SN

ENTRY & EXIT INSPECTION & QUARANTINE STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

SN/T 3365-2012

Determination of lead, iron, titanium, copper, manganese, zinc, chromium, aluminum content in quartz sand - Inductively couple plasma atomic emission spectrometric method

石英砂中铅、铁、钛、铜、锰、锌、铬、铝含量的测定

电感耦合等离子体原子发射光谱法

Issued on: December 12, 2012 **Implemented on: July 01, 2013**

Issued by: General Administration of Quality Supervision Inspection and Quarantine of PRC

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Determination of lead, iron, titanium, copper, manganese, zinc, chromium, aluminum content in quartz sand - Inductively couple plasma atomic emission spectrometric method

1 Scope

This standard specifies the inductively coupled plasma emission spectrometry method for the determination of lead, iron, titanium, copper, manganese, zinc, chromium, aluminum in quartz sand.

This method is suitable for the determination of lead, iron, titanium, copper, manganese, zinc, chromium, and aluminum in quartz sand. The detection limits of each element are: lead 0.57 μg/g; iron 0.67 μg/g, titanium 0.88 μg /g; copper 0.24 μg/g; manganese 0.25 μg/g; zinc 0.35 μg/g; chromium 0.29 μg/g; aluminum 0.56 μg/g.

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 6682 Water for analytical laboratory use - Specification and test methods

3 Method summary

The sample is dissolved with hydrofluoric acid and perchloric acid, heated and evaporated to dryness until the white smoke of perchloric acid is exhausted; the residue is dissolved with dilute nitric acid, measured by inductively coupled plasma emission spectrometer.

4 Reagents and materials

Unless otherwise specified, only use premium grade pure reagents and grade-

5.2 Crucible: Platinum crucible or platinum-gold crucible (95% Pt + 5% Au).

5.3 Electric heating plate: It can be heated to 300 °C; temperature control accuracy is ±5 °C.

5.4 Analytical balance: Sensitivity is 0.1 mg.

5.5 Agate mortar.

6 Specimen

Weigh about 20 g of specimen in a weighing bottle. Place the specimen in an oven at 105 °C for 2 hours. Take it out. Cool in a desiccator. Use an agate mortar (5.5) to grind the dried specimen, until all specimen passes through a 0.08 mm (200 mesh) square-hole sieve. Put the specimen into a sample bottle for analysis.

7 Analytical procedures

7.1 Sample digestion

Accurately weigh 1.0 g of sample (accurate to 0.1 mg) into the crucible (5.2). Add 15 mL of hydrofluoric acid (4.3) and 0.5 mL of perchloric acid (4.1). Use the temperature-controlled electrical heating plate to heat it. Set the temperature to 250 °C. When it evaporates to near dryness, remove it and cool it slightly. Add 3 mL of hydrofluoric acid (4.3). Heat and evaporate until the perchloric acid white smoke is exhausted. Remove it and cool it slightly. Use 10 mL of diluted nitric acid (4.4) to rinse the inner wall of the crucible. Place it on an electrical heating plate and heat it until it boils. Take it off. Cool it to room temperature. Transfer the solution to a 50 mL volumetric flask. Use water to dilute it. Mix it uniformly to prepare for determination.

Carry out a blank test together with the sample.

7.2 Determination

Use the 5% nitric acid solution (4.5) to dilute the mixed standard working solution (4.14) gradually to a series of standard solutions which have concentration of 0.0 μg/mL, 0.5 μg/mL, 2.0 μg/mL, 5.0 μg/mL, 10.01 μg/mL. Refer to Appendix A to set the working conditions of the instrument. After the instrument is stable, measure the spectral intensity of each element to be tested in the series of standard solutions in the order of concentration from low to high at the corresponding wavelength. Use the spectral intensity as the ordinate and element concentration as the abscissa, to draw a working curve.

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