

ICS 71.040.40
CCS G 00;G09

SN

**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

SN/T 1037-2020

Replacing SN/T 1037-2002

Import and export lime nitrogen inspection method

进出口石灰氮检验方法

(English Translation)

Issued on: August 27, 2020

Implemented on: March 1, 2021

Issued by: General Administration of Customs

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Foreword

This standard was drafted in accordance with the regulations given in GB/T 1.1-2009.

This standard replaces SN/T 1037-2002 "Import and Export Lime Nitrogen Inspection Method".

Compared with SN/T 1037-2002, the main technical changes of this standard are as follows.

--In the determination of 4 total nitrogen content, "4.2 Method 2. Combustion Analysis Method (Method 2)" was added to define the original method it is "4.1 Method One. Distillation Titration Method (Arbitration Method)".

This standard was proposed and managed by the General Administration of Customs of the

People's Republic of China.

Drafting organizations of this standard. Yinchuan Customs of the People's Republic of China and Shanghai Customs of the People's Republic of China.

1 Scope

This standard stipulates the sample preparation and general regulations of import and export lime nitrogen, the determination of total nitrogen content, the determination of free calcium carbide, and the sieve the method of measuring the remainder.

This standard applies to the determination of lime nitrogen for import and export.

Warning. According to the provisions of Chapter 6 of GB 12268-2012, this product belongs to category 4, item 4.3, which emits flammable gas in contact with water be cautious when operating. The sample and some reagents used in this test method are toxic and corrosive, and the operator must be in a fume hood Handle with care! If it splashes on the skin, it should be rinsed immediately. In severe cases, it should be treated immediately. When using flammable products, it is strictly forbidden to use an open flame for heating.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article Pieces. For undated reference documents, the latest version (including all amendments) is applicable to this standard.

GB/T 601 Preparation of Standard Titration Solution for Chemical Reagents GB/T 602 Preparation of standard solutions for impurity determination of chemical reagents GB/T 6679 General Rules for Sampling of Solid Chemical Products GB/T 6682 Analytical laboratory water specifications and test methods GB/T 10665 Calcium carbide (calcium carbide) GK 12268-2012 List of Dangerous Goods SN/T 3097 Determination of Total Nitrogen in Fertilizers by Combustion Analysis Method

3.1 Take samples

Sampling in accordance with GB 6679, divide the sample stack into equally divided grids, and the grids will be no less than 20 grids when dividing large samples.

Less than 12 grids, not less than 4 grids for subdividing samples, reduce to 1 kg, save 250 g of samples, 250 g of analytical samples, and pack them in a dry seal in the bottle, indicate the batch number, production date, manufacturer name, sampler and date.

3.2 General provisions

Unless otherwise specified, the reagents used are of analytical grade, and the water is the secondary water specified in GB/T 6682. The test papers are all precision test papers. test the standard titration solution, impurity standard solution, reagents and products required in the test, unless other requirements are specified, shall be in accordance with GB/T 601, Prepared according to GB/T 602.

4.1.1 Method summary

Under the action of the catalyst, the nitrogen in the sample is completely oxidized, decomposed and turned into amine salt in concentrated sulfuric acid, which is absorbed by sulfuric acid standard solution.

Back titration with alkali standard solution to determine total nitrogen.

4.1.2 Reagents and materials

4.1.2.1

Sulfuric acid ($\rho=1.84$ g/mL).

4.1.2.2

Sodium hydroxide solution. 40%.

4.1.2.3

Sulfuric acid standard solution. 0.1 mol/L.

4.1.2.4

Sodium hydroxide standard solution. 0.1 mol/L, see GB/T 601 for the preparation method.

4.1.2.5

Catalyst. Weigh 100 g K_2SO_4 , 10 g $CuSO_4 \cdot 5H_2O$, 1 g selenium powder, and grind it to 80 mesh (0.175 mm) in a mortar, Contained in a dry ground bottle.

4 1.2.6 Indicator. 0.1% methyl red ethanol solution 0.5% cresol green ethanol solution (11).

4.1.3 Apparatus and equipment

4.1.3.1

Semi-automatic protein analyzer (or equivalent equipment).

4.1.3.2

Electric heating digester.

4.1.3.3

Magnetic stirrer and electromagnetic stirrer.

4.1.3.4

Acid burette.

4.1.4.1 Digestion

Weigh 0.1 g sample (accurate to 0.0001 g), put it into the cleaned and dried digestion tube without damage, and add a few grains with a diameter of 3 mm~ 4 mm glass beads, 6 g catalyst (4.1.2.5) and 10 mL sulfuric acid (4.1.2.1), heat digestion for 30 min (extendable 5 min~10 min).

Then cool to room temperature, add 50 mL of water to the digestion tube and mix well.

4.1.4.2 Distillation

Accurately add 25 mL of sulfuric acid standard solution (4.1.2.3) and 2~3 drops of indicator (4.1.2.6) into a 250 mL Erlenmeyer flask.

The bottle is placed on the receiving tube of the distillation apparatus, and the nozzle is immersed in the solution. Add 50 mL of sodium hydroxide solution (4.1.2.2) to the digestion tube, After 7 minutes of distillation, close the steam valve and remove the receiving flask.

4.1.4.3 Determination

Titrate the solution in the receiving bottle with sodium hydroxide standard solution (4.1.2.4), and drop it until the solution turns from purple-red to bright green as the end point.

4.1.4.4 Blank test

Except that no sample is added, all are carried out in accordance with the above-mentioned measurement steps (4.1.4.3).

4.1.5 Calculation and expression of results

The total nitrogen content in the sample is calculated by mass fraction and calculated according to formula (1).

4.1.6 Precision

The precision is shown in Table 1.