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Application and Development Trends of Spectral Analysis in Draft of Non-Ferrous Metal Standards in China

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Abstract

Spectral analysis was a method of identifying substances, determining their chemical composition and calculating their content based on their spectral characteristics. This paper mainly discussed the application of various spectroscopic techniques, mainly including atomic absorption spectrometry (AAS) inductively coupled plasma emission spectrometry (ICP-AES) X-ray fluorescence spectroscopy (XRF) atomic fluorescence spectroscopy (AFS) direct reading spectroscopy (OES) glow discharge emission spectroscopy (GD-OSE) laser-induced breakdown spectroscopy (LIBS), in the formulation of non-ferrous metal standards in China. The AAS method was the most widely used single-element microanalysis method among the non-ferrous metal standards. The ICP-AES method was good at significant advantages in the simultaneous detection of multiple elements. The XRF method was increasingly used in the determination of primary and secondary trace elements due to its simple sample preparation and high efficiency. The AFS was mostly detected by single-element trace analysis. OES GD-OES and LIBS were playing an increasingly important role in the new demand area for non-ferrous metals. This paper discussed matrix elimination, sample digestion, sample preparation, instrument categories and other aspects of some standards, and summarized the advantages of spectral analysis and traditional chemical analysis methods. The new methods of future spectroscopic technology had been illustrated in the process of developing non-ferrous metal standards.

Keywords

Atomic Absorption Spectroscopy, Inductively Coupled Plasma Emission Spectroscopy, X-Ray Fluorescence Spectroscopy, Atomic Fluorescence

Spectroscopy, Direct Reading Spectroscopy, Glow Discharge Emission Spectroscopy, Laser-Induced Breakdown Spectroscopy, Non-Ferrous Metals, Standard Methods Were Formulated

1. Spectral Analysis and Application

Before the 60s of the 20th century, analytical chemistry was mainly based on traditional classical chemical methods [1]. The standard analysis methods issued subsequently were mainly based on titration spectrophotometry gravimetric method and other chemical methods to determine the primary and secondary components of non-ferrous metal products, including ISO793: 1973, ISO795: 1976, ISO796: 1973, ISO797: 1973 and other standard methods, while the analysis technology mainly focused on single element analysis. Analytical chemistry technology had gradually developed into modern analytical chemistry based on instrumental analysis with the development of high-temperature combustion technology and electrochemical technology. Modern spectral analytical chemistry focused on atomic absorption, atomic emission and X-ray characteristic emission, and mainly relied on multi-element simultaneous/in-situ/online analysis technology to analyze the content morphological characteristics surface depth and other information of primary and secondary trace elements. It is precise because of the discovery of atoms electrons protons neutrons cathode rays and X-rays, and the establishment of Ernest Rutherford's nuclear atom model,

The main spectral analysis techniques of non-ferrous metallurgical analysis mainly included atomic absorption spectroscopy (AAS), inductively coupled plasma emission spectroscopy (ICP-AES), X-ray fluorescence spectroscopy (XRF) and atomic fluorescence spectroscopy (AFS) technology [2]. These analytical methods played an important role in the small-scale pilot expanded and formal production lines of mineral processing & metallurgical processes, and the non-ferrous metal system analysis and testing laboratories had basically introduced a large number of modern analytical equipment. In the traditional non-ferrous metal analysis laboratory supporting analysis system was mainly AAS and AFS spectrometer, such as AAS mainly undertook the determination of copper lead zinc gold silver cadmium magnesium sodium calcium nickel antimony bismuth and other elements in raw ore tailings and metal products, while AFS mainly undertook the determination of arsenic antimony bismuth tin lead selenium tellurium germanium mercury cadmium zinc and other element content. ICP-AES and XRF spectroscopic technologies mainly undertook the analysis of primary secondary and trace elements in non-ferrous metal samples in most non-ferrous metal analysis laboratories. Although AAS and AFS were gradually replaced by modern multi-element simultaneous determination and analysis techniques, they were still important standard technologies in the multi-element analysis system of non-ferrous metal samples, and the instrument combination of AAS and AFS was studied and applied in elemental speciation analysis. Direct reading spectrometer (OES) was mainly used in the analysis of trace impurity elements in high-purity metal materials with a wide wavelength range. Glow discharge emission spectroscopy (GD-OES) was mainly used in surface chemical analysis of metal oxide content analysis Laser-induced breakdown spectroscopy (LIBS) was mainly used in the research stage of rocks, soils, mineral ores and other fields with the advantages of high-energy excitation, simultaneous detection of multiple elements and in situ analysis.

Spectral analysis had become one of the common methods for analyzing the composition of substances as a qualitative and quantitative analysis technique for conventional elements, and it had widely used in the analysis and detection of metal elements in metal materials, geological samples, environmental and water samples, food and crops, petroleum and chemicals, biology and medicine [3] with the development of spectral technology, especially the breakthrough of key modules such as crystal/metal materials, background correction, light source stability, optical path system, detector, data calculation, etc., That was used to further promote the application of spectral technology in the draft of non-ferrous metal standards in China by high-precision quantitative analysis method established by the optical spectrum analyzer.

2. Standard Procedures for Spectral Analysis

The standard procedures for spectroscopy were derived from the verification specifications, operating specifications and general rules of routine spectrometers in daily laboratories. Then, combined with China Nonferrous Metals Standard Quality Information Network (http://www.cnsmq.com/) and CSRES.COM (http://www.csres.com), keyword search was carried out, and AAS ICP-AES XRF AFS OES GD-OES and LIBS had relevant verification standards, general principles and specifications.

The verification regulations of JJD 1001-1991 "Verification Regulation of Atomic-Absorption Spectrophotometer" stipulated the verification conditions, identification methods, national standard sample assessment and evaluation of identification results. GB/T 15337-1994 "General rules for atomic absorption spectrometric analysis" stipulated terms analytical principles reagents and materials instruments and determinations.

JSY/T 0567-2020 "General rules for inductively coupled plasma optical emission spectrometry" specified the principles of analytical methods, the expression of reagents and materials, instruments, samples, analysis steps, analysis results, and precautions for safe use. CSM 01 01 01 04-2006 "Specification for Uncertainty Evaluation of Measurement Results by Inductively Coupled Plasma Emission Spectrometry" included analysis methods and measurement parameter descriptions, mathematical model establishment, identification and evaluation of uncertainty sources, etc.

XRF had been widely used in China with the advantages of measuring a wide variety of elements, simultaneous determination of primary and secondary trace elements, simple sample preparation, fast analysis speed, etc., and has successively promulgated JB/T 11145-2011 "X-ray fluorescence spectrometer", JJG 810-1993 "Wavelength Dispersive X-ray Fluorescence Spectrometer", JY/T 0569-2020 "General rules for wavelength dispersive X-ray fluorescence spectrometry", JB/T 12962.1~3-2016 "Energy dispersive X-ray fluorescence spectrometer, Part 1: General specification, Part 2: Element analyzer, Part 3: Plating thickness analyzer". JJF 1133-2005 "Calibration Specification of Gold Gauge Utilizing X-ray Fluorescence Spectrometry" specifies the metrological characteristics, calibration conditions, calibration terms, and calibration methods, calibration result expression, and recalibration interval.

GB/T 21191-2007 "Atomic fluorescence spectrometer" stipulated the classification, requirements, experimental methods, inspection rules and standards, packaging, transportation and storage of atomic fluorescence spectrometer, etc., GB/T 32266-2015 "Method of performance testing for atomic fluorescence spectrometer" specified the method of atomic fluorescence spectrometer performance determination, suitable for single-channel, double-channel and multi-channel atomic fluorescence spectrometers in the laboratory.

JJD1015-1991 "Verification Regulation of Inductively Coupled Plasma spectroanalyzer" specified periodic verification requirements, and can be used with reference to similar instruments of different manufacturers and models.

GB/T 19502-2004 Surface chemical analysis-Glow discharged optical emission spectrometry (GD-OSE)-Introduction to use (ISO14707:2000, IDT). GB/T 32996-2016 Surface chemical analysis-Analysis of metal oxide filmed by glow-discharge optical emission spectrometry (ISO/TS 25138:2010, IDT). GB/T 32997-2016 Surface chemical analysis-General procedured for quantitative compositional depth profiling by glow discharge optical emission spectrometry (ISO 11505:2012, IDT).

GB/T38257-2019 "Laser-induced breakdown spectroscopy" stipulated terms and definitions, basic principles, test conditions, equipment and devices, samples, test procedures, data processing and test reports, JJF (non-ferrous metals) 0008-2021 "Calibration Specification for Laser Induced Breakdown Spectrometers" for the calibration of laser-induced breakdown spectrometers for the composition analysis of non-ferrous metal solid samples. This specification dealt with calibration of wavelength indication errors and repeatability, detection limits, repeatability, and stability. T/CNIA 0109-2021 "Analysis method for non-ferrous metal materials-General rules of application for laser induced breakdown spectroscopy" included general requirements for method principles, instrumentation, test environment, samples, analytical steps, data processing, test reports and safety protection, and it was suitable for qualitative, semi-quantitative and quantitative analysis of metallic and partial non-metallic elements in samples by solid injection using laser-induced breakdown spectrometer.

