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Research Article

Determination of As, Hg, Pb and Zr in Pyrotechnic Compositions by ICP-OES

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Abstract

Fireworks produce spectacular visual effects through the combustion and explosion of pyrotechnic compositions, which are made up of oxidizers, fuels, colorants and binders. To improve safety and environmental protection, the Chinese national standard GB 10631-2013 prohibits the use of arsenic, mercury compounds and zirconium powder in pyrotechnic compositions of all firework products, and lead compounds in specific categories. Currently, the testing methods in GB/T 21242 are qualitative and often suffer from matrix interference. This study aims to establish an inductively coupled plasma optical emission spectrometry (ICP-OES) method for the rapid and accurate quantitative detection of prohibited components in pyrotechnic compositions, as the application of ICP-OES in fireworks quality control has not been explored previously. The research successfully developed an ICP-OES method. Analytical-grade reagents and standard solutions were used, and the ICP-OES operating conditions were optimized. Specific analytical lines (As 189.042 nm, Hg 194.227 nm, Pb 220.353 nm, Zr 343.823 nm) were selected to avoid interference. Different sample preparation methods were applied to effect charge and bursting charge. The calibration curves showed good linearity (correlation coefficients ≥ 0.9990), with low detection limits (0.013-0.031 μg/mL). Interference analysis confirmed negligible inter-element and matrix interference. Precision tests showed relative standard deviations of 1.64% -2.71%, and accuracy tests had recovery rates of 98.5% -101%. The established ICP-OES method enables the simultaneous determination of arsenic, lead, mercury, and zirconium in pretreated pyrotechnic compositions. With its simplicity, rapidity, low detection limits, and high precision and accuracy, this method provides a reliable approach for the quantitative analysis of prohibited components in fireworks, contributing to the quality control of fireworks products.

Keywords

Fireworks, ICP-OES, Arsenic, Mercury, Lead, Zirconium

1. Introduction

The vibrant effects of fireworks are generated by the combustion and explosion of pyrotechnic compositions. These compositions primarily consist of oxidizers (e.g., potassium nitrate, potassium perchlorate), fuels (e.g., sulfur, charcoal, aluminum, magnesium), colorants (e.g., sodium salts, strontium salts, barium salts), and binders (e.g, shellac, resins) [1]. To enhance the safety and environmental performance of pyrotechnic compositions, the Chinese national standard GB

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10631-2013 prohibits the use of arsenic, mercury compounds and zirconium powder in pyrotechnic compositions of all firework products, and lead compounds in specific categories (e.g., firecrackers, fountains, spinners, Roman candles, toys, and combinations set off by ordinary consumers), with a detection limit of 0.1% [2]. Currently, the testing methods for these four categories of substances provided in GB/T 21242 [3], referenced by GB 10631-2013, are only qualitative chemical methods rather than quantitative tests. In practical detection, these methods are sometimes prone to interference from matrix components, making the results difficult to discern. Therefore, it is highly necessary to study and establish the inductively coupled plasma optical emission spectrometry (ICP-OES) method for rapid and accurate quantitative detection of prohibited and restricted components in pyrotechnic compositions [4-7].

Inductively coupled plasma optical emission spectrometer (ICP-OES) is a rapidly developing analytical technique that enables simultaneous multi-element detection with advantages such as simplicity, low detection limits, wide linear range, minimal interference, and high accuracy [8-11]. However, its application in fireworks quality control remains unexplored. This study establishes an ICP-OES method for the determination of arsenic, mercury, lead and Zirconium in pretreated pyrotechnic compositions, demonstrating its simplicity, precision, and reliability [12-14].

2. Experimental

2.1. Reagents and Materials

Standard solutions of arsenic, mercury, lead and zirconium (1.000mg/mL each): National Research Center for Certified Reference Materials (China)

Nitric acid (HNO_3), absolute ethanol, acetone: Analytical grade

Deionized water (resistivity ≥18.2 MΩ·cm)

2.2. Instrumentation and Operating Conditions

ICP-OES spectrometer (Plasma 1000, NCS TESTING TECHNOLOGY CO., LTD):

Wavelength range: 190–500 nm

RF generator: 40.68 MHz, 1,200 W

Plasma gas: Argon (99.999%)

Coolant flow: 14 L/min

Auxiliary flow: 0.5 L/min

Nebulizer pressure: 0.2 MPa

Sample introduction:

Peristaltic pump: 30 rpm

Uptake delay: 22 s

Observation height: 15 mm

Analytical lines: As 189.042 nm, Hg 194.227 nm, Pb 220.353 nm, Zr 343.823nm.

2.3. Standard Working Solutions

To prepare mixed standard solutions, 10.0mL aliquots each of arsenic (As), mercury (Hg), lead (Pb) and Zirconium (Zr) standard stock solutions were transferred into 100mL volumetric flasks, diluted to volume with 5% nitric acid (HNO3), and vortex-mixed to homogeneity to prepare the mixed standard solution.

Subsequently, serial working solutions (1.00, 3.00, 5.00 and 7.00μg/mL for As, Hg, Pb and Zr) were generated by pipetting 1.0, 3.0, 5.0 and 7.0mL aliquots of the mixed standard solution into separate 100mL volumetric flasks, followed by dilution to the mark with 5% $HNO_3$and vortex mixing.

A 5% nitric acid solution was used as the reagent blank.

