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National Standard of the People’s Republic of China

GB/T 15072.12-2008

Replaces GB/T 15072.12-1994

Test methods of precious metal alloys

Determination of vanadium contents for silver alloys

Hydrogen peroxide spectrophotometry

贵金属合金化学分析方法

银合金中钒量的测定

过氧化氢分光光度法

(English Translation)

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Foreword

SAC/TC 243 is in charge of this English translation. In case of any doubt about the contents of English translation, the Chinese original shall be considered authoritative.

This standard is an integrated revision of GB/T 15072-1994(*Chemical analysis methods for precious metals and their alloys*)(all parts), which is divided into 19 parts:

—GB/T 15072.1-2008 *Test methods of precious metal alloys Determination of gold content for gold, platinum and palladium alloys potentiometric titration with ferrous sulfate*.

—GB/T 15072.2-2008 *Test methods of precious metal alloys Determination of silver content for silver alloys potentiometric titration with sodium chloride*.

—GB/T 15072.3-2008 *Test methods of precious metal alloys Determination of platinum content for gold, platinum and palladium alloys Current titration with potassium permanganate*.

—GB/T 15072.4-2008 *Test methods of precious metal alloys Determination of palladium content for palladium and silver alloys butanedione dioxime gravimetry*.

—GB/T 15072.5-2008 *Test methods of precious metal alloys Determination of silver content for gold and palladium alloys potentiometric titration with potassium iodide*.

—GB/T 15072.6-2008 *Test methods of precious metal alloys Determination of iridium content for platinum and palladium alloys potentiometric titration with ferrous sulfate*.

—GB/T 15072.7-2008 *Test methods of precious metal alloys Determination of chromium and iron contents for gold alloys Inductively coupled plasma atomic emission spectrometry*.

—GB/T 15072.8-2008 *Test methods of precious metal alloys Determination of copper content for gold, palladium and silver alloys EDTA complexometric back titration with thiourea precipitation*.

—GB/T 15072.9-2008 *Test methods of precious metal alloys Determination of indium content for gold alloys EDTA complexometric back titration*.

—GB/T 15072.10-2008 *Test methods of precious metal alloys Determination of nickel content for gold alloys EDTA complexometric back titration*.

—GB/T 15072.11-2008 *Test methods of precious metal alloys Determination of gadolinium and beryllium contents for gold alloys Inductively coupled plasma atomic emission spectrometry*.

—GB/T 15072.12-2008 *Test methods of precious metal alloys Determination of vanadium content for silver alloys Hydrogen peroxide spectrophotometry*.

—GB/T 15072.13-2008 *Test methods of precious metal alloys Determination of tin, cerium and lanthanum contents in silver alloys Inductively coupled plasma atomic emission spectrometry*.

—GB/T 15072.14-2008 *Test methods of precious metal alloys Determination of aluminium and nickel contents for silver alloys Inductively coupled plasma atomic emission spectrometry*.

—GB/T 15072.15-2008 *Test methods of precious metal alloys Determination of nickel, zinc and manganese contents for gold, silver and palladium alloys Inductively coupled plasma atomic emission spectrometry*.

—GB/T 15072.16-2008 *Test methods of precious metal alloys Determination of copper and manganese contents for gold alloys Inductively coupled plasma atomic emission spectrometry*.

—GB/T 15072.17-2008 *Test methods of precious metal alloys Determination of tungsten content for platinum alloys Tungsten trioxide gravimetry*.

—GB/T 15072.18-2008 *Test methods of precious metal alloys Determination of zirconium and gallium contents for gold alloys Inductively coupled plasma atomic emission spectrometry*.

—GB/T 15072.19-2008 *Test methods of precious metal alloys Determination of vanadium and magnesium contents for silver alloys Inductively coupled plasma atomic emission spectrometry.*

This part is the twelfth of GB/T 15072-2008.

This part replaced GB/T 15072.12-1994 (*Methods for chemical analysis of precious metals and their alloys Silver alloys Determination of vanadium content*) in whole.

The following deviations have been made with respect to the GB / T 15072.12-1994(the previous edition):

—The title of standard is changed from *Methods for chemical analysis of precious metals and their alloys Silver alloys Determination of vanadium content* to *Test methods of precious metal alloys Determination of vanadium content for silver alloys, Hydrogen peroxide spectrophotometry*.

—This part is drafted in accordance with the rules given in the new document including compound and consistency.

—The scope of the standard is extended from AgCuV10-0.2 of the previous edition to this standard is applicable to the determination of vanadium content for alloys of AgCuV. The determination range of this standard (mass fraction) is 0.1% to 0.4%.

—This part is drafted in accordance with the rules given in the GB/T20001.4-2001.