3. Application

According to the literatures published on https://openstd.samr.gov.cn/bzgk/gb/, http://www.csres.com, http://www.cnsmq.com, the following spectrometric methods including Atomic Absorption Spectrometry (AAS), Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Direct reading spectroscopy (OES), X-ray fluorescence spectroscopy (XRF), Atomic fluorescence spectroscopy (AFS), Glow discharge emission spectroscopy (GD-OES), and Laser-induced breakdown spectroscopy (LIBS) were frequently applied in the draft of non-ferrous metals standards.

3.1. Atomic Absorption Spectrometry (AAS)

The atomic absorption spectroscopy method was based on the resonance absorption of the characteristic radiation from the light source by the ground state atoms of the measured element in the steam phase, and the characteristic absorption spectrum of the atom was produced, and its absorption value was linearly related to the content of the element to be measured in the sample.

AAS had been used since the 50s of the 20th century. Gao Jieping and Li Huachang [4] [5] comprehensively evaluated the progress made by AAS instrument companies at home and abroad in light source, background correction, atomizer, optical path and detector, automation and intelligence, and multi-element simultaneous determination in the 90s. Li Bing [1] pointed out that the development of AAS instrument for multi-element sequential determination of self-continuous light source-high-resolution optical system, AAS spectroscopy instrument had been re-focused.

AAS had been used as the standard analysis method for more than thirty years, mainly used for the determination of trace elements in raw materials, intermediate products, concentrates, metal alloys and their compounds in the non-ferrous metal dressing and metallurgical process, such as copper-lead-zinc raw original and tailing, scopper ores and tailings, laterite nickel ores, copper magnetite, copper concentrates, zinc concentrates, lead and lead alloys, tungsten, molybdenum and other samples. AAS had been used as the arbitration method. AAS in major non-ferrous metal analysis standards was given in **Table 1**.

In order to solve the problems of interference of coexisting elements to be measured or low strength of the element to be measured, it was usually done by either eliminating the interference element, enriching the element to be measured, or adding a signal enhancer during the sample preparation process. The contents of silver [6], zinc [7] and aluminum [8] in tin-lead solder samples were determined by AAS, and the tin was separated by hydrochloric acid-hydrobromic acid volatilization after dissolving the sample by hydrobromic acid, hydrochloric acid and hydrogen peroxide to eliminate the influence of matrix tin on the determination. The contents of silver [9], iron [10], copper, lead, bismuth [11], magnesium, nickel, manganese and palladium [12] in gold ingot samples were determined by AAS, and the hydrochloric acid medium test solution was extracted by hydrochloric acid decomposition test material, ethyl acetate extraction, and the hydrochloric acid medium test solution was prepared by aqueous phase concentration to eliminate the influence of matrix gold on the determination.

Table 1. AAS in major non-ferrous metal analysis standards.

No.	Standard No.	Standard Name
1	YS/T 509.1-2008	Methods for chemical analysis of spodumene and lepidolite concentrates—Determination of lithium oxide sodium oxide and potassium oxide contents—Flame atomic absorption spectometric method
2	YS/T 509.2-2008	Methods for chemical analysis of spodumene and lepidolite concentrates—Determination of rubidium oxide and caesium oxide contents—Flame atomic absorption spectometric method
3	YS/T 509.8-2008	Methods for chemical analysis of spodumene and lepidolite concentrates—Determination of calcium oxide and magnesium oxide content—Flame atomic absorption spectrometric method
4	YS/T 1115.1-2016	Methods for chemical analysis of copper ores and tailings—Part 1: Determination of copper content—Flame atomic absorption spectrometric method
5	YS/T 1115.2-2016	Methods for chemical analysis of Copper ores and tailings—Part 2: Determination of lead content—Flame atomic absorption spectrometric method
6	YS/T 1115.3-2016	Methods for chemical analysis of copper ores and tailings—Part 3: Determination of zinc content—Flame atomic absorption spectrometric method
7	YS/T 1115.4-2016	Methods for chemical analysis of copper ores and tailings—Part 4: Determination of nickel content—Flame atomic absorption spectrometric method
8	YS/T 1115.5-2016	Methods for chemical analysis of copper ores and tailings—Part 5: Determination of cobalt content—Flame atomic absorption spectrometric method
9	YS/T 1115.6-2016	Methods for chemical analysis of copper ores and tailings—Part 6: Determination of cadmium content—Flame atomic absorption spectrometric method
10	YS/T 1115.7-2016	Methods for chemical analysis of copper ores and tailings—Part 7: Determination of manganese content—Flame atomic absorption spectrometric method
11	YS/T 1115.8-2016	Methods for chemical analysis of copper ores and tailings—Part 8: Determination of magnesium content—Flame atomic absorption spectrometric method
12	YS/T 53.1-2010	Methods for chemical analysis of copper, lead, zinc original and tailing ores—Part 1: Determination of gold content—Fire assay-flame atomic absorption spectrometric

Continued

13	YS/T 53.2-2010	Methods for chemical analysis of copper, lead, zinc original and tailing ores—Part 2: Determination of gold content—Separation and preconcentration by flow injection and 8531 fiber micro-column—the flame atomic absorption spectrum method
14	YS/T 53.3-2010	Methods for chemical analysis of copper, lead, zinc original and tailing ores—Part 3: Determination of silver content—Flame atomic absorption spectrometry
15	YS/T 820.1-2012	Methods for chemical analysis of laterite nickel ores—Part 1: Determination of nikel content—Flame atomic absorption spectrometry
16	SN/T 2763.5-2013	Chemical analysis of nickel laterite ore—Part5: Determination of copper, zinc and chrome content—Flame atomic absorption spectrometry
17	YS/T 1047.1-2015	Methods for chemical analysis of copper magnetite—Part 1: Determination of copper content-2,2-biquinoly spectrophotometric method and flame atomic absorption spectrometric method
18	YS/T 1047.13-2020	Methods for chemical analysis of copper magnetite—Part 13: Determination of mercury content—Solid sampling and direct mercury analysis method and cold vapour atomic absorption spectrometry
19	YS/T 1171.2-2017	Methods for chemical analysis of regenerated zinc material—Part 2: Determination of lead content—The atomic absorption spectrometric method and Na2EDTA titrimetric method
20	YS/T 1171.8-2017	Methods for chemical analysis of regenerated zinc material—Part 8: Determination of mercury content—Atomic fluorescence spectrometry and cold atomic absorption spectrometric method
21	YS/T 1171.12-2021	Methods for chemical analysis of regenerated zinc material—Part 12: Determination of indium content—The atomic absorption spectrometric method
22	YS/T 252.3-2007	Methods for chemical analysis of nickel matte—Determination of cobalt content—Flame atomic absorption spectrometric method
23	YS/T 252.7-2020	Methods for chemical analysis of nickel matte- Part 7: Determination of silver content—Flame atomic absorption spectrometric method
24	GB/T 3884.2-2012	Methods for chemical analysis of copper concentrates—Part 2: Determination of gold and silver contents—Flame atomic absorption spectrmetric method and fire assay method
25	GB/T 3884.4-2012	Methods for chemical analysis of copper concentrates—Part 4: Determination of magnesium oxide content—Flame atomic absorption spectrophotometry method

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26	GB/T 3884.6-2012	Methods for chemical analysis of copper concentrates—Part 6: Determination of lead, zinc, cadmium and nickel content—Flame atomic absorption spectrometry method
27	GB/T 3884.11-2005	Methods for chemical analysis of copper concentrates—Determination of mercury content—Cold atomic absorption spectrometric method
28	GB/T 3884.14-2012	Methods for chemical analysis of copper concentrates—Part 14: Determinnation of gold and silver—Fire assay gravimetric and flame atomic absorption spectrmetric method
29	GB/T 8151.5-2012	Methods for chemical analysis of zinc concentrates—Part 5: Determination of lead content—The flame atomic absorption spectrometric method
30	GB/T 8151.6-2012	Methods for chemical analysis of zinc concentrates—Part 6: Determination of copper content—The flame atomic absorption spectrometric method
31	GB/T 8151.8-2012	Methods for chemical analysis of zinc concentrates—Part 8: Determination of cadmium content—The flame atomic absorption spectrometric method
32	GB/T 8151.12-2012	Methods for chemical analysis of zinc concentrates—Part 12: Determination of sliver content—The flame atomic absorption spectrometric method
33	GB/T 8151.14-2012	Methods for chemical analysis of zinc concentrates—Part 14: Determination of nickel content—The flame atomic absorption spectrometric method
34	GB/T 8151.16-2005	Methods for chemical analysis of zinc concentrates—Part 16: Determination ofcobalt content—The flame atomic absorption spectrometric method
35	GB/T 8151.19-2012	Methods for chemical analysis of zinc sulfide concentrates—Part 19: Determination of silver and gold contents—Fire assay and flame atomic absorption spectrometric method using scorification or cupellation
36	GB/T 8151.24-2021	Methods for chemical analysis of zinc concentrates—Part 24: Determination of soluble zinc content—Flame atomic absorption spectrometry
37	YS/T 1149.6-2016	Methods for chemical analysis of zinc concentrate roasting—Part 6: Determination of acid-soluble iron content—Flame atomic absorption spectrometry and Na2EDTA titration method
38	GB/T 8152.7-2006	Methods for chemical analysis of lead concentrates—Determination of copper content—Flame atomic absorption spectrometric method
39	GB/T 8152.9-2006	Methods for chemical analysis of lead concentrates—Determination of magnesium oxide content—Flame atomic absorption spectrometricmethod

