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National Standard of the People’s Republic of China

 GB/T 15072.5-2008

 Replaces GB/T 15072.5-1994

Test method of precious metal alloys

Determination of silver contents for gold and palladium alloys

Potentionmeter titration with potassium iodide

贵金属合金化学分析方法

金、钯合金中银量的测定

碘化钾电位滴定法

(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 fifth of GB/T 15072-2008.

This part replaced GB/T 15072.5-1994 for *（Methods for Chemical Analysis of Precious Metals and Their Alloys Gold palladium, alloys Determination of silver content）.*

The following deviations have been made with respect to the GB / T 15072.5-1994(the previous edition):

—The standard title is changed from *Methods for Chemical Analysis of Precious Metals and Their Alloys Gold palladium, alloys Determination of silver content* to *Test method of precious metal alloys determination of silver contents for gold and palladium alloys potentionmeter titration with potassium*

*iodide*.

—The scope of the standard is extended from AuAgPt25-6.0,AuAgCu35-5.0,AuAgCu20-30, AuAgCuGd35-5.0, AuAgCuMnGd33.5-3.0-2.6-0.4,PdAgCuAuPtZn30-14-10-10-1.0,PdAgCu52-28,PdAgCu54-21, PdAgCu58-32, PdAgCu65-20 PdAgCu68-27, PdAg40, PdAg80 of the previous edition to this standard is applicable to the determination of silver content for alloys of AuAgPt,AuAgCu,AuAgCuGd,AuAgCuMnGd,AuCuPtAgZn, PdAgCuAuPtZn, PdAgCu, PdAg. The determination range of this standard (mass fraction) is 15% to 90%.

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

Appendix A (Annex A) is informative in this part.

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 15072.5-1994.

Test method of precious metal alloys

determination of silver contents for gold and palladium alloys

potentionmeter titration with potassium iodide

1. Scope

This part specifies a method for the determination of silver content in gold and palladium alloys.

This part is applicable to determination of silver contents between 15% - 90% in AuAgPt, AuAgCu, AuAgCuGd, AuAgCuMnGd, AuCuPtAgZn, PdAgCuAuPtZn, PdAgCu, PdAg alloys.

1. Normative references

The following normative documents contain provisions which, through reference in this, 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 of gold and PdAgCuAuPtZn alloys shall be dissolved in a mixture of hydrochloric acid and nitric acid. The test portion of PdAgCu and AgPd alloys shall be dissolved with nitric acid.

Using silver-silver iodide electrode as the indicating electrode, saturated calomel electrode as the reference electrode, silver contents was determined by potassium iodide titrand in ammoniacal medium. The terminal point was detected by potentionmeter.

1. Reagent and material

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

4.1 Hydrochloric acid( ρ1.19g/ml).

4.2 Nitric acid( ρ1.42g/ml),guarantee reagent.

4.3 Ammonium hydroxide( ρ0.91g/ml).

4.4 Silver standard solution

Weigh 2.0000g of silver metal (purity≥99.99%), It should be placed in a 250 ml beaker, then cover the surface dish and add 40 ml of nitric acid(1+1, guarantee reagent) , Heating the solution until test portion completely dissolved, then excluding oxynitride completely. Remove and cool to room temperature. Washing surface dish and cup wall with water. The solution shall be transferred into a 1000 ml brown one-mark volumetric flask with water, diluting to the mark and mix. 1 ml of this solution contains 2mg of silver. It should be kept in dark place.

4.5 Potassium iodide titrand,[c(KI)about0.020mol/L]

4.5.1 Preparation

Weigh3.32g of potassium iodide (mass fraction),dissolved with water, The solution shall be transferred into 1000 ml one-mark volumetric flask with water, diluting to the mark and mix.

4.5.2 Standardization. Standardization and determination of portions should be take place at the same time.

Transfer 20.00 mL silver standard solution into a 150 mL beaker thrice, put the three beakers on electric furnace, heat it under low temperature until them almost dry. Wash the surface dish and cup wall with water, Adding 4mL ammonium hydroxide and 20mL water. use silver-silver iodide electrode as the indicating electrode, saturated calomel electrode as the reference electrode, silver contents was determined by potassium iodide titrand, end point of titration is potential abrupt to the maximum. The range volume of potassium iodide titrand shouldn’t be exceed 0.05mL in twice standardizations. The results should be averaged.

The actual concentration of potassium iodide titrand is calculated according to formula (1):

 $c=\frac{c\_{0}.V\_{1}×10^{−3}}{107.87×V\_{2}}$ -- -- -- -- -- -- -- -- -- -- -- (1)

In the formula:

C is the actual concentration of potassium iodide titrand, the unit is moles per liter(mol/L).

