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**Determination of arsenic, mercury, antimony and selenium
in dye products -- Atomic fluorescence spectrometry**

染料产品中砷、汞、锑、硒的测定

原子荧光光谱法

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Determination of arsenic, mercury, antimony and selenium in dye products -- Atomic fluorescence spectrometry

1 Scope

This document specifies the method for the determination of arsenic, mercury, antimony, and selenium in dye products by atomic fluorescence spectrometer.

This document applies to the determination of arsenic, mercury, antimony, and selenium in dye 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 6682-2008 Water for analytical laboratory use—Specification and test methods

GB/T 8170-2008 Rules of rounding off for numerical values & expression and judgment of limiting values

GB/T 21191 Atomic fluorescence spectrometer

JJG 196-2006 Working Glass Container

3 Terms and Definitions

There are no terms and definitions that need to be defined in this document.

4 Principles

The test solution after wet digestion or microwave digestion enters the atomic fluorescence spectrometer, by the action of potassium borohydride (or sodium borohydride) reduction under acidic conditions, then, arsine, hydrogen antimonide, hydrogen selenide gas, and mercury atoms are generated; The hydride forms ground-state atoms in an argon-hydrogen flame, and the ground-state atoms and mercury atoms

are excited by the light emitted by the element (arsenic, mercury, antimony, selenium) lamp to generate atomic fluorescence. Within a certain range, the strength of the atomic fluorescence is proportional to the content of the element to be measured in the sample.

5 Test methods

5.1 General provisions

Unless otherwise specified, the reagents used in this document are the analytical grade. The water used in the laboratory shall meet the requirements of secondary water in GB/T 6682-2008.

The judgment of the test results shall be carried out according to the rounding value comparison method in 4.3 of GB/T 8170-2008.

5.2 Reagents and materials

5.2.1 Nitric acid: guaranteed reagent.

5.2.2 Perchloric acid: guaranteed reagent.

5.2.3 Mixed acid: Mix well the perchloric acid and the nitric acid in a volume ratio of 1:3.

5.2.4 Hydrochloric acid: guaranteed reagent.

5.2.5 1+9 hydrochloric acid solution: Mix well the hydrochloric acid and water in a volume ratio of 1:9.

5.2.6 1+9 nitric acid solution: Mix well the nitric acid and water in a volume ratio of 1:9.

5.2.7 Hydrogen peroxide: guaranteed reagent.

5.2.8 Ascorbic acid.

5.2.9 Thiourea.

5.2.10 Ascorbic acid-thiourea mixed solution: dissolve 10 g of ascorbic acid and 10 g of thiourea in 100 mL of water. This solution shall be prepared just before use.

5.2.11 Potassium borohydride.

5.2.12 Sodium hydroxide.

5.2.13 High-purity argon: purity $\geq 99.999\%$.

5.2.14 Commonly used laboratory utensils: They shall meet the requirements of Class A glass measuring instruments and glass utensils in JJG 196-2006.

5.2.15 Standard solutions of arsenic, mercury, antimony, and selenium: Purchase from certified reference material suppliers; the concentration shall be 1.000 mg/mL. Seal and refrigerate it.

5.3 Instruments and equipment

5.3.1 Atomic fluorescence spectrometer: The performance index of the instrument shall comply with the provisions of GB/T 21191.

5.3.2 Element lamps (arsenic, mercury, antimony, selenium).

5.3.3 Microwave digestion apparatus.

5.3.4 Heater: the temperature is adjustable.

5.3.5 Balance: the accuracy shall be 0.0001 g.

5.4 Test procedure

5.4.1 Preparation of standard solution for each element

5.4.1.1 Standard stock solution

5.4.1.1.1 Preparation of As standard stock solution: Use a pipette to draw 1.00 mL of As standard solution into a 1000 mL volumetric flask, and dilute to volume with 0.2 mol/L hydrochloric acid solution to make up a standard stock solution of 1.00 mg/L.

5.4.1.1.2 Preparation of Hg standard stock solution: Use a pipette to draw 1.00 mL of Hg standard solution into a 1000 mL volumetric flask, and dilute to volume with 0.2 mol/L nitric acid solution to make up a standard stock solution of 1.00 mg/L.

5.4.1.1.3 Preparation of Sb standard stock solution: Use a pipette to draw 1.00 mL of Sb standard solution into a 1000 mL volumetric flask, and dilute to volume with 0.2 mol/L hydrochloric acid solution to make up a standard stock solution of 1.00 mg/L.

5.4.1.1.4 Preparation of Se standard stock solution: Use a pipette to draw 1.00 mL of Se standard solution into a 1000 mL volumetric flask, and dilute to volume with 0.2 mol/L hydrochloric acid solution to make up a standard stock solution of 1.00 mg/L.

5.4.1.2 Standard working solution

5.4.1.2.1 Concentration range and preparation of the standard working solution

The concentration range of each standard working solution suitable for the

conical flask, then add 10 mL hydrochloric acid (5.2.4) and 10 mL nitrate acid (5.2.1); put the conical flask on the heater (5.3.4) and slowly heat it until the yellow smoke almost completely disappears; add 10 mL mixed acid (5.2.3) after the solution cools slightly, then heat it on the heater with high fire until the sample is completely digested and the solution becomes colorless or slightly yellow and transparent (for this purpose, it is sometimes necessary to replenish the mixed acid according to the circumstances); add 10 mL water after the solution cools slightly, heat it to boil and emit white smoke, and keep heating for a few minutes to drive off the residual mixed acid; then, cool the solution to room temperature, transfer it to a 50 mL volumetric flask (if turbidity, precipitation or mechanical impurities appear in the solution, it shall be filtered to remove impurities), dilute to the scale with water.

At the same time, prepare a blank solution with the same method, and use it as a blank reference solution when measuring.

5.4.2.2 Preparation of sample measurement solution

5.4.2.2.1 Arsenic determination solution

Pipette 20 mL digestion solution prepared according to 5.4.2.1 into a 50 mL volumetric flask, add 5 mL ascorbic acid-thiourea mixed solution (5.2.10), and then dilute to volume with 1+9 hydrochloric acid solution (5.2.5); place it at room temperature for more than 2 h or overnight.

5.4.2.2.2 Mercury determination solution

Pipette 25 mL digestion solution prepared according to 5.4.2.1 into a 50 mL volumetric flask, and then dilute to volume with 1+9 nitric acid solution (5.2.6).

5.4.2.2.3 Antimony determination solution

Pipette 20 mL digestion solution prepared according to 5.4.2.1 into a 50 mL volumetric flask, add 5 mL ascorbic acid-thiourea mixed solution (5.2.10), and then dilute to volume with 1+9 hydrochloric acid solution (5.2.5).

5.4.2.2.4 Selenium determination solution

Pipette 20 mL digestion solution prepared according to 5.4.2.1 into a 50 mL volumetric flask, add 5 mL ascorbic acid-thiourea mixed solution (5.2.10), and then dilute to volume with 1+9 hydrochloric acid solution (5.2.5).

5.4.3 Determination

According to the operating procedures of the atomic fluorescence spectrometer, and according to the parameters provided by the instrument manufacturer, adjust the instrument to the best working state; according to the measurement method provided

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