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40	GB/T 8152.10-2006	Methods for chemical analysis of lead concentrates—Determination of silver and gold content—Fire assay and flame atomic absorption spectrometric method using scorification or cupellation
41	GB/T 8152.12-2006	Methods for chemical analysis of lead concentrates—Determination of cadmium content—Flame atomic absorption spectrometric method
42	GB/T 8152.15-2021	Methods for chemical analysis of lead concentrates—Part 15: Determination of soluble lead content—Flame atomic absorption spectrometry
43	GB/T 8152.16-2022	Methods for chemical analysis of lead concentrates—Part 16: Determination of calcium oxide content—Flame atomic absorption spectrometric method
44	YS/T 461.7-2013	Methods for chemical analysis of lead and zinc bulk concentrates—Part 7: The determination of cadmium content—Flame atomic absorption spectrometry
45	YS/T 461.8-2013	Methods for chemical analysis of lead and zinc bulk concentrates—Part 8: Determination of copper content—Flame atomic absorption spectrometry
46	YS/T 461.9-2013	Methods for chemical analysis of lead and zinc bulk concentrates—Part 9: The determination of silver content—Flame atomic absorption spectrometry
47	GB/T 6150.2-2022	Methods for chemical analysis of tungsten concentrates—Part 2: Determination of tin content—Potassium iodate titrimetry and inductively coupled plasma atomic emission spectrometry
48	GB/T 6150.5-2008	Methods for chemical analysis of tungsten concentrates—Determination of calcium content—EDTA volumetric method and flame atomic absorption spectrometric method
49	GB/T 6150.9-2009	Methods for chemical analysis of tungsten concentrates—Determination of copper content—Flame atomic absorption spectrometry
50	GB/T 6150.10-2008	Methods for chemical analysis of tungsten concentrates—Determination of lead content—Flame atomic absorption spectrometric method
51	GB/T 6150.11-2008	Methods for chemical analysis of tungsten concentrates—Determination of zinc content—Flame atomic absorption spectrometric method
52	GB/T 6150.14-2008	Methods for chemical analysis of tungsten concentrates—Determination of manganese content—The ammonium ferrous sulfate volumetric method and flame atomic absorption spectrometric method

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53	GB/T 6150.15-2008	Methods for chemical analysis of tungsten concentrates—Determination of bismuth content—Flame atomic absorption spectrometric method
54	YS/T 555.6-2009	Methods for chemical analysis of molybdenum concentrate—Determination of copper, lead, bismuth and zinc content—Flame atomic absorption spectrometry
55	YS/T 555.7-2009	Methods for chemical analysis of molybdenum concentrate—Determination of calcium oxide content—Flame atomic absorption spectrometry
56	YS/T 555.9-2009	Methods for chemical analysis of molybdenum concentrate—Determination of potassium and sodium content—Flame atomic absorption spectrometry
57	YS/T 240.2-2007	Methods for chemical analysis of bismuth concentrate—Determination of lead content—Na2EDTA titrimetric method and flame atomic absorption spectrometric method
58	YS/T 240.9-2007	Methods for chemical analysis of bismuth concentrate—Determination of lead content—Iodometric method and the flame atomic absorption spectrometric method
59	YS/T 240.11-2007	Methods for chemical analysis of bismuth concentrate—Determination of silver content—Flame atomic absorption spectrometric method
60	YS/T 556.3-2009	Methods for chemical analysis of antimony concentrates—Part 3: Determination of lead content—Flame atomic absorption spectrometric method
61	YS/T 556.5-2009	Methods for chemical analysis of antimony concentrates—Part 5: Determination of zinc content—Flame atomic absorption spectrometric method
62	YS/T 556.10-2011	Methods for chemical analysis of antimony concentrates Part 10: Determination of copper content—Flame atomic absorption spectrometric method
63	YS/T 556.11-2011	Methods for chemical analysis of antimony concentrates Part 11: Determination of cadmium content—Flame atomic absorption spectrometric method
64	YS/T 556.12-2011	Methods for chemical analysis of antimony concentrates Part 12: Determination of bismuth content—Flame atomic absorption spectrometric method
65	YS/T 556.13-2011	Methods for chemical analysis of antimony concentrates Part 13: Determination of nickel content—Flame atomic absorption spectrometric method
66	YS/T 556.14-2011	Methods for chemical analysis of antimony concentrates Part 14: Determination of silver content—Flame atomic absorption spectrometric method

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67	GB/T 1819.4-2004	Methods for chemical analysis of tin concentrates—Determination of lead content—Flame atomic absorption spectrometric method and EDTA titrimetric method
68	GB/T 1819.6-2004	Methods for chemical analysis of tin concentrates—Determination of antimony content—The malachite green spectrometric method and the flame atomic absorption spectrometric method
69	GB/T 1819.7-2017	Methods for chemical analysis of tin concentrates—Part 7: Determination of bismuth content—Flame atomic absorption spectrometric method
70	GB/T 1819.8-2017	Methods for chemical analysis of tin concentrates—Part 8: Determination of zinc content—Flame atomic absorption spectrometric method
71	GB/T 1819.13-2017	Methods for chemical analysis of tin concentrates—Part 13: Determination of magnesium oxide content, calcium oxide content—Flame atomic absorption spectrometric method
72	GB/T 1819.14-2017	Methods for chemical analysis of tin concentrates—Part 14: Determination of copper content—Flame atomic absorption spectrometry
73	GB/T 1819.16-2017	Methods for chemical analysis of tin concentrats—Part 16: Determination of siliver content—Flame atomic absorption spectrometry
74	GB/T 7739.2-2019	Methods for chemical analysis of gold concentrates—Part 2: Determination of silver content—Flame atomic absorption spectrometric method
75	YS/T 445.2-2019	Methods for chemical analysis of silver concentrates—Part 2: Determination of copper content—Flame atomic absorption spectrometric method
76	YS/T 445.6-2019	Methods for chemical analysis of silver concentrates—Part 6: Determination of magnesium oxide content—Flame atomic absorption spectrometric method
77	YS/T 445.9-2019	Methods for chemical analysis of silver concentrates—Part 9: Determination of lead, zinc and cadmium contents—Flame atomic absorption spectrometric method
78	YS/T 445.10-2019	Methods for chemical analysis of silver concentrates—Part 10: Determination of antimony content—Hydride generation-atomic fluorescence spectrometric method and flame atomic absorption spectrometric method
79	YS/T 445.11-2019	Methods for chemical analysis of silver concentrates—Part 11: Determination of bismuth content—Hydride generation-atomic fluorescence spectrometric method, flame atomic absorption spectrometric method and Na2EDTA titrimetric method

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80	YS/T 1050.7-2015	Methods for chemical analysis of lead antimony concentrates—Part 7: Determination of bismuth and copper contents—Flame atomic absorption spectrometric method
81	YS/T 1050.9-2015	Methods for chemical analysis of lead antimony concentrates—Part 9: Determination of silver content—Flame atomic absorption spectrometric method
82	YS/T 990.2-2014	Methods for chemical analysis of copper matte—Part 2: Determination of gold and silver contents—Atomic absorption spectrmetry and fire assay method
83	YS/T 990.4-2014	Methods for chemical analysis of copper matte—Part 4: Determination of bismuth content—Atomic absorption spectrometry
84	YS/T 990.6-2014	Methods for chemical analysis of copper matte—Part 6: Determination of lead content—Atomic absorption spectrometry and Na2EDTA titration method
85	YS/T 990.7-2014	Methods for chemical analysis of copper matte—Part 7: Determination of cadmium content—Atomic absorption spectrometry
86	YS/T 990.11-2014	Methods for chemical analysis of copper matte—Part 11: Determination of nickel content—Atomic absorption spectrmetry
87	YS/T 990.13-2014	Methods for chemical analysis of copper matte—Part 13: Determination of magnesium oxide content—Atomic absorption spectrmetry
88	YS/T 990.14-2014	Methods for chemical analysis of copper matte—Part 14: Determination of zinc content—Atomic absorption spectrmetry and Na2EDTA titration methods
89	YS/T 990.15-2014	Methods for chemical analysis of copper matte—Part 15: Determination of antimony content—Atomic absorption spectrometry
90	YS/T 990.16-2014	Methods for chemical analysis of copper matte—Part 16: Determination of mercury content—Cold atomic absorption spectrometry
91	YS/T 990.17-2014	Methods for chemical analysis of copper matte—Part 17: Determination of cobalt content—Atomic absorption spectrometry
92	YS/T 716.3-2009	Methods for chemical analysis of low grade blister—Part 3: Determination of bismuth, nickel, lead, antimony and zinc contents—Flame atomic absorption spectrometry
93	YS/T 716.7-2016	Methods for chemical analysis of low grade blister—Part 7: Determination of platinum and palladium contents—Fire assay collection-inductively coupled plasma-atomic emission spectrometric methods and flame atomic absorption spectrometric methods

