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Non-ferrous Metals Industry Standard of the People's Republic of China

YS/T 959-2014

Methods for chemical analysis of silver- Determination of copper,bismuth,iron,lead,antimony,palladium,selenium and tellurium contents-Spark atomic emission spectrometry

银化学分析法

铜、铋、铁、铅、锑、钯、硒和碲量的测定

火花原子发射光谱法

*(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 drafted in accordance with the rules given in the GB/T 1.1-2009.

This standard was proposed by National Nonferrous Metals Standardization Technical Committee.

This standard was prepared by SAC/TC 243 National Nonferrous Metals Standardization Technical Committee.

Methods for chemical analysis of silver- Determination of copper,bismuth,iron,lead,antimony,palladium,selenium and tellurium contents-Spark atomic emission spectrometry

**1 Scope**

This standard specifies the methods for the determination of copper, bismuth, iron, lead, antimony, palladium, selenium and tellurium in silver.

This standard is applicable to the determination of copper, bismuth, iron, lead, antimony, palladium, selenium and tellurium in silver. The test range is shown in Table 1.

**Table 1**

|  |  |  |  |
| --- | --- | --- | --- |
| Element | test range w/% | Element | test range w/% |
| Copper | 0.0002~0.0500 | Antimony | 0.0002~0.0090 |
| Bismuth | 0.0002~0.0080 | Palladium | 0.0002~0.0100 |
| Iron | 0.0002~0.0100 | Selenium | 0.0002~0.0100 |
| Lead | 0.0002~0.0350 | Tellurium | 0.0002~0.0080 |

**2 Theory**

The power supply excites periodically between the electrode and surface of the sample, and the sample atoms are excited to emit a characteristic spectrum. The spectral intensity value is a function of the element concentration value, and the computer will collect the excitation intensity value automatically and calculate the element content.

**3 Reagents and materials**

Unless otherwise stated, use only reagents of recognized analytical grade and distilled or deionized water or water of equivalent purity are used in the analysis.

3.1 Standard sample: Certified pure silver spectral standard sample, whose content of impurity element covers or partially covers the test range of this method.

3.2 Low and high content standard sample for working curve calibration.

3.3 Hydrochloric acid（ρ=1.19g/mL）

3.4 Absolute ethanol

3.5 Hydrochloric acid (1+9)

3.6 Argon (Volume fraction≥99.99%)

**4 Instrument and auxiliary equipment**

4.1 Spark source atomic emission spectrometer (See annex A for working conditions of the instrument.)

4.2 Argon purifier

4.3 Lathes and cutters

4.4 Automatic tablet press

**5 Analysis**

5.1 Sample preparation

5.1.1 Columnar sample [specification: not less than 20mm (ø)\*15mm (H)]: The sample is processed with lathe to produce a smooth surface without stomata for testing.

5.1.2 Small silver block： place the sample on a tablet press and lock the briquetting, Set the pressure to 20 tons for 5 seconds, start the pressing machine. The surface diameter of the processed sample shall not be less than 25 mm and the thickness shall not be less than 0.5 mm. Small silver blocks shall be processed to a larger piece and then be pressed into the required size.

5.1.3 The processed sample shall be boiled with hydrochloric acid solution（3.5） for 3 to 5 minutes. Take it out and wash with water until no chloride ion is present, then wash with absolute ethanol solution（3.4）and dry with air.

5.2 Inspection and confirmation of instrument status

Turn on the instrument, check the parameters of the instrument in order to ensure that it is in a normal state, otherwise, check out the cause and adjust the parameters

5.3 Construction of working curve

After the instrument and the excitation atmosphere are stable, the standard sample is continuously excited by a spark source atomic emission spectrometer to determine the spectral intensity ratios of impurities in the standard sample(3.1). The working curve of the corresponding element can be obtained with the element mass fraction as abscissa and the line intensity ratio as ordinate.

Note: The internal curing curve of the instrument can be used.

5.4 Standardization of instruments

Before testing, the standard sample or quality control sample shall be tested and the test result shall not be greater than the repeatability limit. Otherwise, drift correction shall be performed until the result meets the requirements.

5.5 Determination

Place the smooth surface of the sample on the instrument excitation platform for testing. Sample shall be changed to different positions for multi-point excitation (more than three points) and take the average.

**6 Calculation**

The detected data will be processed by the instrument automatically according to the working curve of the instrument and the correction factors. The computer will calculate and output the contents of copper,bismuth,iron,lead,antimony,palladium,selenium and tellurium automatically. The result shall be accurate to four decimals.

**7 Precision**

7.1Repeatability limit

The absolute difference between the two test results from two independent tests under the repetitive conditions within the average range given in Table 2 shall not be greater than repeatability limit (r). The case of exceeding the repeatability limit does not exceed 5%, and the repeatability limit is obtained by linear interpolation according to the data of Table 2.