This part was proposed by Nonferrous Metals Industry Association of China;

This part was prepared by SAC/TC 243 Chinese Nonferrous Metal Standardization Technical Committee.

This part is explained by the national non ferrous metal Standardization Technical Committee.

The previous edition of this standard is as follow:

—GB/T 15072.12-1994.

Test methods of precious metal alloys

Determination of vanadium contents for silver alloys

Hydrogen peroxide spectrophotometry

1. Scope

This part specifies a method for the determination of vanadium content in silver alloys.

This part is applicable to determination of silver content between0.1% to 0.4%in AgCuV.

1. Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part. For dated references, subsequent amendments (excluding corrections), or revisions, of any of these publications do not apply to this part. However parties to agreements based on this part are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies.

YS/T 371 *Methods for chemical analysis of precious metals alloys General rules and regulations*.

1. Method abstract

The test portion shall be dissolved in nitric acid. The vanadium (V) was reacted with hydrogen peroxide to form an orange complex in the nitric acid solution of 2.5mol/L. The vanadium contents were determined through testing the absorbance by spectrophotometer at the wavelength of 450nm.

1. Reagent and material

Unless otherwise noted, reagents and utensils are in accordance with YS/T 371.

4.1 Nitric acid,(ρ1.42g/mL).

4.2 Nitric acid solution: The nitric acid (4.1) shall be heated in low temperature until the NO2 has evaporated and the solution is colorless, dilute the solution by isopyknic water.

4.3 Hydrogen peroxide (1+9).

4.4 Vanadium standard storage solution: Weighing 0.5g of vanadium (purity≥99.99%) accurate to 0.0001g and dissolve it by adding 30mL Nitric acid (4.1) in a 300mL beaker. Cover with a surface dish and heating in low temperature until the solution was completely dissolved which has a yellow color(continue to add nitric acid and heat if the solution has a green color ).Transfer the solution into a 500mL one-mark volumetric flask, dilute to the mark with water, and mix well. 1 mL of this solution contains 1mg of vanadium.

4.5 Vanadium standard solution: Transfer 5.00mL of vanadium standard storage solution (4.4) into a 50mL one-mark volumetric flask, dilute to the mark with water and mix well.1 mL of this solution contains 100μg of vanadium.

All reagents and the water used in this standard shall not contain chloridion.

1. Apparatus

Visible spectrophotometer.

1. Sample

The sample shall be degreased with acetone, cleaned with water and then dried, processed into chips and mixed well.

1. Analysis procedure

7.1 Test portion

Weigh 0.1g of test portion, accurate to 0.00001g.

7.2 Determination of the number

Two independent determinations should be required, and average the results.

7.3 Blank test

Blank test shall be done along with the test portion.

7.4 Determination

7.4.1 The portion (7.1) should be placed in a 100 ml beaker. Adding 5mL nitric acid solution (4.2) . Cover with a watch-glass and heating in low temperature until the solution was completely dissolved. Take the watch-glass off and evaporating in low temperature until the solution was approximately dried. Add 8mL nitric acid solution (4.2) into the residue and transfer the solution into a 25mL one-mark volumetric flask.

7.4.2 Dilute the solution with water to 20mL~24 mL, add 0.5mL hydrogen peroxide solution (4.3) and dilute to the mark with water and mix well.

7.4.3 Transfer part of the solution into a 3cm absorption cell. Measure the absorbance of the solutions in spectrophotometer at the wave of 450nm. The reference is the reagent use along with the test portion. Determine the vanadium contents through the working curve.

7.5 Preparation of working curve

7.5.1 Transfer the given volumes (0.00mL, 0.50mL, 1.00mL, 2.00mL, 3.00mL, 4.00mL, 5.00mL, 6.00mL) of vanadium standard solution (4.5) into 25mL volumetric flasks. Add 8mL nitric acid solution (4.2) to each flask. Then perform testing as the details given in 7.4.2.

7.5.2 Transfer part of the solution into a 3cm absorption cell. Measure the absorbance of the solutions in spectrophotometer at the wave of 450nm. The reference is the reagent use along with the test portion.

7.5.3 Set up a calibration curve by plotting the vanadium concentrations on the x-axis and the absorbance on the y-axis.

1. Calculation of analysis results

The mass fraction of vanadium $ω\_{V}$, is calculated according to formula (1), and the value is expressed as % :

$w\_{v}=\frac{m\_{1}×10^{-6}}{m\_{0}}×100$ ----------------------------------------------------- (1)

In the formula:

m1 is the vanadium concentration obtained from the working curve, the unit is microgram(µg).

m0 is the mass of the test portion, the unit is gram(g).

The results should be expressed to two decimal places.

1. Tolerance

Difference of testing results between laboratories shall not be greater than 0.04%.