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Non-ferrous Metals Industry Standard of thePeople'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

银化学分析方法
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

National Nonferrous Metals Standardization Technical Committee （ 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 theGB/T 1.1-2009.

This standard was proposed and prepared by SAC/TC243.

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 contents in silver.

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

Table 1

Element	range /%	Element	range /%
Cu	0.0002～0.0500	Sb	0.0002～0.0090
Bi	0.0002～0.0080	Pd	0.0002～0.0100
Fe	0.0002～0.0100	Se	0.0002～0.0100
Pb	0.0002～0.0350	Te	0.0002～0.0080

2 Summary of method

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

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

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

3.3 Hydrochloric acid（ρ=1.19g/ml）.

3.4 Absolute ethanol.

3.5 Hydrochloric acid, diluted 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 Testing procedure

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 gas pocket for testing.

5.1.2 Small silver block: Place the sample on anautomatic tablet press (4.4), and lock the briquetting, set the pressure of the automatic tablet press to 20 t for 5 s and start. The diameter of the surface 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 (3.5) for 3 min to 5 min. Take it out and rinse with water until no chloride ion present, then rinse with absolute ethanol (3.4) and dry in the 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 Drawing of working curve

When 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 instrument

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 determination. Each 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 exceed the repeatability limit (r). The case of exceeding the repeatability limit shall not exceed 5%, and the repeatability limit is obtained by linear interpolation according to the data of Table 2.

Table2

w_{Cu}/%	0.0005	0.0023	0.0070	0.0147	0.0428
r/%	0.0002	0.0003	0.0005	0.0010	0.0024
W_{Bi}/%	0.0008	0.0018	0.0054	–	–
r/%	0.0002	0.0003	0.0005	–	–
W_{Fe}/%	0.0010	0.0016	0.0028	0.0080	–
r/%	0.0002	0.0003	0.0005	0.0008	–
W_{Pb}/%	0.0008	0.0052	0.0193	–	–
r/%	0.0002	0.0004	0.0016	–	–
W_{Sb}/%	0.0004	0.0011	0.0038	0.0090	–
r/%	0.0002	0.0003	0.0005	0.0008	–
W_{Pd}/%	0.0009	0.0024	0.0098	–	–
r/%	0.0002	0.0003	0.0008	–	–
W_{Se}/%	0.0003	0.0013	0.0030	0.0100	–
r/%	0.0003	0.0003	0.0004	0.0013	–
w_{Te}/%	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 exceed the reproducibility limit (R). The case of exceeding the reproducibility limit shall not exceed 5%, and the reproducibility limit is obtained by linear interpolation according to the data of Table 3.

Table3

w_{Cu}/%	0.0005	0.0023	0.0070	0.0147	0.0428
R/%	0.0002	0.0004	0.0008	0.0012	0.0030
W_{Bi}/%	0.0008	0.0018	0.0054	–	–
R/%	0.0003	0.0004	0.0008	–	–
W_{Fe}/%	0.0010	0.0016	0.0028	0.0080	–
R/%	0.0004	0.0004	0.0006	0.0010	–
W_{Pb}/%	0.0008	0.0052	0.0193	–	–
R/%	0.0003	0.0010	0.0020	–	–
W_{Sb}/%	0.0004	0.0011	0.0038	0.0090	–
R/%	0.0003	0.0004	0.0005	0.0010	–
W_{Pd}/%	0.0009	0.0024	0.0098	–	–
R/%	0.0003	0.0005	0.0009	–	–
W_{Se}/%	0.0003	0.0013	0.0030	0.0100	–
R/%	0.0002	0.0003	0.0005	0.0020	–
w_{Te}/%	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;

—document (YS/T 959-2014);

—results and representation;

—discrepancy from basic analysis steps;

—anomalies observed in the determination;

—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 A.1, A.2, A.3.

Table A.1

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 A.2

Optical System	Grating Focal	Diameter of photo multiplier Tube	Grating Line	Read-out System	Wavelength Range
Paschen-h'ungemuanting	1m	ø28mm	2160 lines/mm	TRS Time analysis & measurement system	120nm-850nm

Table A.3

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