

ICS 77.040.30
CCS H 17

YS

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

YS/T 34.3-2011

Replacing YS/T 34.4-1992

**Method for chemical analysis of the high-purity arsenic.
Predicating method for determinating the concentration of
sulfur**

高纯砷化学分析方法极谱法测定硫量

(English Translation)

Issued on: December 20, 2011

Implemented on: July 1, 2012

Issued by: Ministry of Industry and Information Technology

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Foreword

YS/T 34 "high-purity arsenic chemical analysis method" consists of three parts.

--- Part 1. Methods for chemical analysis of high-purity arsenic by inductively coupled plasma mass spectrometry (ICP-MS) Determination of impurities in high-purity arsenic content;

--- Part 2. Determination of the amount of high-purity arsenic and selenium chemical analysis of polarography;

--- Part 3. Determination of the amount of high-purity arsenic and sulfur chemical analysis of polarography.

This section is YS/T Part 3 34.

The partial replacement of YS/T 34.4-1992 "determination of sulfur high-purity arsenic chemical analysis of polarography." this section according to GB/T 1.1-2009. the preparation of "Standardization Guide Part 1 standard structure and writing" requirement.

The non-ferrous metal part by the National Standardization Technical Committee.

This section is responsible for drafting unit. Emei Semiconductor Material Factory.

1 Scope

YS/T of the provisions of section 34 of the method for the determination of arsenic in high purity sulfur content.

This section applies to the determination of arsenic (99.999%) sulfur content. Measurement range 0.00003% 0.00015%.

2 Method summary

Samples with hydrochloric acid - nitric acid dissolved, evaporated to dryness. Join hydrobromide, 120 °C heating volatile arsenic removal. Hydrochloric acid, hydroiodic acid, mixed sodium hypophosphite Was reducing sulfur impurities to produce hydrogen sulfide. -EDTA Absorb sodium hydroxide solution, oscillopolarographic determination.

3 Reagent standard solution

3.1

deionized water (resistivity greater than 15Mohm · cm).

3.2

HCl. 1000mL high purity hydrochloric acid ($\rho=1.19\text{g/mL}$), was added 0.4g two barium chloride, after sub-boiling distillation of use.

Nitrate 3.3. 1000mL high purity nitric acid ($\rho=1.42\text{g/mL}$), 0.4g of barium nitrate was added, after azeotropic distillation using ethylene.

3.4

hydrobromide. 500mL high purity hydrobromic acid (40%), placed in the reflux flask, 20g sodium hypophosphite ($\text{NaH}_2\text{PO}_3 \cdot \text{H}_2\text{O}$), to 2L/min ~ 3L/min of pure argon a rate of (3.10), the solution was heated, kept boiling, the reflux 4h. Back distillate further sub-boiling distillation, abandoned Head to 150mL distillate collecting 250mL fractions spare. Hydrobromide concentration should not be less than 8.4mol/L.

-EDTA 3.5 Sodium hydroxide absorption solution. Pipette 40.0mL ~ 45.0mL high purity sodium hydroxide solution (40%), 5.0mLEDTA Solution (0.5mol/L), placed in 500mL volumetric flask, dilute to the mark, mix and transferred to a polyethylene bottle save. This hydroxide solution Sodium concentration and the concentration of EDTA were 1mol/L, 0.01mol/L.

Note: Commercially available high-purity sodium hydroxide solution (40%) after calibration use.

3.6

reducing agent (I). Pipette 400mL hydroiodic acid (45%), 210mL of hydrochloric acid ($\rho=1.19\text{g/mL}$), 120g sodium hypophosphite into the reflux Bottle with 2L/min of pure argon a rate of (3.10), maintained at boiling heat (approximately 114 deg.] C), the reflux 5h. Cooling to room temperature, transferred to a quartz flask, Store in a cool place (effectively a half months). Boiling water bath heated to dissolve salt use.

3.7

reducing agent (II). Pipette 250mL hydroiodic acid (45%), 60mL acetic acid, 50g sodium hypophosphite, placed in the reflux flask, to 2L/min of pure argon through rate (3.10),

heated and maintained boiling (about 118 °C), the reflux 4h, stored in a cool place (within a half months valid).

3.8

sodium sulfate solution. Accurately weigh 4.431g sodium (105 °C after drying), placed 1000mL volumetric flask, add 50mL salt Acid ($\rho=1.19\text{g/mL}$), sodium sulfate dissolved, diluted with water to volume, and mix. This solution 1mL 1mg containing sulfur.

3.9

sulfur standard solution. accurate Pipette 1.0mL sodium sulfate solution (3.8), placed 1000mL volumetric flask, dilute to the mark with water, mix uniform. This solution 1mL containing 1 μg sulfur.

3.10

pure argon or nitrogen. > 99.9%.

4-volatile devices and instruments

4.1 Oscilloscope Polarograph

Scanning voltage range 0V ~ 2V.