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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 25934.3-2010

Methods for the chemical analysis of high-purity gold - Part 3: Determination of impurity elements by diethyl ether extraction separation and inductively coupled plasma atomic emission spectrometry

高纯金化学分析方法 第3部分：乙醚萃取分离ICP-AES法 测定杂质元素的含量

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Standardization Administration of the PRC

Table of Contents

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 3. This part of the National Gold Standardization Technical Committee (SAC/TC379) and focal points. This section is Henan Zhongyuan gold smelter limited liability company responsible for drafting. This section is Henan Zhongyuan Gold Smelter Co., Ltd., Great Wall gold and silver refinery, Changchun Gold Research Institute, Shenyang Mint, Beijing Mining Research Institute, Jiangsu Skyray Instrument Co., Ltd. drafted. The main drafters of this section. Liu Chengxiang, Zhang Bo, Zhang Yuming, Chen Jie, Huang Rui, Chen Feifei, Chen Yonghong, Laimao Ming, Wang Yu, Lihua Chang, Li Wanchun, in force, Zheng Jianming. Methods for chemical analysis of high purity gold Part 3. ether 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 diethyl ether extraction separation followed by inductively coupled plasma atomic emission spectrometry. It is Part 3 of GB/T 25934 and specifies the principle, the reagents, the apparatus, the procedure and the precision. It solves the same problem as Part 1 with a different solvent, and the two exist side by side because the choice of extractant is not neutral. Diethyl ether is the classical extractant for chloroauric acid and the oldest way of separating gold from a chloride solution: the extraction is fast and close to quantitative in a single contact, and the phase separation is clean. For some impurity elements the partitioning is more favourable than with the ester of Part 1, so the recoveries are better and the determination is more reliable at the lowest levels. Against that, ether is a difficult reagent to work with. It is extremely volatile and flammable, its vapour is heavier than air

and travels along a bench, and on standing it forms explosive peroxides - so its storage, its age and its handling are laboratory safety matters that the standard has to address rather than assume, and the volatility itself affects the analysis, since evaporation changes the phase ratio during the separation. Which of the two parts a laboratory uses therefore depends on the elements being determined and on what its facilities allow. The rest of the method follows the same logic as Part 1: the acidity and the phase ratio that make the extraction complete, the verification that no analyte follows the gold into the organic phase, the calibration and the blank control, and the precision data by which a result is judged. Issued on 23 December 2010 and in force since 1 September 2011.

This section GB/T 25934 provides for high purity gold in silver, copper, iron, lead, antimony, bismuth, palladium, magnesium, tin, chromium, nickel, manganese, aluminum, platinum, rhodium, iridium, Zinc, titanium, cadmium, silicon and arsenic content determination. This section applies to high gold in silver, copper, iron, lead, antimony, bismuth, palladium, magnesium, tin, chromium, nickel, manganese, aluminum, platinum, rhodium, iridium, zinc, titanium, cadmium, arsenic and silicon Determination of the amount. Determination of the range in Table 1. Table

1 Element mass fraction /% elemental mass fraction /% elemental mass fraction /% elemental mass fraction /% Ag 0.00002 ~ 0.00047 Ni 0.00002 ~ 0.00047 Pt 0.00002 ~ 0.00048 Zn 0.00002 ~ 0.00044 Cu 0.00002 ~ 0.00047 Pd 0.00002 ~ 0.00049 Sn 0.00003 ~ 0.00032 Cd 0.00002 ~ 0.00048 Pb 0.00005 ~ 0.00048 Al 0.00005 ~ 0.00045 Ti 0.00002 ~ 0.00049 Ir 0.00006 ~ 0.00052 Fe 0.00002 ~ 0.00048 Mn 0.00002 ~ 0.00047 Cr 0.00002 ~ 0.00048 Si 0.00003 ~ 0.00027 Sb 0.00004 ~ 0.00043 Mg 0.00002 ~ 0.00046 Rh 0.00002 ~ 0.00050 As 0.00005 ~ 0.00046 Bi 0.00002 ~ 0.00043

2 Method summary

Sample with a mixed acid decomposition, in 1mol/L HCl medium, after separation of gold extracted with ether, the aqueous phase was concentrated HCl medium prepared test solution Liquid, using inductively coupled plasma atomic emission spectrometer silver, copper, iron, lead, antimony, bismuth, palladium, magnesium, tin, chromium, nickel, manganese, aluminum, platinum, rhodium, Iridium, zinc, titanium, cadmium, arsenic, and the amount of silicon.

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 hydrochloride (rho1.19g/mL). Nitrate 3.2 (rho1.42g/mL).

3.3 sulfate (rho1.84g/mL).

3.4 hydrofluoric acid (rho1.15g/mL).

3.5 hydrochloride (11).

3.6 nitric acid (11).

3.7 nitric acid (12).

3.8 hydrochloride (19).

3.9 hydrochloride (111).

3.10 hydrochloric acid (129). Mixed acid 3.11. 1 volume of nitric acid (3.2), 3 volumes of hydrochloric acid (3.1) and one volume of water and mix well.

3.12 ether. with hydrochloric acid solution (3.9) and washed 2 to 3 times reserve.