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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 1254-2007

Working chemical - Sodium oxalate

工作基准试剂 草酸钠

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Foreword

The standard Chapter 4, 5.3.1,

5.3.2 mandatory, other provisions are recommended. This standard replaces GB 1254-1990 "work reference reagent (capacity) sodium oxalate", the main changes compared with the GB 1254-1990 as follows:

--- Standard name to "work reference reagent sodium oxalate";

--- Modify the Content Measurement (before version 4.1, this version 5.3). The standard proposed by China Petroleum and Chemical Industry Association. This standard by the National Standardization Technical Committee Chemistry Chemicals Branch (SAC/TC63/SC3) centralized. This standard is drafted by: Beijing Institute of chemical reagents. Participated in the drafting of this standard. Guangdong Guanghua Chemical Factory Co., Ltd. The main drafters of this standard. Han Baoying, strong Jing Lin, Wang Yuhua, Chenhan Zhao. This standard was first published in 1977, in 1990 the first amendment. Working Standard reagent sodium oxalate Molecular formula. Na2C2O

1 Scope

China's mandatory national standard for sodium oxalate as a working chemical. Sodium oxalate is the reducing counterpart of potassium dichromate: it is what standardises permanganate solutions, and permanganate is the workhorse oxidant of volumetric analysis, used for iron, calcium, hydrogen peroxide and chemical oxygen demand. It holds that position because it can be dried to constant weight without decomposing and its reaction with permanganate in hot acid is clean and complete - though it is famously slow to start, which is why the titration is run warm and why the first drops of permanganate seem to do nothing until the manganous ion produced begins to catalyse the reaction. The standard specifies the requirements, the impurity limits, the drying conditions and the test

methods for the working grade.

This standard specifies the operating reference reagent

--- sodium oxalate traits, specifications, testing, packaging and inspection rules and signs. This standard applies to the reference reagent titration analysis work

--- sodium oxalate test.

2 Normative references

The following documents contain provisions which, through reference in this standard and become the standard terms. For dated references, subsequent Amendments (not including errata content) or revisions do not apply to this standard, however, encourage the parties to the agreement are based on research Whether the latest versions of these documents. For undated reference documents, the latest versions apply to this standard. Preparation of

GB/T 601 chemical reagent standard titration solution

GB/T 602 Determination of impurities in chemical reagents prepared standard solution (GB/T 602-2002,

ISO 6353-1. 1982, NEQ) with

GB/T 603 chemical reagent test method Preparations of articles (GB/T 603-2002,

ISO 6353-1 with. 1982, NEQ)

GB/T 609 chemical reagent - General method for determination of total nitrogen (GB/T 609-2006, ISO 6353-1. 1982, NEQ)

GB/T 6682 analytical laboratory use specifications and test methods (GB/T 6682-1992, neq ISO 3696. 1987)

GB/T 9723-2007 Chemicals flame atomic absorption spectrometry General

GB/T 9724 General rule for determination of pH chemical reagent (GB/T 9724-2007, ISO 6353-1. 1982, NEQ)

GB/T 9728 General method for determination of chemical reagents sulfate (GB/T 9728-2007, ISO 6353-1. 1982, NEQ)

GB/T 9737 Determination of chemical reagents easily carbonized substances General (GB/T 9737-1988, eqv ISO 6353-1. 1982)

GB 10738 Job reference reagent titration Determination General Weighing

GB 15346 chemical reagent packaging and marking

3 Characters

This reagent is a white crystalline powder, soluble in water, soluble in ethanol.

4 Specification

Sodium oxalate as shown in Table 1. Table 1 specifications of sodium oxalate Name reference work Content (Na2C2O4), /% 99.95 ~ 100.05 pH value (30g/L, 25 °C) 7.5 ~

5 Test

5.1 Warning Reagents used in this test method are toxic or corrosive, some of the testing process can lead to dangerous situations, the operator should take Appropriate safety and health practices.

5.2 General Provisions Unless specified otherwise in this chapter, the use of standard titration solution, standard solution, preparations and products, according to GB/T 601, GB/T 602, Regulations preparation, test water should be consistent with GB/T 6682 specifications in three water samples according to precise weighing 0.01g, the use of GB/T 603's Solution "%" are represented by the mass fraction.

