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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 25934.1-2010

Methods for the chemical analysis of high-purity gold - Part 1: Determination of impurity elements by ethyl acetate extraction separation and inductively coupled plasma atomic emission spectrometry

高纯金化学分析方法 第1部分：乙酸乙酯萃取分离ICP-AES法 测定杂质元素的含量

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Standardization Administration of the PRC

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Foreword

GB/T 25934 "high pure gold chemical analysis method" is divided into three parts.

--- Part 1. ethyl acetate extraction separation-ICP-AES Determination of impurity element contents; Part

--- Article 2. ICP-MS- standard addition calibration - Determination of impurity elements in the standard method;

--- Part 3. Determination of impurity elements ether extract separated -ICP-AES method. This is Part 1. This part of the National Gold Standardization Technical Committee (SAC/TC379) and focal points. This section is responsible for drafting the Changchun Gold Research Institute. This section Changchun Gold Research Institute, Shenyang Mint, Beijing Nonferrous Metal Research Institute, Beijing General Research Institute of Mining and Metallurgy, the Great Wall of fine gold and silver Refinery, Jiangxi Copper Company Limited, Jiangsu Skyray Instrument Co., Ltd. drafted. The main drafters of this section. Feifei Chen, Huang Rui, Chen Yonghong, Zhang Yu, Wang Yu, Long Shujie, Liu Hong, Li Ai Chang, Li Wanchun, to force, Chen Jie, Bo, Liang subsets, Kay, Li He. Methods for chemical analysis of high purity gold Part 1. ethyl acetate extraction separation -ICP-AES method Determination of impurity elements

1 Scope

China's national method for determining the impurity elements in high-purity gold by ethyl acetate extraction separation followed by inductively coupled plasma atomic emission spectrometry. It is Part 1 of GB/T 25934 and specifies the principle, the reagents, the apparatus, the procedure and the precision. Analysing high-purity gold is a problem of one element against everything else. The gold is more than 99.999 per cent of the sample and the elements of interest are present at parts per million or less, so the difficulty is not the detection - a plasma spectrometer measures these elements easily at those levels - but the fact that the gold matrix overwhelms the measurement. It suppresses the plasma, it deposits on the cones and the torch, and its own emission lines are numerous enough to interfere with almost anything. The practical answer is to take the gold out before

measuring, and that is what this part does. Gold dissolves in aqua regia as chloroauric acid, which is efficiently extracted into ethyl acetate while the base-metal impurities remain in the aqueous phase - so a single separation removes almost all of the matrix and leaves the elements of interest concentrated in a solution that the spectrometer can handle without interference. What the standard has to control is the completeness of that separation in both directions: the gold that is not extracted still interferes, and any impurity element that partitions into the organic phase with it is lost. So the acidity, the phase ratio, the number of extractions and the verification of recovery for each element are prescribed, along with the calibration, the blank - which at these levels is often the limiting factor - and the precision data. Issued on 23 December 2010 and in force since 1 September 2011.

GB/T 25934 in the provisions of this part of the impurity elements in high purity gold determination. This section applies to the determination of 99.999% pure gold in high impurity element, measuring element and measuring an amount ranging from Table 1. Table

1 Content range /% Content range /% Content range /% Content range /% Ag 0.00002 ~ 0.00100 Al 0.00002 ~ 0.00100 As 0.00002 ~ 0.00098 Bi 0.00002 ~ 0.00100 Cd 0.00002 ~ 0.00100 Cr 0.00002 ~ 0.00099 Cu 0.00002 ~ 0.00100 Fe 0.00010 ~ 0.00100 Ir 0.00002 ~ 0.00100 Mg 0.00010 ~ 0.00100 Mn 0.00002 ~ 0.00100 Ni 0.00002 ~ 0.00099 Pb 0.00002 ~ 0.00100 Pd 0.00002 ~ 0.00100 Pt 0.00002 ~ 0.00099 Rh 0.00002 ~ 0.00100 Sb 0.00002 ~ 0.00100 Se 0.00002 ~ 0.00100 Te 0.00002 ~ 0.00100 Ti 0.00002 ~ 0.00099 Zn 0.00010 ~ 0.00100

2 Principle of the method

Sample with a mixed acid dissolved in hydrochloric acid 1mol/L, the separation of gold extracted with ethyl acetate, the aqueous phase was concentrated into a certain acidity The liquid to be tested, inductively coupled plasma atomic emission spectrometer spectral intensity of each element.

3 Reagents

Unless otherwise indicated, used in the analysis confirmed only for the gifted class pure reagents and double distilled water or equivalent purity (resistivity >=18.2MOmega/cm) of water.

3.1 hydrochloric acid (rho1.19g/mL), pure class distinctions.

3.2 nitrate (rho1.42g/mL), pure class distinctions.

3.3 sulfate (rho1.84g/mL), pure class distinctions.

3.4 hydrofluoric acid (rho1.15g/mL), pure class distinctions.

3.5 hydrochloride (11).

3.6 nitric acid (11).

3.7 hydrochloride (19).

3.8 hydrochloride (111). Mixed acid 3.9. 1 volume of nitric acid (3.2), 3 volumes of hydrochloric acid (3.1) and 3 volumes of water and mix well.

3.10 ethyl acetate. with hydrochloric acid (3.8) and washed 2 to 3 times reserve.

3.11 Standard stock solution.

3.11.1 silver standard stock solution. Weigh 0.1000g metallic silver (mass fraction >=99.99%) in 100mL beaker, add 10mL Nitric acid solution (3.6), dissolved by heating low temperature, volatilization of oxides of nitrogen, cooled to room temperature, transferred to 100mL volumetric flask, was added 25mL of hydrochloric acid (3.1), dilute to the mark and mix. 1mL solution containing 1mg silver.