nitric acid: 0.5+99.5.

4.3.2.5 Potassium permanganate solution: 50g/L.

4.3.2.6 Stannous chloride solution: 60g/L. Weigh 6g of stannous chloride ($SnCl_2 \cdot 2H_2O$). Dissolve in 20mL of hydrochloric acid. Use water to dilute to 100mL. Prepare when needed.

4.3.2.7 Anhydrous calcium chloride, analytically pure.

4.3.2.8 Mercury dilute standard solution: 100μg/mL. Use a pipette to draw 10.00mL of mercury standard stock solution (4.2.2.6) into a 100mL volumetric flask. Use nitric acid solution (1+9) to dilute to the mark line. Mix well. The mercury concentration of this solution is 100μg/mL.

4.3.2.9 Mercury working series solution: In a set of 100mL volumetric flasks, respectively add 0.00mL, 0.50mL, 1.25mL, 2.50mL, 5.00mL, 10.00mL of mercury dilute standard solution. Add 5mL of nitric acid. Dilute to the mark line. Mix well. Transfer in a plastic bottle. The concentration of mercury in this series of solutions is 0.00μg/mL, 0.10μg/mL, 0.25μg/mL, 0.50μg/mL, 1.00μg/mL, 2.00μg/mL.

4.3.3 Instruments

4.3.3.1 Cold atomic absorption spectrometer, equipped with hydride generator.

4.3.3.2 Dual-beam mercury measuring instrument: equipped with gas circulation pump, gas drying device, mercury vapor generating device and mercury vapor absorption bottle.

4.3.3.3 Constant temperature drying oven.

4.3.3.4 100mL high pressure digestion tank.

4.3.3.5 Microwave digestion furnace.

4.3.4 Drawing of standard curve

Pipette 5.0 mL of the mercury working series solution prepared in 4.3.2.9 each. Put in the reduction bottle of the mercury vapor generator of the mercury meter. Add 1.0mL of stannous chloride solution separately. Tighten the cork quickly. Then there are bubbles. Measure the absorption value from the highest point displayed by the instrument reading. Then open the three-way valve on the

Transfer to a 1L volumetric flask. Dilute to the mark line. This solution contains 50μg of hexavalent chromium (Cr^{6+}) per milliliter.

4.4.2.12 Hexavalent chromium standard solution: Pipette 10mL of hexavalent chromium stock solution into a 100mL volumetric flask. Dilute to the mark line. This solution contains 5μg of hexavalent chromium (Cr^{6+}) per milliliter.

4.4.2.13 Diphenylcarbazide solution (5g/L): Weigh 0.5g of diphenylcarbazide to dissolve in 50mL of acetone. Use water to set volume to 100mL. Keep in a refrigerator. Do not use if discolored.

4.4.3 Instruments

4.4.3.1 Analytical balance: to the nearest of 0.1mg.

4.4.3.2 Magnetic stirrer.

4.4.3.3 pH meter: accuracy is ±0.03pH.

4.4.3.4 Visible light spectrophotometer: wavelength is 420nm~700nm.

4.4.4 Drawing of standard curve

Respectively pipette 0.00mL, 1.00mL, 2.00mL, 3.00mL, 4.00mL and 5.00mL of hexavalent chromium standard solution into a series of 50mL volumetric flasks. Add the same volume of the stock mixed digestion solution (4.4.2.10) as the digestion sample to the series volumetric flasks. Add 5mL of EDTA solution and 2.0mL of diphenylcarbazide solution. Use 0.1mol/L sulfuric acid to set volume to 100mL. Mix well. If the solution is turbid, filter it. At 540nm of visible spectrophotometer, use 10mm absorption cell. Use blank as reference to determine the absorbance. The standard curve needs to be prepared with the same product every time. The sample and the standard curve shall be compared on the spectrophotometer within 15min of color development.

4.4.5 Test steps

Weigh 0.2g~0.5g of sample, to the nearest of 0.1mg. In a 200mL PTFE beaker, add 40mL of digestion solution (4.4.2.9) and keep stirring at room temperature for 15min. Add 3g of boric acid. Continue stirring 10min to neutralize excess fluoride ion. Cool to room temperature. Add 0.1mol/L sulfuric acid to 80mL. Continue stirring 5min. If necessary, through an acetate filter or centrifugation method, separate insoluble matter. Transfer the filtrate to a 100 mL volumetric flask. Cool to room temperature. Set volume. Pipette 25mL of test solution into a 50mL volumetric flask. Add 5mL of EDTA solution and 2.0mL of diphenylcarbazide solution. Use 0.1mol/L sulfuric acid to set volume to 100mL. At 540nm of visible spectrophotometer, use 10mm absorption cell, take 0.00mL of hexavalent chromium standard solution as blank to determine the

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