*C0*is the mass concentration of silver standard solution, the unit is milligram per milliliter(mg/mL).

*V1* is the volume of transfer silver standard solution, the unit is milliliter(mL).

*V2* is the volume of potassium iodide titrand which is consumed in standardization by silver standard solution , the unit is milliliter(mL).

107.87 is the molar mass of silver, the unit is gram per mole(g/mol).

1. Apparatus

5.1 Potentiometer, accuracy to 1mV.

5.2 Indicating electrode, silver-silver iodide electrode.

5.3 Reference electrode, saturated calomel electrode.

5.4 Balance, sensitive quality to 0.01mg.

1. Sample

Sample should be rolled into thin sheets of thickness, remove oil stains with acetone, and cut into debris, washed with water, dried and mixed well.

1. Analysis procedure

7.1 Test portion

Weigh the portion according to Table 1, accurate to 0.00001g.

Table 1

|  |  |
| --- | --- |
| Mass fraction of silver /% | Test portion /g |
| 15.00~25.00 | 0.16 |
| >25.00 to 30.00 | 0.14 |
| >30.00 to 70.00 | 0.12 |
| >70.00 to 90.00 | 0.10 |

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 Dissolving of the portion

7.4.1.1 The PdAg alloy portion should be placed in a 150mL beaker, then cover the surface dish and add 5mL nitric acid, Heating the solution at low temperature until test portion completely dissolved , evaporating to damp state, cool drown. Wash surface dish and cup wall with water. After that, add 4mL ammonium hydroxide and 30mL water. Next step follow the 7.4.3.

7.4.1.2 The PdAgCu alloy portion should be placed in a 150mL beaker, then cover the surface dish and add 5mL nitric acid, Heating the solution at low temperature until test portion completely dissolved , evaporating to damp state, cool drown. Wash surface dish and cup wall with water. After that, add 5mL hydrochloric acid, cover the surface dish, put it on electric furnace and shall be evaporated to0.5mL in low temperature, cool drown. Wash surface dish and cup wall with water.

7.4.1.3 The gold or PdAgCuAuPtZn alloys portion should be placed in a 150mL beaker, then cover the surface dish and add 40mL hydrochloric acid and 7mL nitric acid, Heating the solution at low temperature until test portion completely dissolved , evaporating to0.5mL. Wash surface dish and cup wall with water.

7.4.2 Dispose of the residue

7.4.2.1 Adding 40mL water into the residue (7.4.1.2)or(7.4.1.3),the solution shall be boiled to clear. Take it down, and cool to room temperature in dark place.

7.4.2.2 The top clear solution shall be filtrated by medium filter paper, wash the precipitate in baker and filter paper with water 3 times.

7.4.2.3 The precipitate in filter paper shall be dissolved into the original baker by 4mL ammonium hydroxide little at a time, wash the filter paper 3 times with water. Shaking the baker until all the precipitates have been dissolved, adding 20 mL water along the baker wall.

7.4.3 Titration

Putting the silver-silver iodide electrode as the indicating electrode and saturated calomel electrode as the reference electrode into test the solution (7.4.1.1)or(7.4.2.3), silver contents can be determined by potassium iodide titrand, end point of titration is the potential abrupt to the maximum.

1. Calculation of analysis results

The mass fraction of silver $ω\_{Ag}$ is calculated according to formula (2), and the value is expressed as % :

  -- -- -- -- -- -- -- -- -- -- -- (2)

In the formula:

C is the actual concentration of potassium iodide titrand, the unit is moles per liter(mol/L).

V1 is the volume of potassium iodide titrand which is consumed in titration by test solution , the unit is milliliter(mL).

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

107.87 is is the molar mass of silver, the unit is gram per mole(g/mol).

The results should be expressed to two decimal places.

1. Tolerance1

Differences between laboratory analysis results allowable should be not more than listed in table 2.

Table 2. %

|  |  |
| --- | --- |
| Mass fraction of palladium | Tolerance1 |
| 15.00~25.00 | 0.12 |
| >25.00~35.00 | 0.15 |
| >35.00~80.00 | 0.20 |
| >80.00~90.00 | 0.25 |

Appendix A

(informative)

Manufacture method of silver-silver iodide electrode

Taking a pure silver wire of 1 millimeter in diameter and 180 millimeter in length, It should be polished by abrasive paper. One end coil 5 spiral circles about 5 millimeter in diameter, washing by water, then it should be putted into potassium iodide solution (0.015mol/L), using silver wire as anode and platinum wire as cathode, polarization by 1 milliampere direct-current main in 2000 second, silver-silver iodide electrode has been made.