ICS 77. 120. 99

H 68

National Standard ofthe People’s Republic of China

GB/T 15072.14-2008

 Replaces GB/T 15072.14-1994

Test method of precious metal alloys

Determination of aluminium and nickel contents for silver alloys

Inductively coupled plasma atomic emission spectrometry

贵金属合金化学分析方法

银合金中铝和镍量的测定

电感耦合等离子体发射光谱法

(*English Translation*)

Issue date:2008-03-31Implementation date: 2008-09-01

Issued by General Administration of Quality Supervision, Inspection and

Quarantine of the people's Republic of China

Standardization Administration of the people's Republic of China

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 fourteenth of GB/T 15072-2008.

This part replaces GB/T 15072.14-1994（*Methods for Chemical Analysis of Precious Metals and Their Alloys silver alloys Determination of aluminium content*）

The following deviations have been made with respect to the GB / T 15072.14-1994(the previous edition):

—The title of standard is changed from *Methods for Chemical Analysis of Precious Metals and Their Alloys silver alloys Determination of aluminium content*to *Test methods of precious metal alloys Determination of aluminium and nickel contents for silver alloys Inductively coupled plasma atomic emission spectrometry.*

—Inductively coupled plasma atomic emission spectrometry replaces spectrophotometry of the previous standard.

—The scope of the method is extended from AgCuNiAl20-2-1 to AgCuNiAl, AgCuNiCe, AgCuNi and AgMgNi of which AgCuNiCe, AgCuNi and AgMgNi,are new.

—The range of aluminium content (mass fraction)is extended from0.5~1.2% to 0.1~2.5%.

—The simultaneous determination of nickel element was added, and the determination range (mass fraction) was 0.1~2.5%.

—Repeatability and relative deviation replace the allowed deviation of original standard.

Appendix A(Annex A) is informative.

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

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

The previous editions of this standard are as follows:

— GB/T 15072.14-1994.

Test Methods of precious metal alloys

Determination of Aluminium and Nickel contents for silver alloys

Inductively Coupled Plasma Atomic Emission Spectrometry

1 scope

This part specifies a method for the determination of aluminum contents and nickel in silver alloys.

This part applicable to determination of aluminium and nickel contents between 1.5% and 2.5% in AgCuNiAl, AgCuNiCe, AgCuNi, and AgMgNi alloys.

2 Normative references

The following normative documents contain provisions which, through reference in this test, 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 resent 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*.

3 Principle

The test portion was dissolved in nitric acid, add hydrochloric acid precipitated the silver into silver chloride. Nebulization of the solution into a ICP optical emission spectrometer and measure the intensity of aluminum  and nickel.

4 Reagents and materials

Unless otherwise stated, use only reagents and materials as specified in YS/T 371.

4.1 Argon gas(ω(Ar)≥99.95%).

4.2 hydrochloric acid (ρ1.19g/ml).

4.3 nitric acid (ρ1.42g/ml).

4.4 hydrochloric acid (1+9).

4.5 hydrochloric acid (1+4).

4.6 Aluminium standard stock solutionWeighing 0.1000g of aluminum metal (ω(Al)≥99.99%(mass fraction))and transfer into a 200 ml beaker. Add 5ml of water and 5ml of hydrochloric acid (4.2). Covered with a surface dish, heating at low temperature until test portion completely dissolved, When solution cool to room temperature, the solution shall be transferred into a 100ml one-mark volumetric flask with hydrochloric acid (4.5),dilute to the mark and mix it well.1 ml of this solution contains 1mg of aluminium.

4.7 Nickel standard stock stock solution Weighing 0.1000g of nickel metal (ω(Ni)≥99.99%(mass fraction))and transfer into a 200 ml beaker. Add 5ml hydrochloric acid (4.2). Cover with a surface dish, heating at low temperature until test portion completely dissolved, When solution cool to room temperature, transfer it into 100ml one-mark volumetric flask with hydrochloric acid (4.4), dilute to the mark and mix it well.1 ml of this solution contains 1mg of nickel.

5 Apparatus

Inductively coupled plasma atomic emission spectrometry.

The instrument used will be satisfactory if, after optimizing in accordance with the manufacturer’s instructions, it meets the performance criteria as the following.

5.1 Light source

Argon plasma light source, the maximum output power of the generator is not less than 1.3KW.

5.2 Resolution

The optical resolution is not more than 0.010nm at about 200nm and not more than 0.020nm at about 400nm.

5.3 Instrument stability

The stability of the instrument within one hour is less than 2.0%, expressed as RSD.