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94	YS/T 1046.2-2015	Methods for chemical analysis of copper slag concentrates—Part 2: Determination of gold and silver contents—Atomic absorption spectrmetry and fire assay method
95	YS/T 1348.2-2020	Methods for chemical analysis of silver separating residue from lead smelting—Part 2: Determination of lead content—Flame atomic absorption spectrometry and Na2EDTA titration
96	YS/T 1348.3-2020	Methods for chemical analysis of silver separating residue from lead smelting—Part 3: Determination of copper content—Flame atomic absorption spectrometry and Iodine titration method
97	YS/T 1348.4-2020	Methods for chemical analysis of silver separating residue from lead smelting—Part 4: Determination of antimony content—Flame atomic absorption spectrometry and cerium sulfate titrimetric method
98	YS/T 1348.5-2020	Methods for chemical analysis of silver separating residue from lead smelting—Part 5: Determination of bismuth content—Flame atomic absorption spectrometry and Na2EDTA titration
99	YS/T 1512.1-2021	Methods for chemical analysis of copper smelting soot—Part 1: Determination of copper content—Flame atomic absorption spectrometric method and Iodine titration method
100	YS/T 1512.2-2021	Methods for chemical analysis of copper smelting soot—Part 2: Determination of lead content—Flame atomic absorption spectrometric method and Na2EDTA titration
101	YS/T 1512.3-2021	Methods for chemical analysis of copper smelting soot—Part 3: Determination of zinc content—Flame atomic absorption spectrometric method and Na2EDTA titration
102	YS/T 1512.4-2021	Methods for chemical analysis of copper smelting soot—Part 4: Determination of bismuth content—Flame atomic absorption spectrometric method and Na2EDTA titration
103	YS/T 1512.6-2021	Methods for chemical analysis of copper smelting soot—Part 6: Determination of indium content—Flame atomic absorption spectrometric method
104	YS/T 1512.7-2021	Methods for chemical analysis of copper smelting soot—Part 7: Determination of cadmium content—Flame atomic absorption spectrometric method and titration method
105	YS/T 1512.8-2021	Methods for chemical analysis of copper smelting soot—Part 8: Determination of silver and gold content—Flame atomic absorption spectrometric method and fire assay method
106	YS/T 1512.9-2021	Methods for chemical analysis of copper smelting soot—Part 9: Determination of antimony content—Flame atomic absorption spectrometric method

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107	YS/T 745.7-2010	Methods for chemical analysis of copper anode slime—Part 7: Determination of bismuth content—Flame atomic absorption spectrophotometry and Na2EDTA titration method
108	YS/T 745.9-2012	Methods for chemical analysis of copper anode slime—Part 9: Determination of antimony content—Flame atomic absorption spectrophotometry method
109	YS/T 775.2-2011	Methods for chemical analysis of lead anode slime—Part 2: Determination of bismuth content—Flame atomic absorption spectrophotometry and Na2EDTA titrimetric method
110	YS/T 775.4-2011	Methods for chemical analysis of lead anode slime—Part 4: Determination of antimony content—Flame atomic absorption spectrophotometry and cerium sulfate titrimetric method
111	YS/T 1116.3-2016	Methods for chemical analysis of tin anode slime—Part 3: Determination of copper, lead and bismuth contents—Flame atomic absorption spectrometric method
112	YS/T 1116.5-2016	Method for chemical analysis of tin anode slime—Part 5: Determination of indium content—Flame atomic absorption spectrometric method
113	YS/T 521.4-2009	Methods for chemical analysis of blister copper—Determination of lead, bismuth and antimony contents—Flame atomic absorption spectrometry
114	YS/T 521.5-2009	Methods for chemical analysis of blister copper—Part 5: Determination of zinc and nickel contents—Flame atomic absorption spectrometry
115	YS/T 248.3-2007	Methods for chemical analysis of crude lead—Determination of antimony content—Flame atomic absorption spectrometric method
116	YS/T 248.5-2007	Methods for chemical analysis of crude lead—Determination of copper content—Flame atomic absorption spectrometric method
117	YS/T 248.7-2007	Methods for chemical analysis of crude lead—Determination of silver content—Flame atomic absorption spectrometric method
118	YS/T 248.8-2007	Methods for chemical analysis of crude lead—Determination of zinc content—Flame atomic absorption spectrometric method
119	YS/T 248.9-2007	Methods for chemical analysis of crude lead—Determination of bismuthcontent—Flame atomic absorption spectrometric method
120	YS/T 248.10-2007	Methods for chemical analysis of crude lead—Determination of iron content—Flame atomic absorption spectrometric method

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121	YS/T 1341.2-2019	Methods for chemical analysis of crude zinc—Determination of lead content—Flame atomic absorption spectrometric method
122	YS/T 1341.3-2019	Methods for chemical analysis of crude zinc—Determination of iron content—Flame atomic absorption spectrometric method
123	YS/T 1341.4-2019	Methods for chemical analysis of crude zinc—Determination of cadmium content—Flame atomic absorption spectrometric method
124	YS/T 1341.5-2019	Methods for chemical analysis of crude zinc—Determination of copper content—Flame atomic absorption spectrometric method
125	YS/T 1341.10-2019	Methods for chemical analysis of crude zinc—Determination of indium content—Flame atomic absorption spectrometric method
126	YS/T 1462.2-2021	Methods for chemical analysis of crude Tin Part 2: Determination of lead content—Flame atomic absorption spectrometric method and Na2EDTA titrimetric method
127	YS/T 1462.3-2021	Methods for chemical analysis of crude Tin Part 3: Determination of copper content—Flame atomic absorption spectrometric method and Iodometric method
128	YS/T 1462.4-2021	Methods for chemical analysis of crude Tin Part 4: Determination of bismuth content—Flame atomic absorption spectrometric method and Na2EDTA titrimetric method
129	YS/T 1462.5-2021	Methods for chemical analysis of crude Tin Part 5: Determination of antimony content—Flame atomic absorption spectrometric method and cerium sulfate titrimetric method
130	YS/T 955.2-2014	Methods for chemical analysis of crude silver—Part 2: Determination of palladium content—Flame atomic absorption spectrometric method
131	YS/T 1157.3-2016	Methods for chemical analysis of crude cobalt hydroxide—Part 3: Determination of calcium and magnesium contents—Flame atomic absorption spectrometry and inductively coupled plasma atomic emission spectrometry
132	YS/T 1229.2-2018	Methods for chemical analysis of crude nickel hydroxide—Part 2: Determination of cobalt content—Flame atomic absorption spectrometric method
133	YS/T 1345.5-2020	Methods for chemical analysis of bismuth-rich lead—Part 5: Determination of copper content—Flame atomic absorption spectrometric method
134	YS/T 746.2-2010	Methods for chemical analysis of tin-based lead-free solders—Part 2: Determination of silver content—Flame atomic absorption spectrometric method and potassium thiosulfate titrimetric method

Continued

135	GB/T 11064.4-2013	Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride—Part 4: Determination of potassium and sodium content—Flame atomic absorption spectrometric method
136	GB/T 11064.6-2013	Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride—Part 6: Determination of magnesium content—Flame atomic absorption spectrometric method
137	GB/T 20931.1-2007	Methods for chemical analysis of lithium—Determination of potassium content—Flame atomic absorption spectrometric method
142	GB/T 12689.7-2010	Methods for chemical analysis of zinc and zinc alloys—Part 7: Determination of magnesium content—Flame atomic absorption spectrometric method
143	GB/T 4103.14-2009	Methods for chemical analysis of lead and lead alloys—Part 14: Determination of cadmium content—Flame atomic absorption spectrophotometry
144	GB/T 4324.1-2012	Methods for chemical analysis of tungsten—Part 1: Determination of lead content—Flame atomic absorption spectrometry
145	GB/T 4324.2-2012	Methods for chemical analysis of tungsten—Part 2: Determination of bismuth content—Flame atomic absorption spectrometry
146	GB/T 4324.3-2012	Methods for chemical analysis of tungsten—Part 3: Determination of tin content—Flame atomic absorption spectrometry
147	GB/T 4324.4-2012	Methods for chemical analysis of tungsten—Part 4: Determination of antimony content—Flame atomic absorption spectrometry
155	GB/T 4325.1-2013	Methods for chemical analysis of molybdenum—Part 1: Determination of lead content—Graphite furnace atomic absorption spectrometry
156	GB/T 4325.2-2013	Methods for chemical analysis of molybdenum—Part 2: Determination of cadmium content—Flame atomic absorption spectrometry
157	GB/T 4325.8-2013	Methods for chemical analysis of molybdenum—Part 8: Determination of cobalt content—5-Cl-PADAB spectrophotometry and flame atomic absorption spectrometry
158	GB/T 4325.9-2013	Methods for chemical analysis of molybdenum—Part 9: Determination of nickel content—Dimethylglyoxime spectrophotometry and flame atomic absorption spectrometry

