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National Standard ofthe People’s Republic of China

GB/T 15072.15-2008

Replaces GB/T 15072.14-1994

Test method of precious metal alloys

Determination of nickle,zinc,andmanganese contents for gold,silver and palladium alloys

Inductively coupledPlasma atomic emission spectrometry

贵金属合金化学分析方法

金、银、钯合金中镍、锌和锰量的测定

电感耦合等离子体原子发射光谱法

(*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 fifteenth of GB/T 15072-2008.

This part replaces GB/T 15072.15-1994(*Methods for Chemical Analysis of Precious Metals and Their Alloys Gold, silver and palladium alloys Determination of nickel, zinc and manganese contents*)

The following deviations have been made with respect to the GB / T 15072.15-1994(the previous edition):

—The title of standard is changed from *Methods for Chemical Analysis of Precious Metals and Their Alloys Gold, silver and palladium alloys Determination of nickel, zinc and manganese contents* to *Test method of precious metal alloys Determination of nickel, zinc and manganese contents for gold, silver and palladium alloys Inductively coupled plasma atomic emission spectrometry*.

—Inductively coupled plasma atomic emission spectrometry replaces flame atomic absorption spectroscopy of the previous standard.

—The scope of the method is extended from AuCuNiZnMn18-1.8-0.7-0.03、AuCuNiZn22-2.5-0.7、AuCu-NiZn30-3-1、AuNiCr5-1、AuNiCr5-2、AgCuNiA120-2-1、PdAgCuAuPtZn30-14-10-10-1 alloys to AuCuNiZn(Mn)、AuNiCr、PdAgCuAuPtZn、AuGeNi(Cu)、AuNiFeZr alloys, of which AuGeNi(Cu) and AuNiFeZr are new.

—The range of nickel (mass fraction) is extended from 1% ~5% to 0.5% ~ 6%.The range of zinc is extended from 0.5% ~1.5% to 0.5%~ 6%, The range of manganese is extended from 0.01% ~ 0.05% to 0.01% ~0.5%;

—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 243 Chinese Nonferrous Metal Standardization Technical Committee.

The previous editions of this standard are as follows:

— GB/T 15072.15-1994.

Test method of precious metal alloys

Determination of nickel,zinc and manganese contents for gold,silver and palladium alloys

Inductively coupled plasma atomic emission spectrometry

1 scope

This part specifies a method for the determination of nickel, zinc and manganese in gold, silver and palladium alloys.

This part applies to the determination of nickel, zinc and manganese in AuCuNiZn. AuCuNiZnMn, AuNiCr,PdAgCuAuPtZn, AuGeNi, AuGeNiCu and AuNiFeZr alloys.The determination range (mass fraction) is Ni and Zn 0.5% - 6%, Mn 0.01% - 0.5%.

2 Normative references

The following normative documents contain provision 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 hydrochloric acid and nitric acid. Silver was separated and precipitated by hydrochloric acid and gold, platinum, palladium and part of copper were separated and reduced by hydrazine hydrate. Yttrium was added as internal standard. The contents of nickel, zinc and manganese were calculated by inductively coupled plasma atomic emission spectrometry (ICP-AES).

4 Reagents and materials

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

4.1Argon(w(Ar)≥99.95%).

4.2Hydrochloric acid(ρ 1.19 g/mL).

4.3Nitric acid(ρ 1.42 g/mL).

4.4 Mixed acid of hydrochloric acid and nitric acid (3+1),prepare it just before using.

4.5Hydrochloric acid (1+9).

4.6 Hydrazine hydrate(N2H4·H2O,Content not less than 80).

4.7 Nickel standard stock solution

Weighing 0.1000g metallic nickel (ω(Ni) ≥ 99.99%) and transfer into a 200mL beaker. Add 10mL Mixed acid of hydrochloric acid and nitric acid (4.4), cover with surface dish, Heating the solution at low temperature until completely dissolved. Cool to room temperature, transfer it into a 100 mL one-mark volumetric flask with hydrochloric acid (4.5) and dilute to the mark. Mix it well. 1mL of this solution contains 1mg nickel.

4.8 Zinc standard stock solution. Weighing 0.1000g of metal zinc (ω(Zn) ≥ 99.99%) and transfer into a 200 mL beaker. Add 10mL hydrochloric acid (4.5), cover with surface dish, heating at low temperature until completely dissolved. Cool to room temperature, transfer it into a 100 mL one-mark volumetric flask with hydrochloric acid (4.5) and dilute to the mark. Mix it well. 1mL of this solution contains 1mg zinc.

4.9 Manganese standard stock solutionWeighing0.1000g metal manganese (ω(Mn) ≥ 99.99%) and transfer into a 200 mL beaker. Add 5 mL water and 5 mL nitric acid (4.3), cover with surface dish, heat at low temperature until completely dissolved. And boil to remove nitrogen oxides. Cool to room temperature, transfer it into a 100 mL one-mark volumetric flask with hydrochloric acid (4.5) and dilute to the mark. Mix it well.1mL of this solution contains 1mg manganese.

