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

GB/T 36590-2018

**Method for the chemical analysis of high purity silver -
Determination of trace impurity elements - Glow discharge
mass spectrometry**

高纯银化学分析方法 痕量杂质元素的测定 辉光放电质谱法

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Table of Contents

- 1 Scope
- 2 Principle of the method
- 3 Reagents and materials
- 4 Instruments

Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009. This standard was proposed by the China Nonferrous Metals Industry Association. This standard is under the jurisdiction of the National Nonferrous Metals Standardization Technical Committee (SAC/TC243). This standard is drafted by: National Standard (Beijing) Inspection and Certification Co., Ltd. Participated in the drafting of this standard. Anhui Huajing Microelectronics Materials Technology Co., Ltd., Jinchuan Group Co., Ltd., Emeishan City, half High Purity Materials Co., Ltd. The main drafters of this standard. Li Na, Zhang Jinxi, Zhang Ling, Liu Ying, Liu Hong, Li Aizhen, Yang Fuguang, Zhang Xiao, Cheng Ping, Qiu Ping, Sun Ping, and Zhan Ke. High purity silver chemical analysis method Determination of trace impurity elements Glow discharge mass spectrometry

1 Scope

China's national method for determining trace impurities in high purity silver by glow discharge mass spectrometry. High purity silver - five nines and above - is used where its unmatched electrical and thermal conductivity matters and where an impurity would ruin the application: in electronic contacts, in brazing alloys for vacuum work, in sputtering targets, and as the reference material against which other silver is assayed. At those purities the analytical problem inverts. There is so little of everything else that dissolving the sample and measuring the solution introduces more contamination than it detects. Glow discharge mass spectrometry avoids that entirely by working on the solid: the sample is made the cathode of a low pressure discharge, argon ions sputter atoms from its surface, those atoms are ionised in the plasma and counted by the mass spectrometer. Nothing is dissolved, nothing is added, and the detection limits reach parts per billion for most elements at once.

This standard specifies the method for determination of trace impurity elements in high purity silver. This standard applies to the determination of trace impurity elements in high purity silver. The measurement range of each element is 0.001 µg/g to 5.0 µg/g.

2 Principle of the method

The sample is used as a cathode for glow discharge, and the surface atoms are sputtered and separated from the sample into the glow discharge plasma, and then ionized. It was introduced into a mass spectrometer for measurement. Integrate the corresponding peaks with the preset number of scan points and integration time at each element. The resulting area is the peak intensity. In the absence of a standard sample, the computer automatically counts according to the "typical relative sensitivity factor" in the instrument software.

Calculate the mass fraction of each element; when there is a standard sample, it needs to pass the same analysis conditions, ion source structure and measurement as the sample to be tested. Under the test conditions, the standard sample is independently determined to obtain the relative sensitivity factor, and the relative sensitivity factor is used to calculate the quality of each element. fraction.

3 Reagents and materials

3.1 Anhydrous ethanol.

3.2 Nitric acid (1 1).

3.3 Argon ($\phi \geq 99.999\%$).

3.4 Mass calibration reference sample. The Glow Discharge Mass Spectrometer should be used with the instrument's own quality calibration sample if conditions permit. Accurate quality correction.

3.5 Unless otherwise stated, the reagents used in the tests were of excellent grade; the water used was primary water with a resistivity of $\geq 18 \text{ M}\Omega \cdot \text{cm}^{-1}$.

4 Instruments

4.1 Glow Discharge Mass Spectrometer The resolution in medium resolution mode can reach 3000~4000, and the resolution in high resolution mode can reach 9000~10000. The isotope and resolution are shown in Table 1. The peak intensity of the isotope ^{107}Ag is required to be not less than 5×10^9 cps when measured, and the peak shape is consistent. Resolution requirements.