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Methods for chemical analysis of precious metal alloys electroplating wastewater - Part 3: Determination of sulfate content - Barium sulphate gravimetric method

贵金属合金电镀废水化学分析方法 第3部分:硫酸盐含量的测定 硫酸钡重量法

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0 Warning and structure of the series

A warning on the first page of the body states that the persons using this document need practical experience of regular laboratory work, that the document does not point out all the possible safety problems, and that it is the responsibility of the user to take suitable safety and health measures and to ensure compliance with the conditions laid down by the relevant national laws and regulations.

This document is Part 3 of GB/T 43753, Methods for chemical analysis of precious metal alloys electroplating wastewater. The parts already published are Part 1, determination of the gold, silver, platinum, palladium and iridium contents by inductively coupled plasma atomic emission spectrometry; Part 2, determination of the zinc, manganese, chromium, cadmium, lead, iron, aluminium, nickel, copper and beryllium contents by inductively coupled plasma atomic emission spectrometry; Part 3, determination of the sulfate content by the barium sulphate gravimetric method; and Part 4, determination of the chloride ion

content by the silver chloride turbidimetric method.

The Introduction records that enterprises electroplating and electroforming precious metal alloys produce large quantities of wastewater during production, such as the mixed water of plated part rinse water, spent bath solutions, equipment cooling water, flushing water and floor water, containing large amounts of sulfate that affects the wastewater treatment process and the environment, and that accurate, rapid, advanced and efficient analytical methods are urgently needed to guide the treatment schemes and the operation of the water treatment processes. It states that this part reduces gold and similar elements with hydroxylammonium chloride, forms a barium sulphate precipitate from the sulfate ion and barium chloride, separates the precipitate from iron, copper and other ions, filters, washes and ignites it to constant mass, and calculates the mass concentration of the sulfate ion in the wastewater.

1 Scope

This document describes a method for determining the sulfate content of the wastewater from precious metal alloy electroplating and electroforming by the barium sulphate gravimetric method.

It applies to the determination of the sulfate content of the wastewater from precious metal alloy electroplating and electroforming, over a determination range of 0.01 g/L to 18.00 g/L of sulfate ion mass concentration.

2 Normative references

The contents of the following document constitute indispensable provisions of this document through normative reference in the text. For dated references, only the edition corresponding to that date applies; for undated references, the latest edition, including all amendments, applies.

The document listed is GB/T 6682, Water for analytical laboratory use - Specification and test methods.

3 Terms and definitions

This document has no terms and definitions that need to be defined.

4 Principle

Gold and similar elements in the test material are reduced with hydroxylammonium chloride, the sulfate ion and barium chloride form a barium sulphate precipitate, the precipitate is separated from iron, copper and other ions, and it is then filtered, washed and ignited to constant mass; the sulfate content is expressed as the sulfate ion content and the

mass concentration of the sulfate ion is calculated.

5 Reagents and materials

Unless otherwise stated, only reagents recognised as analytical grade are used in the analysis.

The list comprises water, of grade two according to GB/T 6682; hydroxylammonium chloride; anhydrous ethanol, of density 0.79 g/mL; aqueous ammonia, of density 0.90 g/mL; hydrochloric acid, of density 1.18 g/mL; aqueous ammonia 1+1; hydrochloric acid 1+1; hydrochloric acid 1+99; silver nitrate solution at 1 %, prepared by weighing 0.10 g of silver nitrate into a 50 mL beaker, adding 10 mL of water, dissolving with heating and mixing, the solution containing 1 % of silver nitrate; barium chloride solution at 10 %, prepared by weighing 10.0 g of barium chloride into a 150 mL beaker, adding 100 mL of water and mixing, the solution containing 10 % of barium chloride; and paper pulp, prepared by taking a measured quantity of filter paper, adding water and boiling.

6 Apparatus

A muffle furnace with a maximum temperature not lower than 850 °C and a temperature control tolerance of +/- 20 °C.

7 Samples

The samples are stored in plastic bottles for use.

8 Test procedure

Test portion: the sample is pipetted accurately as shown in Table 1. Table 1 pairs the sulfate concentration range in grams per litre with the volume taken in millilitres, giving 50.00 mL for a range of 0.01 to 1.00, 10.00 mL for a range above 1.00 up to 10.00, and 5.00 mL for a range above 10.00 up to 18.00.

Parallel tests: two parallel tests are carried out. Blank test: a blank test is run together with the test portion.

Determination: the test portion is pipetted according to Table 1 into a 250 mL beaker and water is added to 150 mL. Then 0.5 g of hydroxylammonium chloride is added, the pH is adjusted to 3.1 to 4.4 with the 1+1 aqueous ammonia or the 1+1 hydrochloric acid, 35 mL of anhydrous ethanol is added and the solution is stirred evenly with a glass rod. The solution is heated on a hot plate almost to boiling, removed, and the precipitate is filtered off, the filtrate being received in a 500 mL beaker and made up with water to 300 mL. With constant stirring, the barium chloride solution is added slowly to the filtrate until no more barium sulphate precipitate forms. A little paper pulp is added and the solution is held in a

water bath at 60 °C to 70 °C for 2 h, cooled to room temperature and left standing overnight; alternatively it is heated for 5 min with constant stirring, held near boiling for 1 h, then removed and left to settle for 30 min.

The solution is filtered through slow quantitative filter paper and the filtrate is discarded. The beaker is washed three times with hot 1+99 hydrochloric acid and the washings are filtered, the precipitate is then washed four to five times with hot 1+99 hydrochloric acid, and the paper and precipitate are washed with hot water until the filtrate no longer turns white when checked with the silver nitrate solution.

The barium sulphate precipitate together with the filter paper is transferred into a 30 mL porcelain crucible that has been ignited to constant mass, charred at low temperature, ignited at 800 °C to 850 °C for 30 min, then taken out, placed in a desiccator, cooled to room temperature and weighed; the ignition is repeated to constant mass.

9 Treatment of test data

The sulfate content is expressed as the mass concentration of the sulfate ion. It is obtained from the mass of barium sulphate found for the test material and the mass of barium sulphate found for the blank, both in grams, a barium sulphate to sulfate ion conversion factor of 0.411 6, the unit conversion between millilitres and litres, and the volume of the test portion taken, in millilitres; the result is the mass concentration of the sulfate ion in the precious metal alloy electroplating wastewater, in grams per litre.

The calculated result is expressed to two decimal places.

10 Precision

Repeatability: for two independent test results obtained under repeatability conditions, within the range of mean values given in Table 2, the absolute value of the difference between the two results does not exceed the repeatability limit, and the cases in which it exceeds the repeatability limit are not more than 5 %. The repeatability limit is obtained from the data of Table 2 by interpolation or extrapolation, and the original data of the precision test are given in Annex A.

Table 2 pairs six values of the sulfate ion mass concentration in grams per litre with six repeatability limits: 0.02 with 0.01, 0.12 with 0.02, 0.96 with 0.06, 4.76 with 0.12, 7.00 with 0.21 and 17.44 with 0.46.

Reproducibility: for two independent test results obtained under reproducibility conditions, within the range of mean values given in Table 3, the absolute value of the difference between the two results is not greater than the reproducibility limit, and the cases in which it exceeds the reproducibility limit are not more than 5 %. The reproducibility limit is obtained from the data of Table 3 by interpolation or extrapolation, and the original data of the precision test are given in Annex A.