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**NATIONAL STANDARD OF THE
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**Method for chemical analysis of lead concentrates -
Determination of antimony content - $\text{Ce}(\text{SO}_4)_2$ titration method**

铅精矿化学分析方法 锑含量的测定 硫酸铈滴定法

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Foreword

This guiding technical document was drafted in accordance with the rules given in GB/T 1.1-2009. This guiding technical document was proposed by China Nonferrous Industry Association. This guiding technical document is under the jurisdiction of the National Nonferrous Metals Standardization Technical Committee (SAC/TC243). Drafting units of this guiding technical document. Lianyungang Customs of the People's Republic of China, Zhuzhou Smelting Group Co., Ltd., Shenzhen Zhong Jinlingnan Nonferrous Metal Co., Ltd., Yunnan Chihong Zinc and Germanium Co., Ltd., National Recycled Nonferrous Metal Rubber and Plastic Material Quality Supervision and Inspection Center (Anhui), Beikuang Testing Technology Co., Ltd., Kunming Metallurgical Research Institute, Henan Yuguang Gold and Lead Co., Ltd., Shandong Hengbang Smelting Co., Ltd. Co., Ltd., National Standard (Beijing) Inspection and Certification Co., Ltd., Xi'an Hantang Analysis and Testing Co., Ltd., Guangdong Leading Thin Material Co., Ltd., South China University of Technology. The main drafters of this guiding technical document. Zhao Xiurong, Wang Heng, Jiang Yu, Qin Lijun, Qiao Zhu, Zheng Lixia, Shi Shilong, Cheng Lin, Ding Yicong, Chen Diangeng, Xiang Delei, Zhou Yi, Liu Yingbo, Huang Ping, Yao Mula, Qian Junfei, Sun

Weijia, Liu Chunfeng, Yang Wei, Wu Rongxian, Zhou Junling, Zhang Yanfeng, Luan Haiguang, Zhang Lijiu, Dai Fengying, Lu Ni, Xiang Qinghua, Zhu Zhanfang, Wang Andi, Wang Xuefei. This technical guidance document is for reference only. For suggestions and opinions on this guiding technical document, report to the standardization administrative director of the State Council Department reflects. Chemical analysis method of lead concentrate Determination of antimony content Cerium Sulfate Titration

1 Scope

GB/Z 39124-2020 is the Chinese national standard on method for chemical analysis of lead concentrates - determination of antimony content - $\text{Ce}(\text{SO}_4)_2$ titration method, in the field of metallurgy. The /Z suffix marks it as a guiding technical document: it does not prescribe requirements that can be certified against, but sets out the technique, the method or the state of the art that the standards bodies recommend following. It was issued on 11 October 2020 by the State Administration for Market Regulation; Standardization Administration of China. As a guiding technical document it carries no separate date of entry into force: it applies from publication. Classification: ICS 77.120.60, CCS D42. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This guiding technical document specifies the determination method of antimony content in lead concentrate. This guiding technical document is applicable to the determination of antimony content in lead concentrates. Measuring range. 0.80%~12.00%.

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. For undated references, the latest version (including all amendments) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods

GB/T 14262 Sampling and sample preparation method of bulk flotation lead concentrate

3 Method summary

The sample was dissolved with sulfuric acid and nitric acid, and carbonized with filter paper as a reducing agent to reduce Sb(V) to Sb(III) . In hydrochloric acid medium, use phosphoric acid Masking high-valent iron, using methyl orange and methylene blue as indicators, at 80°C ~ 90°C , Titrate with cerium sulfate standard titration solution until the solution changes to bright blue (yellow-green when iron is high) as the end point.

4 Reagents or materials

Unless otherwise specified, only use reagents confirmed to be analytically pure and third-grade water that meets the requirements of GB/T 6682 in the analysis.

4.1 Metal antimony ($w_{\text{Sb}} \geq 99.99\%$).

4.2 Potassium sulfate.

4.3 Filter paper (quantitative).

4.4 Sulfuric acid ($\rho = 1.84 \text{ g/mL}$).

4.5 Phosphoric acid ($\rho = 1.70 \text{ g/mL}$).

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

4.7 Ethanol (95%).

4.8 Hydrochloric acid (1:1).

4.9 Sulfuric acid (892).

4.10 Cerium sulfate standard titration solution $\{c[\text{Ce}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}] \sim 0.02 \text{ mol/L}\}$.

a) Weigh 8.08g of cerium sulfate tetrahydrate, place it in a 250mL beaker, add 100mL of sulfuric acid (4.9) and heat it until it is completely dissolved. Remove and cool, transfer to a 1000mL volumetric flask, dilute to the mark with sulfuric acid (4.9), shake well.

b) Weigh 0.040g~0.050g (accurate to 0.00001g) of metal antimony (4.1) into a 250mL conical flask, and add 5 pieces at the same time~ 8 glass beads, 15mL sulfuric acid (4.4), heat to complete dissolution, continue to heat until sulfuric acid fumes are emitted for 5min~10min