5.4 Detection limit of spectrometer

The detection limits of aluminum and nickel in blank solution are not more than 0.05mg/l.

6 Sample

The sample in the form of drillings or flakes shall be processed into chips, then cleaned with acetone to degrease, rinsed with water, dried and mixed well.

7 Analysis procedure

7.1 Test portion

Weigh, to the nearest 0.1mg, 0.1g of the test sample.

Measure independently twice and calculate the mean value.

7.2 Blank test

Blank test shall be done along with the test portion.

7.3 Analysis procedure

7.3.1 Preparation of the test solution

Place the test portion (7.1) into a 150 ml beaker. Add 2ml of nitric acid (4.3), Cover with a surface dish, heating in low temperature until the solution was completely dissolved. Add 20ml water and 2ml hydrochloric acid (4.2), stirring it, keeping boiling for 20minuntil silver precipitated completely. The precipitate of silver chloride shall be filtered with medium speed filter paper, Wash the surface dish, beaker wall, precipitate and filter paper with hydrochloric acid four times each. Collect the filtrate into a 100ml one-mark volumetric flask, Dilute to the mark with hydrochloric acid (4.7)and mix it well.

7.3.2 Preparation of the calibration solution

Transfer appropriate volume of nickel and aluminum standard solution (4.6,4.7)respectively into a series of 100 ml one-mark volumetric flasks, which is diluted step by step. Dilute to the mark with hydrochloric acid (4.4) and mix it well. Such, obtain the calibration solution shown in Table 1.

Table 1 in microgram per millilitre

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Calibration solution label | 0# | 1# | 2# | 4# | 5# |
| Concentration of aluminum | 0.00 | 0.50 | 5.00 | 10.00 | 30.00 |
| Concentration of nickel | 0.00 | 0.50 | 5.00 | 10.00 | 30.00 |

7.4 Determination

7.4.1 Condition of determination

Refer to Appendix A, optimize the condition of instrument to set parameter.

7.4.2 Analytical line

Recommended wavelengths are given in Table 2.

Table 2

|  |  |
| --- | --- |
| Element | Wavelength/nm |
| Al | 396.153 |
| Ni | 231.604 |

7.4.3 Preparation of working curve

Input mixed differential standard solution(7.3.2) into ICP-AES to determine in accordance with the prepared procedure.Establish the calibration curve using the concentrations of elements to be measured on the x-axis and the corresponding wavelength intensities on the y-axis. The correlation coefficient of calibration curve shall be higher than 0.9999.

7.4.4 Measurement of the test solution

Input the test sample solution (7.3.1) to ICP-AES in accordance with the prepared procedure.

8 Expression of results

The mass fraction of aluminium and nickel, expressed as a percentage, is given by Formula (1):

 (1)

In the formula:

 is the concentration of aluminum or nickel obtained from the working curve, in µg/ml.

V is the volume of the test solution, in ml.

*m0* is the mass of the test portion, in g.

Calculate the mass fraction to the third decimal place in the case of a mass fraction more than 1%, the second decimal place in the case of a mass fraction less than 1%.

9 Precision

9.1 Repeatability

Within the mean values given below, the absolute difference in results of two parallel determinations under repeatability conditions shall not be greater than repeatability limit. If the difference exceeds the limit, it shall be not more than 5%.The repeatability limit is obtained by linear interpolation according to the data in Table 3.

Table 3 in %

|  |  |  |  |
| --- | --- | --- | --- |
| Mass fraction of Nickel | 0.10 | 0.30 | 2.00 |
| repeatability limit of Nickel | 0.004 | 0.01 | 0.07 |
| Mass fraction of aluminum | 0.1 | 1 | 2 |
| repeatability limit of aluminum | 0.006 | 0.04 | 0.06 |

9.2 Tolerance

The difference in analysis results between laboratories shall not be greater than tolerance given in Table 4.

Table 4 in %

|  |  |
| --- | --- |
| Mass fraction of Nickel and Aluminum | relative tolerance |
| 0.10~1.00 | 6 |
| ≥1.00～2.00 | 4 |

Annex A

(informative)

 Working conditions of the apparatus

Optimize the instrument in accordance with the operating manual. Set up the parameters refer to as follows:

A.1 High Radio Frequency power of the generator is 1.2KW.

A.2 Argon flow-rate: of which cooling gas is 15 l/min, protection gas is 0.8l/min, and carrier gas is 0.3 l/min.

A.3 Vertical observation height is 15 mm.

A.4 Integration time is 5 s.

A.5 Flowvelocity of injection pump is 15ml/min.