5.3 Content Measured according to GB 10738 requirements.

5.3.1 standard titration solution of potassium permanganate titration standard substance sodium oxalate Weigh 0.2g to (105 ± 2) °C dried to a constant standard substance sodium oxalate and precise to 0.00001g. Placed in a reaction flask, dissolved in 100mL sulfuric acid solution (80mL/L), titration with potassium permanganate standard solution [Ba (15KMnO4) = 0.1mol/L] titration, plus near the end point Heat to 65 °C, continue titration until the solution was pink, and maintain 30s. Weighing potassium permanganate standard titration solution should be accurate to 0.0001g. Determination

5.3.2 Content

5.3.1 Determination of the same, with the measurement sample instead of the standard material loss after drying. Sodium oxalate mass fraction of 1, the value in the "%" means, according to equation (1). (1) Where. B

--- material content standard sodium oxalate (mass fraction), the value to "%" means; 3g weighed sample was dissolved in 100mL of carbon dioxide-free water, determined according to the provisions of GB/T 9724's.

5.5 Clarity test Weigh 4g sample was dissolved in 100mL hot water. Turbidity is not greater than HG/T 3484 standard specified in the 2nd clarity.

5.6 Loss on drying Weigh 10g sample, accurate to 0.0001g, has been placed in (105 ± 2) °C constant weighing bottle, at (105 ± 2) °C in an electric oven Dried to a constant weight. Samples retained after constant for determination. Loss on drying 2, the value in the "%" means, according to equation (2). 100 (2) Where.

5.7 Chloride Weigh 1g sample was dissolved in 20mL water and 8mL nitric acid solution (25%), add 1mL silver nitrate solution (17g/L), shake well and let Set 10min. The turbidity of the solution was not greater than the standard turbidity solution. Preparation of the standard solution is to take the turbidimetric 0.01mg containing chloride (Cl) standard solution, the same treatment simultaneously with the sample.

5.8 sulfur compounds Weigh 1g sample was placed in an evaporating dish, add 2mL water, nitric acid and 2mL 2mL "30% hydrogen peroxide", covered with a watch glass, in a water bath The insulation 1h, evaporated to dryness. Plus a small amount of water dissolved, 6mL hydrochloric acid solution (20%), and evaporated to dryness. The residue was dissolved in 10mL of water (if necessary through Filter), diluted to 20mL, determined according to the provisions of GB/T 9728's. The turbidity of the solution was not greater than the standard turbidity solution. Standard preparation turbid solution is to take the ratio of 0.02mg containing sulfate (SO4) standard solution was diluted to 20mL, at the same time with the same volume of test solution The same treatment.

5.9 total nitrogen content Weigh 1g sample, add 100mL water, heat gently to dissolve, cooled, diluted to 140mL, determined according to the provisions of GB/T 609's. Solution Yellow No deeper than the standard colorimetric solution. Preparation of the standard developing solution containing 0.01mg ratio is taken of nitrogen (N) standard solution was diluted to 140mL, at the same time with the same volume of sample solution The same treatment.

5.10 K Determined according to the provisions of GB/T 9723-2007 of.

5.10.1 Instrument Conditions Source. Potassium hollow cathode lamp; Wavelength. 766.5nm; Flame. acetylene - air.

5.10.2 Determination Weigh 1g sample, add 100mL water, heat gently to dissolve, cooled. Take 20mL, a total of four copies, according to GB/T 9723-2007

7.2.2 The provisions of the determination, the results calculated according to the provisions of 7.2.3.

5.11 Iron Weigh 1g sample, add water, 30mL, micro-heat to dissolve, add 2mL sulfosalicylic acid solution (100g/L), shake, add 5mL ammonia solution Solution (10%) and shake. The yellow solution can not be deeper than the standard colorimetric solution. Preparation of the standard solution is to take the colorimetric containing 0.005mg of iron (Fe) standard solution, the same treatment simultaneously with the sample.

5.12 Heavy Metal Weigh 2g sample was placed in an evaporating dish, add 2mL water, nitric acid and 2mL 2mL "30% hydrogen peroxide", covered with a watch glass, in a water bath The insulation 1h, evaporated, add 4mL solution of nitric acid (1 + 1), then evaporated to dryness. The residue was dissolved in water, neutralized with aqueous ammonia solution (10%) adjusting pH of the solution To 4, diluted to 40mL. Take 30mL, add 0.2mL acetic acid