Continued

159	GB/T 4325.10-2013	Methods for chemical analysis of molybdenum—Part 10: Determination of copper content —Flame atomic absorption spectrometry
160	GB/T 4325.13-2013	Methods for chemical analysis of molybdenum—Part 13: Determination of calcium content—Flame atomic absorption spectrometry
161	GB/T 4325.14-2013	Methods for chemical analysis of molybdenum—Part 14: Determination of magnesium content—Flame atomic absorption spectrometry
162	GB/T 4325.15-2013	Methods for chemical analysis of molybdenum—Part 15: Determination of sodium content—Flame atomic absorption spectrometry
163	GB/T 4325.16-2013	Methods for chemical analysis of molybdenum—Part 16: Determination of potassium content—Flame atomic absorption spectrometry
168	GB/T 10574.7-2017	Methods for chemical analysis of tin-lead solders—Part 7: Determination of silver content—Flame atomic absorption spectrometry and potassium thiocyanate potentiometric titra
169	GB/T 10574.8-2017	Methods for chemical analysis of tin-lead solders—Part 8: Determination of zinc content—Flame atomic absorption spectrometric method
170	GB/T 10574.9-2017	Methods for chemical analysis of tin-lead solders—Part 9: Determination of aluminium content—Graphite furnace atomic absorption spectrometric method
171	GB/T 10574.10-2017	Methods for chemical analysis of tin-lead solders—Part 10: Determination of cadmium content—Flame atomic absorption spectrometry and Na2EDTA titration method
172	GB/T 15249.5-2009	Methods for chemical analysis of crude gold—Part 5: Determination of mercury content—Cold atomic absorption spectrometry
173	GB/T 11066.2-2008	Methods for chemical analysis of gold—Determination of silver content—Flame atomic absorption spectrometry
174	GB/T 11066.3-2008	Methods for chemical analysis of gold—Determination of iron content—Flame atomic absorption spectrometry
175	GB/T 11066.4-2008	Methods for chemical analysis of gold—Determination of copper, lead and bismuth contents—Flame atomic absorption spectrometry
176	GB/T 11066.6-2009	Methods for chemical analysis of gold—Determination of magnesium, nickel, manganese, palladium contents—Flame atomic absorption spectrometry
177	GB/T 11067.1-2006	Methods for chemical analysis of silver—Determination of silver content—Silver chloride precipitation-flame atomic absorption spectrometric method

The simultaneous analysis technology of multiple elements had gradually been adopted, along with the development of continuous light source for atomic absorption spectroscopy, high-resolution optical system and other technologies.

3.2. Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

That was the analysis principle of inductively coupled plasma atomic emission spectrometry which the sample is brought into the plasma torch flame by a carrier gas in a certain form, and is fully evaporated, atomized, excited and ionized in a high temperature and inert atmosphere, emitting characteristic spectral lines of the contained elements. According to the presence of characteristic spectral lines, qualitative analysis was carried out to identify whether the sample contains a certain element, and quantitative analysis which determines the content of the corresponding element in the sample based on the characteristic spectral line intensity.

ICP-AES was suitable for qualitative and quantitative analysis of constant to trace elements in samples, and it had been widely used. ICP-AES in some non-ferrous metal analysis standards was given in **Table 2**.

The amounts of lead zinc bismuth cadmium chromium arsenic and mercury in gold ore were determined by ICP-AES [13], and the amounts of lead zinc bismuth cadmium arsenic and mercury were determined by hydrochloric acid and nitric acid dissolution. The amount of chromium was determined by dissolving it with thion mixed acid. In dilute nitric acid medium, under the selected conditions of inductively coupled plasma atomic emission spectrometer, the spectral intensity of each element in the test solution is determined, and the amount of lead zinc bismuth cadmium chromium arsenic and mercury in the sample were calculated according to the standard curve method using yttrium as the internal standard. The lower limit of method element determination can be as low as 0.005% and as high as 5.00%. The amount of silver copper iron lead antimony bismuth palladium magnesium nickel manganese and chromium in 99.95% - 99.99% gold ingots was determined by ICP-AES [14], and ethyl acetate was extracted and separated from the element to be measured in the aqueous phase after mixing acid to dissolve the sample. The lower limit of this method for elemental determination can be as low as 0.0001%.

3.3. X-Ray Fluorescence Spectroscopy (XRF)

X-ray fluorescence spectroscopy was a qualitative and quantitative analysis method based on the wavelength and intensity of X-ray fluorescence of the elemental characteristics produced after X-ray irradiation of a sample. XRF was an analytical technique for simultaneous determination of primary, secondary and trace multiple elements. Analysis principle: X-ray was a kind of electromagnetic radiation with a short wavelength, and the energy range was between 0.1 - 100 keV. If the sample was irradiated with sub-X-rays emitted by an X-ray tube, the

Table 2. ICP-AES in some non-ferrous metal analysis standards.

No.	Standard No.	Standard Name
1	GB/T 14352.19-2021	Methods for chemical analysis of tungsten ores and molybdenum ores—Part 19: Determination of bismuth, cadmium, cobalt, copper, iron, lithium, nickel, phosphorus, lead, strontium, vanadium and zinc content—Inductive-coupled plasma atomic emission spectrophotometry (ICP-AES)
2	GB/T 20899.13-2017	Methods for chemical analysis of gold ores—Part 13: Determination of lead, zinc, bismuth, cadmium, chromium, arsenic and mercury contents—Inductively coupled plasma atomic emission spectrometry
3	GB/T 20899.14-2017	Methods for chemical analysis of gold ores—Part 14: Determination of thallium content—Inductively coupled plasma atomic emission spectrometry and inductively coupled plasma mass spectrometry
4	SN/T 2763.4-2012	Chemical analysis of nickel laterite ore—Part 4: Determination of nickel, cobalt, aluminum, magnesium and phosphorus content—Inductively coupled plasma atomic emission spectrometric method
5	SN/T 2763.6-2014	Chemical analysis of nickel laterite ore—Part 6: Determination of nickel, calcium, titanium, magnesium, copper, cobalt, chromium, zinc and phosphorus content—Inductively coupled plasma atomic emission spectrometric method
6	YS/T 1115.12-2016	Methods for chemical analysis of copper ores and tailings—Part 12: Determination of copper, lead, zinc, nickel, cobalt, cadmium, magnesium and manganese contents—Inductively coupled plasma atomic emission spectrometric method
7	SN/T 5580-2023	Determination of gold content in copper concentrate Foam-based granular activated carbon enrichment and separation-inductively coupled plasma emission spectrometry
8	GB/T 3884.18-2014	Methods for chemical analysis of copper concentrates—Part 18: Determination of arsenic, antimony, bismuth, lead, zinc, nickel, cadmium, cobalt, magnesium oxide, calcium oxide contents—Inductively coupled plasma atomic emission spectrometry
9	YS/T 1046.6-2015	Methods for chemical analysis of copper slag concentrates—Part 6: Determination of aluminum oxide content—Inductively coupled plasma atomic emission spectrometry
10	YS/T 1046.7-2015	Methods for chemical analysis of copper slag concentrates—Part 7: Determination of arsenic, antimony, bismuth, lead , zinc and magnesium oxide content—Inductively coupled plasma atomic emission spectrometry

Continued

11	YS/T 1047.7-2015	Methods for chemical analysis of copper magnetite—Part 7: Determination of copper, manganese, aluminium, calcium, magnestium, titanium and phosphorus contents—Inductively coupled plasma atomic emission spectrometric method
12	YS/T 1047.12-2020	Methods for chemical analysis of copper magnetite—Part 12: Determination of sulfur contents—Inductively coupled plasma atomic emission spectrometric method
13	GB/T 6150.2-2022	Methods for chemical analysis of tungsten concentrates—Part 2: Determination of tin content—Potassium iodate titrimetry and inductively coupled plasma atomic emission spectrometry
14	GB/T 7739.13-2019	Methods for chemical analysis of gold concentrates—Part 13: Determination of lead, zinc, bismuth, cadmium, chromium, arsenic and mercury contents—Inductively coupled plasma atomic emission spectrometry
15	GB/T 7739.14-2019	Methods for chemical analysis of gold concentrates—Part 14: Determination of thallium content—Inductively coupled plasma atomic emission spectrometry and inductively coupled plasma mass spectrometry
16	GB/T 8151.20-2012	Methods for chemical analysis of zinc concentrates—Part 20: Determination of copper, lead, iron, arsenic, cadmium antimony, calcium, magnesium contents—Inductively coupled plasma atomic emission spectrometry
17	YS/T 1050.10-2016	Methods for chemical analysis of lead-antimony concentrate—Part 10: Determanation of thallium—Inductively coupled plasma mass spectrometry and inductively coupled plasma atomic emission spectrometry
18	GB/T 18114.4-2010	Chemical analysis methods of rare earth concentrates—Part 4: Determination of niobium oxide, zirconium oxide and titanium oxide contents—Inductively coupled plasma atomic emission spectrometry
19	GB/T 18114.5-2010	Chemical analysis methods of rare earth concentrates—Part 5: Determination of aluminum oxide content—Inductively coupled plasma atomic emission spectrometry
20	SN/T 5054-2018	Determination of zinc, cadmium, lead, manganese, iron, magnesium, calcium and copper content in antimony concentrate by inductively coupled plasma emission spectrometry
21	YS/T 1085-2015	Refined nickel—Determination of Silicon, manganese, phosphorus, iron, copper, cobalt, magnesium, aluminum, zinc, chromium—Inductively coupled plasma atomic emission spectrometric method