Table 2

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| wCu/% | 0.0005 | 0.0023 | 0.0070 | 0.0147 | 0.0428 |
| r/% | 0.0002 | 0.0003 | 0.0005 | 0.0010 | 0.0024 |
| WBi/% | 0.0008 | 0.0018 | 0.0054 | - | - |
| r/% | 0.0002 | 0.0003 | 0.0005 | - | - |
| WFe/% | 0.0010 | 0.0016 | 0.0028 | 0.0080 | - |
| r/% | 0.0002 | 0.0003 | 0.0005 | 0.0008 | - |
| WPb/% | 0.0008 | 0.0052 | 0.0193 | - | - |
| r/% | 0.0002 | 0.0004 | 0.0016 | - | - |
| WSb/% | 0.0004 | 0.0011 | 0.0038 | 0.0090 | - |
| r/% | 0.0002 | 0.0003 | 0.0005 | 0.0008 | - |
| WPd/% | 0.0009 | 0.0024 | 0.0098 | - | - |
| r/% | 0.0002 | 0.0003 | 0.0008 | - | - |
| WSe/% | 0.0003 | 0.0013 | 0.0030 | 0.0100 | - |
| r/% | 0.0003 | 0.0003 | 0.0004 | 0.0013 | - |
| wTe/% | 0.0008 | 0.0036 | 0.0087 | - | - |
| r/% | 0.0002 | 0.0004 | 0.0006 | - | - |

7.2 Reproducibility limit

The absolute difference between the two test results from two independent tests under reproducible conditions within the average range given in Table 3 shall not be greater than reproducibility limit (R). The case of exceeding the reproducibility limit does not exceed 5%, and the reproducibility limit is obtained by linear interpolation according to the data of Table 3.

Table 3

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| wCu/% | 0.0005 | 0.0023 | 0.0070 | 0.0147 | 0.0428 |
| R/% | 0.0002 | 0.0004 | 0.0008 | 0.0012 | 0.0030 |
| WBi/% | 0.0008 | 0.0018 | 0.0054 | - | - |
| R/% | 0.0003 | 0.0004 | 0.0008 | - | - |
| WFe/% | 0.0010 | 0.0016 | 0.0028 | 0.0080 | - |
| R/% | 0.0004 | 0.0004 | 0.0006 | 0.0010 | - |
| WPb/% | 0.0008 | 0.0052 | 0.0193 | - | - |
| R/% | 0.0003 | 0.0010 | 0.0020 | - | - |
| WSb/% | 0.0004 | 0.0011 | 0.0038 | 0.0090 | - |
| R/% | 0.0003 | 0.0004 | 0.0005 | 0.0010 | - |
| WPd/% | 0.0009 | 0.0024 | 0.0098 | - | - |
| R/% | 0.0003 | 0.0005 | 0.0009 | - | - |
| WSe/% | 0.0003 | 0.0013 | 0.0030 | 0.0100 | - |
| R/% | 0.0002 | 0.0003 | 0.0005 | 0.0020 | - |
| wTe/% | 0.0008 | 0.0036 | 0.0087 | - | - |
| R/% | 0.0003 | 0.0006 | 0.0008 | - | - |

**8 Test report**

The test report shall contain at least the following information:

* Samples;
* Standards (YS/T 959-2014);
* Results and representation;
* Discrepancy from basic analysis steps;
* Anomalies observed in the test;
* Date of test.

Annex A

(informative annex)

The recommended conditions of instrument and system parameters

The recommended conditions of spark source atomic emission spectrometer are shown in Table A1,A2,A3 .

Table A1

|  |  |  |
| --- | --- | --- |
| Analytical element | Wavelength/nm | Internal standard line |
| Cu | 324.754 | Bg7 |
| Bi | 306.772 | Bg7 |
| Fe | 371.994 | Bg7 |
| Pb | 405.782 | Bg7 |
| Sb | 206.838 | Bg7 |
| Pd | 340.458 | Bg7 |
| Se | 196.090 | Bg7 |
| Te | 185.720 | Bg7 |

Table A2

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Optical System | Grating Focal | Diameter of photomultiplier Tube | Grating Line | Read-out System | wavelength Range |
| Paschen-h'unge muanting | 1m | 28mm | 2160 lines/mm | TRS Time analysis & measurement system | 120nm~850nm |

Table A3

|  |  |  |
| --- | --- | --- |
| Parameter | Time /s | Voltage /v |
| Argon washing | 10 | - |
| Pre-excitation | 10 | 25 |
| Spark excitation | 10 | 25 |
| Excitation delay | 0 | - |
| Fatigue Lamp setting | - | 0 |