

ICS 77.040.30
CCS H 17

YS

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

YS/T 37.4-2018

Methods for chemical analysis of high pure germanium dioxide -- Determination of magnesium,aluminum,cobalt,nickel,copper,zinc indium,lead,calcium,iron and arensic content by inductively coupled plasma mass spectrometric method

高纯二氧化锗化学分析方法 电感耦合等离子体质谱法测定镁、铝、钴、镍、铜、
锌、镉、铅、钙、铁和砷量

(English Translation)

Issued on: October 22, 2018

Implemented on: April 1, 2019

**Issued by: Ministry of Industry and Information Technology of the
People's Republic of China**

Table of Contents

Foreword...	3
1 Scope...	5
2 Method Summary...	6
3 Reagents...	6
4 Instruments...	8
5 Analytical Procedure...	8
6 Calculation and Expression of Analytical Results...	9
7 Precision...	10
8 Quality Assurance and Control...	12
9 Test Report...	12

1 Scope

This Part specifies the determination of magnesium, aluminum, cobalt, nickel, copper, zinc, indium, lead, calcium, iron and arsenic content in high pure germanium dioxide.

This Part applies to the determination of magnesium, aluminum, cobalt, nickel, copper, zinc, indium, lead, calcium, iron and arsenic content in high pure germanium dioxide; the determination range is shown in Table 1.

Table 1

2 Method Summary

After decomposition of the test material by heating with hydrochloric acid; the matrix, in the form of germanium tetrachloride, is evaporated and removed. The residue is dissolved in nitric acid; and the contents of magnesium, aluminum, cobalt, nickel, copper, zinc, indium, lead, calcium, iron, and arsenic in the solution are determined by inductively coupled plasma mass spectrometry (ICP-MS) using cesium standard solution as an internal standard.

3 Reagents

3.1

The water used in this Part is deionized water with a resistivity of no less than 18 Mohm·cm.

3.2

Hydrochloric acid ($\rho=1.20$ g/mL), guarantee reagent.

3.3

Nitric acid ($\rho=1.42$ g/mL), guarantee reagent.

3.4 Magnesium standard stock solution. Weigh 1.0000 g of magnesium [w(Mg) $\geq$ 99.99%]; place

it in a 300 mL beaker. Add 20 mL of water; slowly add 20 mL of hydrochloric acid (3.2); dissolve at low temperature; remove it and cool. Transfer to a 1000 mL volumetric flask. Add 100 mL of hydrochloric acid (3.2); dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 1 mg of magnesium.

3.5 Aluminum standard stock solution. Weigh 1.0000 g of aluminum [w(Al) $\geq$ 99.9%]; place it

in a 300 mL beaker. Add 20 mL of hydrochloric acid (3.2); titrate 1 mL~2 mL nitric acid (3.3); dissolve at low temperature. Transfer to a 1000 mL volumetric flask; add 80 mL of hydrochloric acid (3.2); dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 1 mg of aluminum.

3.6 Cobalt standard stock solution. Weigh 1.0000 g of cobalt [w(Co) $\geq$ 99.9%]; place it in a 300 mL beaker. Add 20 mL of hydrochloric acid (3.2); titrate 1 mL~2 mL of nitric acid (3.3); dissolve at low temperature. Transfer to a 1000 mL volumetric flask; add 80 mL of hydrochloric acid (3.2); dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 1 mg of cobalt.

3.7 Nickel standard stock solution. Weigh 1.0000 g of nickel [w(Ni) $\geq$ 99.9%]; place in a 300 mL beaker. Add 10 mL of nitric acid (3.3); dissolve at low temperature; heat to remove nitrogen oxides; remove it and cool. Transfer to a 1000 mL volumetric flask; add 100 mL of nitric acid (3.3); dilute with water; make constant volume to the mark and mix well. 1 mL of this solution contains 1 mg of nickel.

3.8 Copper standard stock solution. Weigh 1.0000 g of copper [w(Cu) $\geq$ 99.9%]; place in a 300 mL beaker. Add 10 mL of nitric acid (3.3); dissolve at low temperature; heat to remove nitrogen oxides; remove it and cool. Transfer to a 1000 mL volumetric flask; add 100 mL of nitric acid (3.3); dilute with water, make constant volume to the mark, and mix well. 1 mL of this solution contains 1 mg of copper.

3.9 Zinc standard stock solution. Weigh 1.0000 g of zinc [w(Zn) $\geq$ 99.9%]; place in a 300 mL beaker. Add 20 mL of hydrochloric acid (3.2); dissolve at low temperature; and cool. Transfer to a 1000 mL volumetric flask; add 80 mL of hydrochloric acid (3.2); dilute with water; make

constant volume to the mark, and mix well. 1 mL of this solution contains 1 mg of zinc.