Continued

22	YS/T 358.5-2011	Methods for chemical analysis of tantalite, columbite concentrate—Part 5: Determination of uranium content—Inductively coupled plasma atomic emission spectrometry
23	YS/T 358.6-2011	Methods for chemical analysis of tantalite, columbite concentrate—Part 6: Determination of thorium oxide content—Inductively coupled plasma atomic emission spectrometry
24	YS/T 358.7-2011	Methods for chemical analysis of tantalite, columbite concentrate—Part 7: Determination of iron content—Inductively coupled plasma atomic emission spectrometry
25	YS/T 358.9-2011	Methods for chemical analysis of tantalite, colubite concentrate—Part 9: Determination of antimony content—Inductively coupled plasma atomic emission spectrometry
26	YS/T 1171.11-2019	Methods for chemical analysis of regenerated zinc material—Part 11: Determination of germanium content—Inductively coupled plasma atomic emission spectrometry
27	YS/T 1171.13-2021	Methods for chemical analysis of regenerated zinc material—Part 13: Determination of thallium content by inductively coupled plasma mass spectrometry and inductively coupled plasma atomic emission spectrometry
28	YS/T 1171.3-2017	Methods for chemical analysis of recycled zinc material—Part 3: Determination of copper, lead, iron, indium, cadmium, arsenic, calcium and aluminum Inductively coupled plasma atomic emission spectrometry
29	YS/T 1314.2-2019	Methods for chemical analysis of silver separating residue from copper smelting—Part 2: Determination of platinum and palladium contents—Fire assay collection-inductively coupled plasma atomic emission spectrometry
30	YS/T 1314.5-2019	Methods for chemical analysis of silver separating residue from copper smelting—Part 5: Determination of copper, antimony, bismuth, selenium, telluriumand and tin contents—Inductively coupled plasma atomic emission spectrometry
31	YS/T 1348.6-2020	Methods for chemical analysis of silver separating residue from lead smeltering—Part 6: Determination of lead, copper, antimony and bismuth contents—Inductively coupled plasma atomic emission spectrometry

Continued

32	YS/T 1344.1-2020	Methods for chemical analysis of indium oxide powder doped with tin—Part 1: Determination of iron, aluminum, lead, nickel, copper, cadmium, chromium and thallium content Inductively coupled plasma atomic emission spectrometry
33	GB/T 12690.13-2003	Chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides—Determination of molybdenum and tungsten content—Inductively coupled plasma atomic emission spectrographi
34	GB/T 16477.2-2010	Chemical analysis methods of rare earth ferrosilicon alloy and rare earth ferrosilicon magnesium alloy—Part 2: Determination of calcium, magnesium and manganese contents—Inductively coupled plasma atomic emission spectrometry
35	GB/T 16477.3-2010	Chemical analysis methods of rare earth ferrosilicon alloy and rare earth ferrosilicon magnesium alloy—Part 3: Determination of magnesia content—Inductively coupled plasma atomic emission spectrometry
36	GB/T 16477.5-2010	Chemical analysis methods of rare earth ferrosilicon alloy and rare earth ferrosilicon magnesium alloy—Part 5: Determination of titanium content—Inductively coupled plasma atomic emission spectrometry
37	GB/T 16484.3-2009	Chemical analysis methods of rare earth chloride and light rare earth carbonate—Part 3: Determination of fifteen REO relative contents—Inductively coupled plasma atomic emission spectrometry
38	GB/T 16484.5-2009	Chemical analysis methods of rare earth chloride and light rare earth carbonate—Part 5: Determination of barium oxide content—Inductively coupled plasma atomic emission spectrometry
39	GB/T 23273.8-2009	Methods for chemical analysis of cobalt oxalate—Part 8: Determination of nicke, copper, iron, zinc, aluminium, manganese lead, arsenic, calcium, magnesium and sodium content—Inductively coupled plasma atomic emission spectrometry
40	GB/T 26416.2-2022	Chemical analysis method for rare earth ferroalloy—Part 2: Determination of rare earth impurity contents—Inductively coupled plasma emission spectrometry
41	GB/T 26416.3-2022	Chemical analysis methods for rare earth ferroalloy—Part 3: Determination of calcium, magnesium, aluminium, nickel and manganese contents—Inductively coupled plasma emission spectrometry

Continued

42	GB/T 4325.7-2013	Methods for chemical analysis of molybdenum—Part 7: Determination of iron content-1, 10-phenanthroline spectrophotometry and inductively coupled plasma atomic emission spectrometry
43	YS/T 1179.4-2017	Chemical analysis methods of aluminum slag—Part 4: Determination method of silicon, magnesium, calcium—Inductively coupled plasma spectroscopy
44	YS/T 281.19-2011	Methods for chemical analysis of cobalt—Part 19: Determination of calcium magnesium manganese iron cadmium zinc content—Inductively coupled plasma atomic emission spectrometery
45	YS/T 956.1-2014	Chemical analysis method of gold and germanium alloys—Part 1: Determination of germanium content—Inductively coupled plasma atomic emission spectrometry
46	YS/T 509.1-2008	Methods for chemical analysis of cobaltosic oxide—Determination of magnetic impurities content—Magnetic separation—Inductively coupled plasma atomic emission spectrometry
47	YS/T 3015.4-2013	Methods for chemical analysis of gold-loaded carbon—Part 4: Determination of copper, iron, calcium and magnesium contents—Inductively coupled plasma-atomic emission spectrometry
48	YS/T 710.6-2009	Method for chemical analysis of cobalt oxide—Part 6: Determination of calcium, cadmium, copper, iron, magnesium, manganese, nickel, lead, zinc content—Inductively coupled plasma-atomic emission spectromertry
49	YS/T 745.3-2010	Methods for chemical analysis of copper anode slime—Part 3: Determination of platinum content and palladium content—Fire assay collection-inductively coupled plasma atomic emission spectrometric methods
50	YS/T 956.1-2014	Chemical analysis method of gold and germanium alloys—Part 1: Determination of germanium content—Inductively coupled plasma atomic emission spectrometry
51	GB/T 11064.16-2013	Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride—Part 16: Determination of calcium, magnesium, copper, lead, zinc, nickel, manganese, cadmium and aluminum content—Inductively coupled plasma atomic emission spectrometry
52	GB/T 10574.13-2017	Methods for chemical analysis of tin-lead solders—Part 13: Determination of antimony, bismuth, iron, arsenic, copper, silver, zinc, aluminium, cadmium, phosphorous and gold contents—Inductively coupled plasma atomic emission spectrometric method

Continued

53	GB/T 5121.27-2008	Methods for chemical analysis of copper and copper alloys—Part 27: The inductively coupled plasma atomic emission spectromertric method
54	YS/T 1014.2-2014	Methods for chemical analysis of bismuth trioxide—Part 2: Determination of silver, copper, magnesium, nickel, cobalt, manganese, calcium, iron, cadmium, lead, zinc, antimony, aluminium, sodium and sulfur contents—Inductively coupled plasma atomic emission spectrographic method
55	GB/T 39143-2020	Methods for chemical analysis of gold-arsenic alloys—Determination of arsenic content—Inductively coupled plasma atomic emission spectrometry
56	GB/T 41769-2022	Methods for chemical analysis of cadmium zinc telluride—Determination of zinc and cadmium contents—Inductively coupled plasma atomic emission spectrometry
57	GB/T 11066.8-2009	Method for chemical analysis of gold—Determination of silver copper iron lead antimony bismuth palladium magnesium nickel manganese and chromium content—Ethyl acetate extration-inductively coupled plasma—Atomic emission spectrometry
58	GB/T 11067.3-2006	Methods for chemical analysis of silver—Determination of selenium and tellurium content—Inductively coupled plasma atomic emission spectrometric method
59	GB/T 11067.4-2006	Methods for chemical analysis of silver—Determination of antimony content—The inductively coupled plasma atomic emission spectrometric method
60	GB/T 42273-2022	Methods for chemical analysis of zirconium compounds—Determination of calcium, hafnium, titanium, sodium, iron, chromium, cadmium, zinc, manganese, copper, nickel, lead contents—Inductively coupled plasma atomic emission spectrometry
61	GB/T 4324.11-2012	Methods for chemical analysis of tungsten—Part 11: Determination of aluminum content—Inductively coupled plasma atomic emission spectrometry
62	GB/T 4324.13-2008	Methods for chemical analysis of tungsten—Determination of calcium content—The inductively coupled plasma atomic emission spectrometry

inner electrons of the elements in the sample are excited by it, producing characteristic X-ray fluorescence, or secondary X-rays. By measuring and analyzing the X-ray fluorescence generated by the sample, the elements in the sample could be qualitatively and quantitatively analyzed, and the application in the major non-ferrous metal analysis standards was given in **Table 3**.

Compared with energy dispersive X-ray fluorescence spectroscopy (ED-XRF), wavelength dispersive X-ray fluorescence spectroscopy (WD-XRF) was an analytical instrument that entered the analytical laboratory earlier. WD-XRF is an earlier technique that emerged as a standard method.

The amount of copper sulfur lead zinc iron aluminum calcium magnesium and manganese in copper concentrate was determined by glass fusion-wavelength chromatographic X-ray fluorescence spectrometry [15], and under the combined action of sodium carbonate and lithium nitrate, low-valence elements were oxidized to high-valence elements. According to the dilution ratio of about 1:83, the copper concentrate sample and lithium tetraborate-lithium metaborate mixed flux were melted into the test sheet, and the X-ray fluorescence intensity of the characteristic spectral line of the element to be measured in the test piece was measured under the optimal measurement conditions of the instrument, and the inter-element interference effect was corrected, and the content of the element to be measured in the test piece was obtained from the calibration curve.

The amount of tin antimony arsenic bismuth copper cadmium calcium and silver in lead and lead alloys was determined by WD-XRF [16], and the surface to be measured of metal samples was directly processed to uniform, flat and smooth, and there were no defects such as cracks, cavities and looseness, and measured in time. No less than 4 pieces of lead and lead alloy standard materials were prepared to determine the X-ray fluorescence intensity of the characteristic spectral lines of each element, and one of the basic parameter method, theoretical α coefficient method, empirical α coefficient method and other methods was selected for matrix effect correction and spectral line interference correction, and the standard curve was drawn. Based on the calibration curve and the measured X-ray fluorescence intensity, the mass fraction of tin, antimony, arsenic, bismuth copper cadmium calcium and silver content in the sample was calculated.

