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Chemical Reagent – General Method for the Determination of Total Nitrogen

(ISO 6353-1:1982, Reagents for Chemical Analysis – Part 1: General Test Methods, NEQ)

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Chemical Reagent – General Method for the Determination of Total Nitrogen

1 Scope

This Standard specifies a general method for the determination of total amount of trace inorganic nitrogen compounds by Nessler's reagent colorimetric method and salicylic acid-hypochlorite colorimetric method.

This Standard is applicable to the determination of the total amount of trace inorganic nitrogen compounds (including nitrates, nitrites and ammonium salts) in the chemical reagents. The detection range of Nessler's reagent visual colorimetric method and spectrophotometry is 0.05μg/mL~0.8μg/mL (in terms of N); while the detection range of salicylic acid=hypochlorite visual colorimetric method and spectrophotometry is 0.04μg/mL ~1μg/mL (in terms of N).

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 document.

GB/T 601 Chemical Reagent - Preparations of Standard Volumetric Solutions

GB/T 602 Chemical Reagent – Preparations of Standard Solutions for Impurity

GB/T 603 Chemical Reagent – Preparations of Reagent Solution for Use in Test Method

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 9721 Chemical Reagent - General Rules for the Molecular Absorption Spectrophotometry (Ultraviolet and Visible)

3 Method Principle

3.1 Nessler's reagent colorimetric method

1 – lifting stand;

2 – Electric furnace (1000W~1500W, adjustable voltage or adjustable temperature);

3 – Kelvin nitrogen bottle (250mL);

4 – safety ball;

5 – condenser (200mm long);

6 – colorimetric tube (100mL).

Figure 1 – Distillation Device Schematic Diagram

4.4 Determination

4.4.1 Nessler's reagent colorimetric method

4.4.1.1 Visual colorimetric method

Sampling and preparing the sample solution as per the provisions of product standard; dilute to 140mL; place into the Kelvin nitrogen bottle; add 5mL of sodium hydroxide solution (320g/L), 1.0g of Devarda's alloy; stand for 1h. Heat and distill to approximately 75mL; which is received by 100mL colorimetric tube containing 5mL of sulfuric acid solution (0.5%). Add 3mL of sodium hydroxide solution (320g/L), 2mL of Nessler's reagent; dilute to 100mL; shake up. When the solution turns to yellow, compare it with the standard colorimetric solution.

The standard colorimetric solution is prepared by taking a nitrogen (N) standard solution with specified mass; dilute to 140mL; treat in the same manner at the same time together with the same volume of sample solution.

4.4.1.2 Spectrophotometry

4.4.1.2.1 Preparation and determination of standard colorimetric solutions

According to the provisions of the product standard, prepare 4 ~ 5 nitrogen (N) standard solutions with mass concentration in ratio; dilute to 140mL; place into Kelvin nitrogen bottle; add 5mL of sodium hydroxide solution (320g/L), 1.0g of Devarda's alloy; stand for 1h. Heat and distill approximately 75mL; receive by the 100mL colorimetric tube containing 5mL of sulfuric acid solution (0.5%). Add 3mL of sodium hydroxide (320g/L), 2mL of Nessler's reagent; dilute to 100mL; shake up. Meanwhile, do the blank test of the reagent. Use a spectrophotometer and a 1cm cuvette at a wavelength of 410nm to determine the absorbance with the reagent blank as the reference.

4.4.1.2.2 Drawing of standard working curve

Draw the standard working curve with the mass concentration of nitrogen standard solution as the abscissa, and the absorbance as the ordinate.

Appendix A

(Normative)

Preparation Method of Sodium Hypochlorite Stock Solution and Determination of Effective Chlorine Concentration and Free Base Mass Concentration

A.1 Preparation method

Titrate the hydrochloric acid to the potassium permanganate solid; introduce the evolved chlorine gas into the sodium hydroxide solution (2mol/L) for absorption; obtain the pale greenish sodium hypochlorite stock solution, which was stored in a plastic bottle and placed in the dark. This solution is unstable, and determine the effective chlorine concentration prior to use.

A.2 Determination of mass concentration of effective chlorine

Take 10.00mL sodium hypochlorite stock solution; place into 100mL volumetric flask; add water to dilute to the mark; shake up. Pipette 10.00mL; place into 250mL iodine volumetric flask; add 40mL of water and 2g of potassium iodide; shake up. Add 10mL of sulfuric acid solution (20%); cover with water seal; shake up; stand in the dark place for 5min; titrate with the sodium thiosulfate standard titration solution [$c(Na_2S_2O_3)$ = 0.1mol/L] till the solution turns to light yellow; add 1mL of starch indicator solution (10g/L); continue to titrate till the blue color of the solution disappears. Meanwhile do the blank test.

Calculate the effective chlorine concentration as per the Formula (A.1):

$$\rho = \frac{(V - V_0) \times c \times M}{V_1 \times \frac{10}{100}} \qquad \cdots\cdots (A.1)$$

Where:

ρ – mass concentration of effective chlorine, in g/L;

V – volume of sodium thiosulfate standard titration solution, in mL;

V_0 - volume of sodium thiosulfate standard titration solution consumed by blank test; in mL;

c – concentration of sodium thiosulfate standard titration solution; in mol/L;

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