YBB

National Drugs Packing Containers (Materials) Standards

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Test for release of arsenic antimony lead and cadmium

砷、锑、铅、镉浸出量测定法

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Test for release of arsenic antimony lead and cadmium

This method is applicable to the determination of leaching amounts of arsenic, antimony, lead and cadmium in various types of medicinal glass containers and pipes.

Preparation of test product solution When the test product is a container, clean the test product; use the 4% acetic acid solution to fill the product to 90% of the full volume. For containers with smaller volumes such as ampoule, fill the acetic acid solution to the shoulders of the bottle. Use the inversed beaker [it needs to be made of borosilicate glass with an average linear thermal expansion coefficient α (20 ~ 300 °C) of approximately 3.3 x $10^{-6}K^{-1}$; the new beaker must be aged] or an inert material aluminum foil to cover the mouth. Steam and boil it at 98 °C ± 1 °C for 2 hours. Take it out after cooling. The solution is the test solution. The sampling number is as shown in Table 1.

When the test product is a glass tube, take a glass tube which has a total surface area (including the internal and external surfaces of each segment of tube as well as the cross-sections of both ends) of about 500 cm^2. The two ends of the glass tube are carefully ground and cleaned, placed in a glass container containing 1000 mL of 4% acetic acid solution (the glass container shall not contain arsenic, antimony, lead, cadmium). Steam and boil it at 98 °C ± 1 °C for 2 hours. Take it out after cooling. The solution is the test solution.

1. Determination of arsenic leaching amount

Test principle The high-valent arsenic contained in the test solution is reduced to trivalent arsenic by potassium iodide and stannous chloride, then reacts with zinc particles and acid to produce new ecological hydrogen, to generate arsine hydrogen, which is absorbed by the silver salt solution to form a red colloid. Compare it with the standard curve or with the specified limit; determine its content or control its limit.

Method 1: Standard curve determination method Precisely measure 10 mL of test solution, 10 mL of blank solution, 1 mL, 2 mL, 3 mL, 4 mL, 5 mL (if necessary, according to the sample Adjust the linear range according to the actual situation) of standard arsenic solution (each 1 ml is equivalent to 1 μg of As). Respectively put them in the arsenic test bottles. Make determination according to (the second method of the Chinese Pharmacopoeia 2015 edition the four volumes of general rules 0822). Determine the absorbance at a wavelength of 510 nm. Use concentration as the X axis and absorbance

as the Y axis, to draw a standard curve. Compare it with the standard curve to determine the concentration of the test solution.

Method 2: Limit inspection method Precisely measure 10 ml of test solution, 10 ml of blank solution, 2 ml (when measuring container) or 3.5 ml (when measuring tube) of standard arsenic solution (each 1 ml is equivalent to 1 μg of arsenic); respectively put them in the arsenic test bottle. Make determination according to (the second method of the Chinese Pharmacopoeia 2015 edition the four volumes of general rules 0822). Determine the absorbance at a wavelength of 510 nm, respectively. The absorbance of the test solution shall not be higher than that of the standard arsenic solution.

Expression of results The glass containers are expressed as arsenic (mg/L); the glass tubes are expressed as arsenic (mg/dm^2).

2. Determination method of antimony leaching amount

Test principle Malachite green ($C_{23}H_{25}N_2Cl$) forms a green complex with pentavalent antimony ions. After extraction with toluene, the organic phase is extracted for colorimetry. It is compared with the standard curve or with the specified limit, to determine its content or control its limit.

Method 1: Standard curve determination method Accurately measure 10 ml of test solution, 10 ml of blank solution, 0.5 ml, 1 ml, 1.5 ml, 2 ml, 2.5 ml (If necessary, adjust the linear range according to the actual situation of the sample) of standard antimony solution (each 1 ml is equivalent to 1 μg of antimony). Place them in a separatory funnel. Add 10 ml of hydrochloric acid solution (1→2) and 6 drops of 10% stannous chloride-hydrochloric acid solution. Shake well. Place for 1 minute. Respectively add 1 ml of 14% sodium nitrite solution (newly made for immediate use). Shake well. Add 1 ml of 50% urea solution. Shake until the bubbles escape. Respectively add 1 ml of phosphoric acid solution (1→2), 10 ml of water, 10 ml of toluene, 0.5 ml of 0.2% malachite green solution. Shake for 1 to 2 minutes. Let stand for stratification. Discard the water layer. Take the toluene layer. Use the ultraviolet-visible spectrophotometry ("Chinese Pharmacopoeia" The 2015 edition the Four General Rules (0401), to determine the absorbance at a wavelength of 634 nm. Use the concentration as the X axis and the absorbance as the Y axis, to draw a standard curve. Compare with the standard curve to determine the concentration of the test solution.

Method 2: Limit inspection method When measuring the container, accurately measure 3 ml of the test solution, 3 ml of the blank solution, 2 ml of the standard antimony solution (1 ml is equivalent to 1 μg of antimony). Respectively place them in the separatory funnel. Add 10 ml of hydrochloric acid solution (1→2) and 6 drops of 10% stannous chloride hydrochloric acid solution. Shake well. Let stand for 1 minute. Respectively add 1 ml of 14% sodium nitrite solution (newly made for immediate use). Shake well. Respectively add 1 ml of 50% urea solution and shake until the bubbles escape. Respectively add 1 ml of phosphoric acid solution (1→2), 10 ml of water, 10 ml of toluene, 0.5 ml of 0.2% malachite green solution. Shake for 1 to 2 minutes. Let stand. Discard the water layer. Take the toluene layer. Use the ultraviolet-visible spectrophotometry ("Chinese Pharmacopoeia" The 2015 edition the Four General Rules (0401), to determine the absorbance at a wavelength of 634 nm. The absorbance of the test product solution shall be not higher than the absorbance of the standard antimony solution.

When measuring the tube, accurately measure 0.6 ml of the test solution, 0.6 ml of the blank solution, 2 ml of the standard antimony solution (1 ml is equivalent to 1 μg of Sb).

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