The amount of aluminum chromium iron magnesium manganese nickel and silicon in laterite nickel ore was determined by ED-XRF [17], the powder sample was pressed or prepared into a glass melt, placed in the beam emitted by the X-ray source, the fluorescence X-ray intensity generated when the sample was excited was determined by the analytical device, and the content of the element to be measured was calculated by the calibration curve.

3.4. Atomic Fluorescence Spectroscopy (AFS)

Atomic fluorescence is an atomic spectroscopic method that uses the fluorescence intensity emitted by atoms during radiation excitation for elemental quantification. AFS can be used to measure the non-dispersive vapor generation-atomic fluorescence spectra of the elements arsenic, antimony, bismuth, selenium, tellurium, lead, tin, germanium, atomic vapor mercury, and volatile compounds zinc, cadmium and other elements that can form hydrides. In 1964, Winefordner and Vickers [18] published the first chemical analysis paper on the

Table 3. XRF in major non-ferrous metal analysis standards.

No.	Standard No.	Standard Name
1	YS/T 1047.6-2015	Methods for chemical analysis of copper magnetite—Part 6: Determination of copper, total iorn, silicon dioxide, alumina oxide, calcium oxide, magnesium oxide, titanium dioxide, manganese oxide and phosphorus contents—Wavelength dispersive X-ray fluorescence spectrometric method
2	YS/T 820.19-2012	Methods for chemical analysis of laterite nickel ores—Part 19: Determination of aluminum, chromium, iron, magnesium, manganese, nickel and silicon contents—Energy-dispersive X-ray fluorescence spectrometry
3	YS/T 820.23-2012	Methods for chemical analysis of laterite nickel ores—Part 23: of cobalt, iron, nickel, phosphorus, aluminium oxide, calcium oxide, chromium oxide, magnesium oxide, manganese oxide, silicon dioxide and titanium dioxide content—Wavelength dispersive X-ray fluorescence spectrometry
4	SN/T 2763.1-2011	Determination of Multiple Components in Laterite Nickel Ore—Part 1: X-ray fluorescence spectroscopy
5	YS/T 575.23-2021	Methods for chemical analysis of bauxite—Part 23: Determination of element contents—X-ray fluorescence spectrometry method
6	GB/T 3884.21-2018	Methods for chemical analysis of copper concentrates—Part 21: Determination of copper, sulfur, lead, zinc, iron, aluminium, calcium, magnesium, manganese content—Wavelength dispersive X-ray fluorescence spectrometric method
7	SN/T 4365-2015	Determination of elemental content of copper, lead, chromium, arsenic, silver, antimony, bismuth, nickel, iron, aluminum in copper concentrate Wavelength dispersive X-ray fluorescence spectrometry
8	GB/T 8151.22-2020	Methods for chemical analysis of zinc concentrates—Part 22: Determination of zinc, copper, lead, iron, aluminium, calcium and magnesium contents—Wavelength dispersive X-ray fluorescence spectrometric method
9	SN/T 3604-2013	Determination of copper, silicon, magnesium, zinc, aluminum, iron content in zinc concentrate by X-ray fluorescence spectroscopy
10	SN/T 3012-2011	Determination of tungsten trioxide content in tungsten concentrate by X-ray fluorescence spectroscopy
11	SN/T 3916-2014	Determination of the content of molybdenum, iron, lead, copper, silicon and calcium in molybdenum concentrate Wavelength dispersive X-ray fluorescence spectrometry

Continued

12	SN/T 5412-2022	Determination of cobalt, copper and manganese content in cobalt concentrate by wavelength dispersive X-ray fluorescence spectrometry
13	SN/T 2780-2011	Determination of lead, cadmium and chromium in alumina by wavelength dispersive X-ray fluorescence spectrometry
14	GB/T 6609.30-2022	Chemical analysis methods and determination of physical performance of alumina—Part 30: Determination of trace elements—Wavelength dispersive X-ray fluorescence spectrometric method
15	SN/T 0829-1999	Determination of magnesium oxide, silicon oxide, calcium oxide, iron oxide, alumina, manganese oxide, titanium oxide in magnesia X-ray fluorescence spectrometry
16	SN/T 1793-2006	Determination of copper, lead, iron, bismuth, antimony, phosphorus, arsenic in brass Wavelength dispersive X-ray fluorescence spectrometry
17	YS/T 1340-2019	Methods for chemical analysis of lead and lead alloys—Determination of tin, antimony, arsenic, bismuth, copper, cadmium, calcium, silver contents—Wavelength dispersive X-ray fluorescence spectrometry
18	YS/T 273.14-2008	Chemical analysis methods and determination of physical performance of synthetic cryolite—Part 14: X-ray fluorescence spectrometric method for the determination of elements content
19	YS/T 273.15-2012	Chemical analysis methods and physical properties of cryolite—Part 15: X-ray fluorescence spectrometric method for the determination of elements content using pressed powder tablets
20	YS/T 581.10-2006	Determination of chemical contents and physical properties of aluminium fluoride Part 10: Determination of sulphur content by X-ray fluorescence spectrometric method
21	YS/T 581.16-2008	Chemical analysis methods and determination of physical performance of industrial aluminium fluoride Part 16: X-ray fluorescence spectrometric method for the determination of elements content
22	YS/T 581.18-2012	Chemical analysis methods and physical properties of Aluminum Fluoride—Part 18: X-ray fluorescence spectrometric method for the determination of elements content using pressed powder tablets
23	YS/T 483-2022	Methods for analysis of copper and copper alloys-X-Ray fluorescence spectrometric (wavelength dispersive)
24	YS/T 806-2020	Methods for chemical analysis of aluminum and aluminum alloys Determination of elemental content X-ray fluorescence spectroscopy

determination of mercury, zinc and cadmium by flame atomic fluorescence spectrometry, after which the technology has made great achievements in applied research and standardization in China, Feng Xianjin and Zhang Lianxiang [19] summarized the research of AFS technology in China's standardization, among which the application in the main non-ferrous metal analysis standards is given in **Table 4**.

Atomic fluorescence instruments and analysis technology were at the international leading level In China. The amount of arsenic in tungsten ore and molybdenum ore was determined by hydride generation-atomic fluorescence spectrometry [20], and the sample was decomposed by hydrochloric acid-nitric acid to prepare a sample test solution. Thiourea-ascorbic acid solution reduced pentavalent arsenic in the test solution to trivalent arsenic, and reacted with potassium borohydride to generate hydrogen arsenide, which was loaded into the atomizer by argon, and under the irradiation of arsenic high-intensity hollow cathode lamp, the ground state arsenic atoms were excited to a high-energy state, and when it returned to the ground state, it emits fluorescence of characteristic wavelengths, and its fluorescence intensity was proportional to the content of arsenic in the sample, and the calibration curve was used to quantitatively determine the amount of arsenic in the solution. In the method for determination of mercury in tin concentrate using cold steam generation-atomic fluorescence spectrometry, the sample material was dissolved with hydrochloric acid and nitric acid, and the ionic mercury in the dilute hydrochloric acid medium was reduced to atomic mercury by stannous chloride, and the argon gas was introduced into the quartz furnace atomizer, and the fluorescence intensity of mercury was measured on the atomic fluorescence spectrometer.

The amount of arsenic mercury cadmium lead and bismuth in gold ore were determined by AFS [21], and the samples were prepared by water bath and tetraic acid two methods as arsenic-mercury, bismuth and arsenic-bismuth lead-cadmium to be tested, which can realize the simultaneous determination of arsenic and bismuth elements, and the three elements of mercury, cadmium and lead were determined separately. The AFS of arsenic mercury cadmium lead and bismuth in gold concentrates could also be determined simultaneously for multiple elements [22]. The laboratory was equipped with a three-channel fluorescence instrument, which can be sufficient for these analytical methods.

3.5. Direct Reading Spectroscopy (OES)

Direct reading spectrometer was an emission spectrometer, which was mainly an instrument for quantitative analysis of samples by measuring the intensity of characteristic spectral light representing each element when the sample was excited.

The non-ferrous metal standard developed by direct reading spectroscopic analysis method was YS/T 464-2019 "Method for analysis of copper cathode—The optical emission spectrometry", which used direct reading spectrometry to

Table 4. AFS in some non-ferrous metal analysis standards.