3.10 Indium standard stock solution. Weigh 1.0000 g of indium [$w(\text{In}) \geq 99.9\%$]; place in a 300 mL beaker. Add 20 mL of hydrochloric acid (3.2); dissolve at low temperature; cool. Transfer to a 1000 mL volumetric flask; add 80 mL of hydrochloric acid (3.2); dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 1 mg of indium.

3.11 Lead standard stock solution. Weigh 1.0000 g of lead [$w(\text{Pb}) \geq 99.9\%$]; place it in a 300 mL beaker. Add 10 mL of nitric acid (3.3); dissolve at low temperature; heat to remove nitrogen oxides; remove it and cool. Transfer to a 1000 mL volumetric flask; add 100 mL of nitric acid (3.3); dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 1 mg of lead.

3.12 Calcium standard stock solution. Weigh 2.4970 g of calcium carbonate (guarantee reagent), baked at 105°C for 1 h and cool to room temperature; place in a 300 mL beaker. Add 20 mL of water; add hydrochloric acid (3.2) until completely dissolved. Then add another 10 mL of hydrochloric acid (3.2); boil to remove carbon dioxide; remove it and cool. Transfer to a 1000 mL volumetric flask; dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 1 mg of calcium.

3.13 Iron standard stock solution. Weigh 1.0000 g of iron [$w(\text{Fe})$ can be purchased from certified national standard materials 99.99%]; place it in a 300 mL beaker. Dissolve it with 30 mL of nitric acid (3.3); heat to remove nitrogen oxides; cool. Transfer to a 1000 mL volumetric flask; dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 1 mg of iron.

3.14 Arsenic standard stock solution. Weigh 1.3203 g of arsenic trioxide [$w(\text{As}_2\text{O}_3) \geq 99.9\%$] and dissolve it in 20 mL of sodium hydroxide solution (100 g/L). Heat gently until completely dissolved. Transfer to a 1000 mL volumetric flask; dilute with water to 200 mL; add 2 drops of phenolphthalein; neutralize with nitric acid (3.3) to neutral, adding an excess of 4 drops. Dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 1 mg of arsenic.

3.15 Mixed Standard Stock Solution A of magnesium, aluminum, cobalt, nickel, copper, zinc, indium, lead, calcium, iron, and arsenic. Transfer 10 mL of each of the following standard stock solutions of magnesium (3.4), aluminum (3.5), cobalt (3.6), nickel (3.7), copper (3.8), zinc (3.9), indium (3.10), lead (3.11), calcium (3.12), iron (3.13), and arsenic (3.14) to the same 1000 mL volumetric flask; add 5 mL of nitric acid (3.3); dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 10 µg each of magnesium, aluminum, cobalt, nickel, zinc, indium, lead, calcium, iron, and arsenic.

3.16 Mixed Standard Stock Solution B of magnesium, aluminum, cobalt, nickel, copper, zinc, indium, lead, calcium, iron, and arsenic. Transfer 10 mL of Mixed Standard Stock Solution A (3.15) to a 100 mL volumetric flask; add 5 mL of nitric acid (3.3); dilute with

water; make constant volume to the mark, and mix well. 1 mL of this solution contains 1 µg each of magnesium, aluminum, cobalt, nickel, copper, zinc, indium, lead, calcium, iron, and arsenic.

3.17 Mixed Standard Solution C of magnesium, aluminum, cobalt, nickel, copper, zinc, indium, lead, calcium, iron, and arsenic. Transfer 10 mL of Mixed Standard Stock Solution B (3.16) to a 100 mL volumetric flask; add 5 mL of nitric acid (3.3); dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 0.1 µg of each of magnesium, aluminum, cobalt, nickel, copper, zinc, indium, lead, calcium, iron, and arsenic.

3.18 Cesium Internal Standard Stock Solution A. 1 mg/mL.

3.19 Cesium Internal Standard Stock Solution B. Transfer 1 mL of Cesium Internal Standard Stock Solution A (3.18) to a 100 mL volumetric flask; add 5 mL of nitric acid (3.3); dilute with water; make constant volume to the mark, and mix well. 1 mL of this solution contains 10 µg of cesium.

3.20 Cesium Internal Standard Solution C. Transfer 10 mL of Cesium Internal Standard Stock

Solution B (3.19) to a 100 mL volumetric flask; add 5 mL of nitric acid (3.3); dilute with water;

make constant volume to the mark, and mix well. 1 mL of this solution contains 1 µg of cesium.

3.21 Ammonia (volume fraction $\geq 99.99\%$).

3.22 Nationally certified reference materials can be purchased.

4 Instruments

Inductively coupled plasma mass spectrometry (ICP-MS), instrument specifications are detailed in Appendix A.

NOTE: Iron, calcium, and arsenic are determined in dynamic reaction cell mode.

5.1 Test material

Weigh 0.5g of the specimen, accurate to 0.0001g.

5.2 Parallel test

Perform two parallel tests.

.....