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SN

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

SN/T 0837.4-2006

**Determination of the amount of chlorine content in arsenic
trioxide. Photometric method of distillation-mercuric
thiocyanate**

三氧化二砷中氯离子含量的测定 蒸馏-硫氰酸汞分光光度法

(English Translation)

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Foreword

SN/T 0837 is divided into 4 parts.

- Method for determining the purity of arsenic trioxide exported;
- Determination of moisture in the export of arsenic trioxide;
- Determination of iron in the export of arsenic trioxide;
- Determination of chloride ion content in arsenic trioxide - Distillation - Mercury thiocyanate spectrophotometric method.

This part is the fourth part of SN/T 0837.

This part is proposed and managed by the National Certification and Accreditation Administration.

This section drafted by. Yunnan Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

1 Scope

This section specifies the method for determining the chloride ion content of arsenic trioxide.

This section applies to the determination of chloride ion content in arsenic trioxide. The measurement range is from 0.0010% to 0.0300%.

2 Method summary

The sample is dissolved in nitric acid and distilled in a nitric acid medium at a temperature of 230 ° C to 270 ° C. The evolved gas is absorbed in sodium hydroxide.

In the liquid. Adding thiocyanate and iron perchlorate, the formed chloride reacts with thiocyanate to free thiocyanate and iron (III) to develop color.

The absorbance of the spectrophotometer was measured at a wavelength of 460 nm.

3 Reagents

Unless otherwise stated, only reagents confirmed to be analytically pure and secondary distilled water distilled with a small amount of sodium hydroxide were used in the analysis.

3.1

sodium hydroxide, excellent grade pure.

3.2

Reduce iron powder.

3.3

Mercury thiocyanate.

3.4

Potassium chloride, excellent grade pure.

3.5

Methanol.

3.6

Perchloric acid ($\rho=1.67\text{g/mL}$).

3.7

Nitric acid ($\rho=1.42\text{g/mL}$), excellent grade.

3.8

Nitrogen, high purity.

3.9

Nitric acid (2+1).

3.10

sodium hydroxide solution (0.2mol/L).

3.11

Iron perchlorate solution. Weigh 7g of reduced iron powder into a 400mL beaker, add 200mL of nitric acid (3.9), and heat to dissolve.

Add 60 mL of perchloric acid, heat to the perchloric acid fumes and continue to heat until the solution is reddish brown and cool. Dilute to 500 mL with water and mix.

3.12

Mercury thiocyanate-methanol solution (5g/L). Weigh 0.5g of thiocyanate in a 250mL beaker, add 100mL of methanol, dissolve it.

Move all the way back into a brown bottle for storage.

3.13

Chloride standard storage solution. Weigh 0.2103g of superior pure potassium chloride (burned at 500 °C for 30min, and cooled in a desiccator to at room temperature, place in a 250mL beaker, dissolve in an appropriate amount of water, transfer to a 1000mL volumetric flask, dilute to the mark with water, and mix. This solution 1 mL contains 100 µg of chloride ion.

3.14

Chloride standard solution. Pipette 50.00mL of chloride ion standard storage solution (3.13) in a 500mL volumetric flask and dilute with water until Scale and mix. 1 mL of this solution contained 10 µg of chloride ion.

4 Instruments and devices

4.1

Spectrophotometer.

4.2

Distillation unit (see Figure 1).