No.	Standard No.	Standard Name
1	YS/T 1047.6-2015	Methods for chemical analysis of copper magnetite—Part 6: Determination of copper, total iorn, silicon dioxide, alumina oxide, calcium oxide, magnesium oxide, titanium dioxide, manganese oxide and phosphorus contents—Wavelength dispersive X-ray fluorescence spectrometric method
2	YS/T 820.19-2012	Methods for chemical analysis of laterite nickel ores—Part 19: Determination of aluminum, chromium, iron, magnesium, manganese, nickel and silicon contents—Energy-dispersive X-ray fluorescence spectrometry
3	YS/T 820.23-2012	Methods for chemical analysis of laterite nickel ores—Part 23: of cobalt, iron, nickel, phosphorus, aluminium oxide, calcium oxide, chromium oxide, magnesium oxide, manganese oxide, silicon dioxide and titanium dioxide content—Wavelength dispersive X-ray fluorescence spectrometry
4	SN/T 2763.1-2011	Determination of Multiple Components in Laterite Nickel Ore—Part 1: X-ray fluorescence spectroscopy
5	YS/T 575.23-2021	Methods for chemical analysis of bauxite—Part 23: Determination of element contents—X-ray fluorescence spectrometry method
6	GB/T 3884.21-2018	Methods for chemical analysis of copper concentrates—Part 21: Determination of copper, sulfur, lead, zinc, iron, aluminium, calcium, magnesium, manganese content—Wavelength dispersive X-ray fluorescence spectrometric method
7	SN/T 4365-2015	Determination of elemental content of copper, lead, chromium, arsenic, silver, antimony, bismuth, nickel, iron, aluminum in copper concentrate Wavelength dispersive X-ray fluorescence spectrometry
8	GB/T 8151.22-2020	Methods for chemical analysis of zinc concentrates—Part 22: Determination of zinc, copper, lead, iron, aluminium, calcium and magnesium contents—Wavelength dispersive X-ray fluorescence spectrometric method
9	SN/T 3604-2013	Determination of copper, silicon, magnesium, zinc, aluminum, iron content in zinc concentrate by X-ray fluorescence spectroscopy
10	SN/T 3012-2011	Determination of tungsten trioxide content in tungsten concentrate by X-ray fluorescence spectroscopy
11	SN/T 3916-2014	Determination of the content of molybdenum, iron, lead, copper, silicon and calcium in molybdenum concentrate Wavelength dispersive X-ray fluorescence spectrometry

Continued

12	SN/T 5412-2022	Determination of cobalt, copper and manganese content in cobalt concentrate by wavelength dispersive X-ray fluorescence spectrometry
13	SN/T 2780-2011	Determination of lead, cadmium and chromium in alumina by wavelength dispersive X-ray fluorescence spectrometry
14	GB/T 6609.30-2022	Chemical analysis methods and determination of physical performance of alumina—Part 30: Determination of trace elements—Wavelength dispersive X-ray fluorescence spectrometric method
15	SN/T 0829-1999	Determination of magnesium oxide, silicon oxide, calcium oxide, iron oxide, alumina, manganese oxide, titanium oxide in magnesia X-ray fluorescence spectrometry

determine 18 elements such as arsenic antimony bismuth sulfur selenium tellurium iron silver tin nickel lead zinc chromium tin cobalt silicon phosphorus and manganese in cathode copper. The sample preparation was prepared according to the 5.3 copper cathode sampling and sample preparation method in GB/T467-2010, and the copper cathode international standard sample was used as the monitoring sample. A set of standard samples were used for accuracy and precision tests. The smooth plane of the specimen was the upper electrode and the tungsten needle is the lower electrode when measuring, and the specimen was excited with a high-performance spark light source, and the excitation position was changed, and at least four excitation determinations were carried out on the same surface. It needed to take at least four measured values according to the working curve of the instrument and each calibration factor, and the instrument automatically processed the detected data, calculated and outputted the analysis results of arsenic antimony bismuth sulfur selenium tellurium iron silver tin nickel lead zinc chromium cadmium cobalt silicon phosphorus and manganese.

3.6. Glow Discharge Emission Spectroscopy (GD-OES)

Glow discharge emission spectroscopy was a technique mainly used to study the elemental composition of materials. It was mainly used in the field of materials manufacturing to determine whether there was any oxidation, surface treatment or contaminants in or on the sample.

Glow discharge was plasma formed when an electric current passes through a gas. It was generated when a voltage was applied between the cathode and anode in a glass tube containing a low-pressure gas such as helium. This ionized the gas, causing the lamp to glow brightly, which could be kept bright when the applied voltage exceeds the trigger voltage. The color of the light produced depends on the type of gas used in the tube. The excited atoms and ions in the discharge plasma produce different emission spectra for each element, and a single element could produce several different emission spectral lines that made up the light produced by the discharge.

Glow discharge spectrometer consisted of discharge lamp, spectrometer and data detection and analysis system. Spectrometers were used to analyze the emission spectra of gases, while data detection and analysis systems enabled qualitative and quantitative analysis of interactions in gases. Magnetron discharge and radio frequency discharge were the two most common types of glow discharge plasma generators. If a depth distribution of elemental composition up to 150 μm of a sample was required, glow discharge light emission spectroscopy should be used. This was particularly ideal for metals and insulators, which provided fast elemental analysis.

GB/T 32996-2016 "Surface chemical analysis of metal oxide film by glow discharge emission spectrometry" specified the method for determining the thickness, unit area mass and chemical composition of metal oxide film by glow discharge emission spectroscopy. This method was suitable for the determination of oxide film with a thickness of 1 nm - 10,000 nm on metal, and the metal elements of the oxide included one or more of iron chromium nickel copper titanium silicon molybdenum zinc magnesium manganese and aluminum. Other measurable elements included oxygen carbon nitrogen hydrogen, phosphorus and sulfur.

3.7. Laser-Induced Breakdown Spectroscopy (LIBS)

Laser-induced breakdown spectroscopy was suitable for the detection and analysis of chemical elements of solid liquid and gaseous substances. The LIBS Basic principle was which the laser emits the laser, and the laser focusing system concentrates the laser to ablate the substance to be analyzed to produce plasma. Atoms molecules or electrons in ions in plasma were excited to an excited state and emit characteristic photons when they transitioned from an upper energy level to a lower energy level. After the plasma radiation collection system collected the characteristic photon signal, it was dispersed by the spectrometer, and the data processing system performs qualitative analysis according to the characteristic spectral lines of the elements, and quantitatively analyzed according to the characteristic spectral line intensity of the elements or the overall spectral information. GB/T 38257-2019 [23] and T/CNIA 0109-2021 provided specifications for draft of standard methods for this technology in the future, and the research work on related analysis methods had been carried out.

4. Summary

The flame absorption part of the atomic absorption spectrometer was mainly used for the determination of constant to trace metals and alkaline earth metal elements, the flame emission part was mainly used for the determination of constant to trace alkali metals and alkaline earth metals, and the atomic absorption part of graphite furnace was mainly used for the determination of trace, ultra-trace metal and non-metallic elements. ICP-AES was widely used with the advantages of low detection limit, high precision, high sensitivity, wide linear range and simultaneous detection of multiple elements, which was not only an experimental instrument for routine laboratory analysis and testing, but also one of the common analysis methods in standard methods. It had the advantage of high-throughput detection to meet the need for more accurate and faster results of secondary and trace element tests, especially in the field of non-ferrous metals. XRF was characterized by a wide variety of analytical elements, a wide range of determination concentrations, high analytical sensitivity, simple sample preparation, and fast detection speed, while its application objects are becoming more and more extensive [24]. AFS was widely used in the analysis of trace and ultra-trace elements with its high analytical sensitivity. OES was simple fast and consumes fewer samples. The outstanding advantage of glow discharge emission spectroscopy was that it can analyze surfaces as well as sample bodies with considerable depth. The method could simultaneously analyze up to 43 elements, including all metals sulfur carbon oxygen chloride and hydrogen. LIBS has the unique advantages of being non-destructive and requiring little or no sample preparation. This technology has the ability to measure any form of sample, real-time online long-distance detection of multiple elements or harsh environments [25].

Spectral analysis technology plays a crucial role in the analysis and detection of non-ferrous metals. Compared to traditional chemical analysis methods, it has the following advantages:

- The sampling form is flexible, and the representative sampling amount of rare and rare precious metals is less than that of traditional chemical methods;
- The sample pretreatment operation is simple, and there is no need for complex separation operations;
- The reference metal demand is low, and spectral qualitative analysis can be achieved in the known map;
- Fast analysis and high efficiency, set multi-channel instant multi-point acquisition, real-time output through calculator;
- Good selectivity, for the determination of elements and chemicals with similar chemical properties, such as niobium, tantalum, zirconium, hafnium, and rare earth oxides can be separated without interference, as a technical support for the determination of the content of such elements and compounds;
- High sensitivity, trace analysis can be performed using spectroscopy;
- On-site and in-situ non-destructive testing is possible.

5. Development Trend

Spectral analysis technology occupied an important position in the primary secondary trace and trace analysis fields of non-ferrous industry providing an important role in promoting the development of Chinese non-ferrous industry.

With the implementation and landing of the "dual carbon" policy, the technical requirements for non-ferrous metals have become more and more stringent, and the revision of standards has developed in the direction of green, environmental protection, energy conservation and high efficiency. Spectral analysis will likely be the direction of future standard development in the following technical areas:

1) Simultaneous determination technology of trace multiple elements;

2) Analysis techniques for elemental morphology and mineral occurrence states;

3) Analysis techniques for light mass elements;

4) On-line sampling and intelligent detection technology for field instruments.

The high-purity non-ferrous metal materials are used in spectroscopic instruments to provide services for the non-ferrous metal industry by improvement of non-ferrous metal material detection technology. The use of characteristic spectrum extraction technologies such as Genetic Algorithms [26] [27] continuum-removal and principal component analysis can achieve a better model for selecting specific variables suitable for wavelength bands, thereby reducing data redundancy and irrelevant information and improving the accuracy of analysis and detection in the field of chemical analysis.

The establishment of the standard system of spectral technology in the standard method of non-ferrous metals will further promote the development of spectroscopic technology in the field of non-ferrous metal analysis.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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