GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 81.040.10

Q 33

GB/T 36269-2018

Determination of trace nickel for plate glass

平板玻璃中微量镍的测定方法

Issued on: June 7, 2018 **Implemented on: May 1, 2019**

Issued by: State Administration for Market Regulation of the People's Republic of China;
Standardization Administration of the People's Republic of China.

Table of Contents

Determination of trace nickel for plate glass

WARNING – Persons using this Standard should be familiar with normal laboratory practice. This Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This Standard specifies the apparatus, general rules, sample preparation, determination method, result expression, accuracy requirements and test report for the determination of trace nickel in plate glass.

This Standard applies to the chemical composition analysis of trace nickel in glass substrate, as well as the appraisal analysis of nickel sulfide in glass.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition dated applies to this document. For undated references, the latest edition of the referenced documents (including all amendments) applies to this document.

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

GB/T 8170, *Rules of rounding off for numerical values and expression and judgement of limiting values*

GB/T 9721, *Chemical reagent – General rules for the molecular absorption spectrophotometry (ultraviolet and visible)*

GB/T 12806, *Laboratory glassware – One-mark volumetric flasks*

GB/T 12808, *Laboratory glassware – One-mark pipettes*

GB/T 15337, *General rules for atomic absorption spectrometric analysis*

3 Apparatus

3.1 Balance

mL volumetric flask; cool to room temperature; use water to dilute to scale; shake up. Or use a nationally recognized nickel standard solution.

6.1.2.14 Nickel standard working solution (5 μg/mL)

Absorb 25 mL of nickel standard stock solution (6.1.2.13) to pour into a 500 mL volumetric flask; use water to dilute to scale; shake up.

6.1.3 Plotting of standard curve

Accurately absorb 0.00 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL and 5.00 mL of nickel standard working solution (6.1.2.14) to pour into a group of 100 mL volumetric flasks; add water respectively to 30 mL ~ 40 mL; add in succession 5 mL of ammonium citrate aqueous solution (6.1.2.9), 2.5 mL of iodine solution (6.1.2.10), 10 mL of dimethylglyoxime solution (6.1.2.11), 5 mL of ethylenediaminetetraacetic acid disodium salt solution (6.1.2.12). After a solution is added to each flask, shake up. Finally, use water to dilute to scale; shake up and make the standard solution series containing nickel 0.00 μg, 5.00 μg, 10.00 μg, 15.00 μg, 20.00 μg and 25.00 μg. After 5 min, on the spectrophotometer, at 530 nm of wavelength, use the blank reagent as the reference, use a 3 cm cuvette to measure the absorbance of the solutions, and plot the standard curve of absorbance corresponding to the content of nickel in the nickel standard solutions. The testing shall be finished within 30 min.

6.1.4 Analytical procedures

6.1.4.1 Accurately weigh 0.1 g ~ 0.5 g (accurate to 0.1 mg) of sample (5.2) to place into a platinum evaporating dish; use a small amount of water for wetting; add 10 mL of hydrofluoric acid (6.1.2.1) and 0.5 mL of perchloric acid (6.1.2.2); evaporate on a low-temperature electric furnace until white smoke comes out from perchloric acid. Take down to cool; add another 5 mL of hydrofluoric acid (6.1.2.1); continue to evaporate on the low-temperature electric furnace until dry. Take down to cool. Add 5 mL of hydrochloric acid solution (6.1.2.6) and an appropriate amount of water; leach on the low-temperature electric furnace until the solution clarifies; transfer to a 100 mL volumetric flask; control the volume of the solution at about 40 mL.

Add 5 mL of ammonium citrate aqueous solution (6.1.2.9); use ammonium hydroxide solution (6.1.2.7) and hydrochloric acid solution (6.1.2.6) to adjust the pH of the solution to neutral; add in succession 2.5 mL of iodine solution (6.1.2.10), 10 mL of dimethylglyoxime solution (6.1.2.11) and 5 mL of ethylenediaminetetraacetic acid disodium salt solution (6.1.2.12). After a solution is added to each flask, shake up. Finally, use water to dilute to scale; shake up. After 5 min, on the spectrophotometer, at 530 nm of wavelength, use the blank reagent as the reference and use a 3 cm cuvette to measure the absorbance of the solutions. The testing shall be finished within 30 min.

6.1.4.2 Blank test

Density about 1.68 g/mL; mass fraction 70% ~ 72%; guaranteed reagent.

6.2.2.3 Nitric acid

Density about 1.42 g/mL; mass fraction 65% ~ 68%; guaranteed reagent.

6.2.2.4 Absolute ethyl alcohol

Density about 0.789 g/mL; mass fraction 99%.

6.2.2.5 Nitric acid solution (1 + 1)

6.2.2.6 Nitric acid solution (1 + 4)

6.2.2.7 Nitric acid solution (1 + 99)

6.2.2.8 Nickel standard stock solution (containing 100 μg/mL of Ni)

Accurately weigh 0.100 0 g of high-purity metal nickel (of mass fraction above 99.9%: before weighing, use diluted nitric acid to scour off the oxides on the surface; then use water and absolute ethyl alcohol to wash completely; dry in the air) to place in a beaker; add 20 mL of nitric acid solution (6.2.2.5); after heating to dissolve, transfer to a 1,000 mL volumetric flask; cool to room temperature; use water to dilute to scale; shake up. Or use a nationally recognized nickel standard solution.

6.2.2.9 Nickel standard working solution (5 μg/mL)

Absorb 25.00 mL of nickel standard stock solution (6.2.2.8) to pour into a 500 mL volumetric flask; use water to dilute to scale; shake up.

6.2.3 Plotting of standard curve

Accurately absorb 0.00 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL and 5.00 mL of nickel standard working solution (6.2.2.9) to pour into a group of 100 mL volumetric flasks; add in succession nitric acid solution (6.2.2.7) to dilute to scale; shake up. Make the standard solution series containing nickel 0.00 μg, 5.00 μg, 10.00 μg, 15.00 μg, 20.00 μg and 25.00 μg.

As shown in Table 1, set the working parameters of the atomic absorption spectrophotometer and adjust to the optimum condition. Use the air-acetylene to measure the absorbance of each standard solution at the wavelength of 232.0 nm of the nickel hollow cathode lamp. Plot the standard curve of absorbance corresponding to the content of nickel in the nickel standard solutions.

This is an excerpt of the PDF (Some pages are marked off intentionally)

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