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4.10 Yttrium standard stock solution

Weigh 0,1270g of yttrium oxide(Ⅲ) (purity≥99.99%(mass fraction)) and transfer into a 200 mL beaker. Add 5 mL of water and 5 mL of hydrochloric acid (4.2). Cover with a watch-glass and heat gently until completely dissolved. Cool to room temperature and transfer into 100 mL one-mark volumetric flask with hydrochloric acid (4.5) and dilute to the mark and mix. 1 mL of this solution contains 1mg of yttrium.

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.

5.1Light 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 Nickel, Zinc and Manganese in blank solution are not more than 0.01mg/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 the sample according to table 1 and accurate to 0.0001g.

Table 1

|  |  |
| --- | --- |
| Mass fraction of manganese/% | Test portion /g |
| ≤0.1 | 0.2 |
| >0.1 | 0.1 |

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 200 mL beaker, add 20 mL mixed acid (4.4), cover with a surface dish, heat to dissolves the metal completely at low temperature(acid can be added repeatedly), and evaporate to nearly dry, add 2 mL hydrochloric acid (4.2), evaporate until nearly dry and repeat three times. Rinse the surface dish and beaker wall with hydrochloric acid (4.5) to a volume of about 40 mL, stir until dissolve the soluble salts, heat and boil to precipitate silver, add 1.5 mL hydrazine hydrate (4.6) to the test solution under stirring, cover the watch glass, heat and boil at 150℃ for 30 min until the solution is clear and cool to temperature. Rinse the watch glass and beaker wall with hydrochloric acid (4.5), filter with glass-fritted funnel (4.11), wash funnel wall and precipitation with hydrochloric acid (4.5) for five times. The filtrate is taken into a 100 mLone-mark volumetric flask, added with 1.00 mL yttrium standard solution (4.10) and diluted to the mark with hydrochloric acid (4.5). Mix it well.

7.3.2 Preparation of the calibration solution

Transfer appropriate volume of nickel, zinc and manganese standard sock solutions (4.7.4.8.4.9) respectively into a series of 100 mL one-mark volumetric flasks, which is diluted step by step. Add 1,00 mL of yttrium standard solution (4.9) respectively, dilute to the mark with hydrochloric acid (4.5) and mix. Such, obtain the calibration solution shown in Table 1.

Table 2 in microgram per millilitre

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Calibration solution label | 1# | 2# | 3# | 4# | 5# | 6# |
| Concentration of nickel | 0.50 | 2.00 | 10.00 | 25.00 | 50.00 | 70.00 |
| Concentration of zinc | 0.50 | 2.00 | 10.00 | 25.00 | 50.00 | 70.00 |
| Concentration of magnganese | 0.10 | 1.00 | 10.00 | 25.00 | 50.00 | 70.00 |
| Concentration of yttrium as internal standard | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 |

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

The recommended wavelengths of analytical lines are given in Table 3.

Table 3

|  |  |
| --- | --- |
| Elements | Wavelength/ nm |
| Y (as internal standard) | 371.029 |
| Ni | 346.165 |
| Zn | 206.200 |
| Mn | 259.372 |

7.4.3 Preparation of working curve

According to the approximate concentration range of elements to be measured in test solution, choose to input 1#～4# or 3#～6# 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 nickel, zinc and manganese, expressed as a percentage, is given by Formula (1):

 (1)

In the formula:

 is the concentration of nickel, zinc and manganese obtained from the working curve, inµ

g/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 4.

Table 4 in %

|  |  |  |  |
| --- | --- | --- | --- |
| Mass fraction of Nickel Ni | 0.50 | 2.00 | 6.00 |
| Repeatability limit of Nickel | 0.02 | 0.05 | 0.07 |
| Mass fraction of zinc | 0.50 | 1.00 | 6.00 |
| Repeatability limit of zinc n | 0.02 | 0.05 | 0.10 |
| Mass fraction of manganese | 0.01 | 0.10 | 0.50 |
| repeatability limit of manganese | 0.001 | 0.002 | 0.007 |

9.2 Tolerance

The difference in analysis results between laboratories shall not be greater than tolerance given in Table 5.

Table 5 in %

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Mass fraction of Ni | relative tolerance of Ni | Mass fraction of Zn | relative tolerance of Zn | Mass fraction of Mn | relative tolerance of Mn |
| 0.5-2.00 | 4 | 0.5-1.00 | 5 | 0.01-0.10 | 10 |
| ≥2.00-6.00 | 3 | ≥1.00-6.00 | 3 | ≥0.10-0.50 | 5 |

Annex A

(informative)

 Working conditionsof 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 Flow velocity of injection pump is 15mL/min.