GB

NATIONAL STANDARD OF THE

PEOPLE’S REPUBLIC OF CHINA

ICS 87.040

G 50

GB/T 30647-2014

Determination of harmful elements total content of coatings

涂料中有害元素总含量的测定

Issued on: December 31, 2014 **Implemented on: July 01, 2015**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;

Standardization Administration of PRC.

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1 Scope

This standard specifies the test method for the total content of harmful elements in coatings.

This standard applies to the determination of the total content of various harmful elements in coatings, including but not limited to the exemplified elements, such as lead, cadmium, mercury, chromium, arsenic, etc.

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) are applicable to this standard.

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

GB/T 6682-2008 Water for analytical laboratory use - Specification and test methods

3 Principle

After the coating film is dried, suitable methods such as dry ashing method, wet acid digestion method or microwave digestion method are used to remove all organic substances; after dissolving, filtering, volume-adjusting treatment, a suitable analytical instrument [such as atomic absorption spectrometer (AAS), inductively coupled plasma emission spectrometer (ICP-OES), inductively coupled plasma mass spectrometer (ICP-MS), etc.] are used to determine the content of test elements in the test solution after treatment.

4 Reagents and materials

Only the reagents identified as analytically pure are used in the analysis test. The water used meets the requirements of grade 3 water in GB/T 6682-2008.

4.1 Nitric acid: about 65% (mass fraction). Nitric acid that has turned yellow washed and dried by water.

6 Test procedures

6.1 Preparation of coating film

Stir the sample to be tested evenly. Prepare a mixed sample according to the product's stated ratio (no need to add a diluent). After mixing well, prepare a coating film of appropriate thickness on a glass plate or Teflon plate (see 5.12). Under the drying conditions specified in the product manual, after the coating film is completely dry [if the self-drying paint is dried, the temperature must not exceed (60 ± 2) °C], remove the coating film. Use a crushing equipment (see 5.2) to crush it, to make the size of the coating film be less than 5 mm.

Note 1: For the coating film (such as elastic and plastic coating film) that cannot be crushed by the crusher, use a clean scissors to cut the coating film so that the size of the coating film is less than 5 mm.

Note 2: For powder samples, the test solution is directly prepared.

Note 3: For samples that cannot form a film, the test solution is prepared directly after drying.

6.2 Preparation of test solution

6.2.1 General

6.2.1.1 This standard provides the following three test solution’s preparation methods. The laboratory may choose one of them according to the conditions.

6.2.1.2 When the following methods are used to digest samples, it may also determine appropriate digestion conditions according to the actual conditions of the samples, to ensure that all organic compounds in the sample are removed and all elements to be tested are dissolved. If there are residues in the processed samples, the residues shall be detected by appropriate measuring methods [such as X-ray fluorescence spectrometer (XRF)], to ensure that no tested element exists. Otherwise, it shall change the digestion conditions to completely dissolve the test elements.

6.2.1.3 When determining the volatile and harmful elements (such as mercury, arsenic, etc.), it shall not use the dry ashing method or the wet acid digestion method; it shall select the microwave digestion method.

6.2.1.4 It shall prepare two sets of test solutions at the same time for testing.

6.2.2 Dry ashing method

filter membrane (see 5.13) to filter the digestion solution and transfer it to a 50 mL volumetric flask (see 5.9). Use water to rinse the inner wall and inner lid of the digestion tank. Collect the washing solution in the same volumetric flask. Meanwhile use water to rinse the filter membrane. Collect the resulting solution in the same volumetric flask. Use water to dilute it to the mark. Meanwhile carry out the reagent blank test.

6.3 Testing

6.3.1 General

For the test solution prepared in accordance with 6.2, use appropriate analytical instrument (see 5.1) to determine the content of the tested elements.

When using any kind of analytical instrument for measurement, the analyst shall operate it in accordance with the instrument manual or operation manual; indicate the analytical instrument used in the test report.

6.3.2 Preparation of standard working solution

Select a suitable volumetric flask (see 5.9) and a pipette (see 5.11). Use nitric acid solution (see 4.3) to dilute the standard stock solution of the tested element (see 4.5) in stages, to prepare a series of standard working solutions for instrument testing (the concentration range of the standard working solution can be determined according to the instrument used and the condition of the test sample, so that its concentration is equivalent to the concentration in the sample solution).

6.3.3 Establishment of calibration curve

Adjust the instrument and lead the standard working solution prepared in accordance with 6.3.2 into the test instrument in turn. Various types of instruments will establish a calibration curve based on the relationship between their characteristic response values and their corresponding concentrations. The calibration curve shall include at least one blank sample and five standard working solutions. The correlation coefficient of the linear regression curve shall be ≥ 0.995; otherwise, it shall make a new calibration curve again.

6.3.4 Determination of tested element content in test solution

Lead the test solution prepared according to 6.2 into the test instrument. Various types of instruments will automatically give the concentration value of the element to be tested in the test solution according to the calibration curve and the characteristic response value of the test solution. If the concentration of the element to be tested in the test solution exceeds the highest point of the calibration curve, the test solution shall be appropriately diluted by a nitric acid solution (see 4.3) before testing.

------ The End ------