2.4. Sample Preparation

Dissect the fireworks that may contain the above-mentioned prohibited ingredients in the pyrotechnic compositions, carry out the separation and pretreatment of pyrotechnic compositions, and adopt different treatment methods for different types of pyrotechnic compositions (e.g., effect charge, bursting charge).

2.4.1. Effect Charge

Weigh a 0.5 g sample of crushed blue-light effect charge (containing potassium perchlorate, copper (II) oxide, barium nitrate, aluminum-magnesium alloy powder, polyvinyl chloride, and phenolic resin), transfer it into a G4 sintered glass crucible. Extract the material sequentially using 20 mL anhydrous ethanol in multiple portions, then filter and rinse with the same solvent. Rinse the residue three times with 20 mL acetone under vacuum to remove residual solvents. Air-dry the residue and dissolve it in 30mL 25% of nitric acid (HNO_3) by heating on a hot plate. After complete dissolution, cool the solution to room temperature and transfer it quantitatively to a 100mL volumetric flask. Dilute to the mark with deionized water, mix thoroughly, and filter through a 0.45 μm membrane to obtain a clear filtrate for analysis.

2.4.2. Bursting Charge

Weigh a 0.5g sample of bursting charge (containing potassium perchlorate, magnesium-aluminum alloy powder, and sulfur) into a 100mL beaker, added 30mL of 25% HNO_3, and heated the mixture until fully dissolved. After cooling, transfer the solution to a 100 mL volumetric flask, diluted it to the mark with deionized water, and mixed thoroughly. The mixture was membrane-filtered, and the filtrate was stored for analysis.

2.5. Analytical Procedure

The blank solution, calibration standard solutions, and

sample solutions were sequentially introduced into the ICP-OES instrument. Under optimized instrumental parameters (e.g., RF power: 1150 W, auxiliary gas flow: 0.5 L/min, nebulizer gas flow: 1.0 L/min, observation height: 12 mm), the instrument software automatically generated the calibration curve and calculated element concentrations.

3. Results and Discussion

3.1. Selection of Analytical Lines

Analytical line selection: The spectral line database within the instrument software was queried to evaluate intensity, interference, and background characteristics for candidate lines. Based on selection criteria including:

1) Absence of spectral interference from matrix elements (Cu, Al, Mg, K),
2) Symmetric background profiles,
3) High sensitivity,
4) Optimal signal-to-noise ratio, the following analytical lines were selected: As 189.042nm, Hg 194.227nm, Pb 220.353nm and Zr 343.823nm. When the As 193.76nm line is selected, there is spectral interference of the matrix element Al.

3.2. Calibration Curves and Detection Limits

Under optimized instrument parameters, blank solutions and a series of standard working solutions were injected for determination to obtain the standard working curves of each element. The blank solutions were repeatedly measured 11 times, and the detection limits of each element were calculated by dividing the standard deviation of the blank signal by three times the slope of the standard working curve.

Results are summarized in Table 1.

Table 1. *Standard working curve and detection limit.*

Analyte	Linear Equation	Correlation Coefficient	Detection Limit/(μg/mL)
As	y=166.8x-12.4	0.999 7	0.031
Hg	y=464.8x-5.4	0.999 0	0.019
Pb	y=325.0x-9.5	0.999 9	0.026
Zr	y=118.1x-14.9	0.999 4	0.013

Note: y denotes the spectral line intensity; x represents the concentration of the standard working solution (μg·mL-1)

3.3. Interference Analysis

Scan the test samples near the selected As, Hg, Pb and Zr analytical lines while ensuring uniform background signals on both sides of the spectral peaks and confirming no interference from overlapping line [15, 16].

Perform eleven repeated measurements of spectral line intensities for each element's standard solution (1.0μg/mL) and the mixed working solution, and conduct a t-test to verify that intensities remain consistent before and after mixing, indicating negligible inter-element interference.

Add 2mL of mixed standard solutions containing 10.0, 20.0, 40.0 and 60.0μg/mL Cu, Al, Mg, K and Ba to 1.0 μg/mL As, Hg, Pb and Zr mixed standards, measure the spectral line intensity changes, and demonstrate that As, Hg, Pb and Zr intensities remain unaffected by variations in Cu, Al, Mg and K concentrations, proving matrix compatibility.

3.4. Precision and Accuracy

Transfer 10mL each of effect charge and explosive charge samples solution into 100mL volumetric flasks, dilute to volume with 5% nitric acid solution, and perform 11 repeated measurements to calculate relative standard deviation (RSD). Additionally, transfer 10mL each of effect charge and explosive charge samples solution into 100mL volumetric flasks, add 2mL of mixed standard solution, dilute to volume with 5% nitric acid solution, conduct 11 repeated measurements, and calculate recovery rates based on precision measurement results. The data are shown in Table 2.

Table 2. Results of precision and recovery.

Sample	Analyte	Unspiked/(μg/mL)	RSD/%	Spike added /(μg/mL)	Measured/(μg/mL)	Recovery/%
Effect Charge	As	2.10	2.41	2.00	4.12	101.0
	Hg	2.65	2.63	2.00	4.64	99.5
	Pb	2.81	2.71	2.00	4.79	99.0
	Zr	1.25	1.64	2.00	3.24	99.5
Bursting Charge	As	2.31	1.99	2.00	4.33	101.0
	Hg	3.04	2.46	2.00	5.01	98.5
	Pb	2.36	1.97	2.00	4.37	100.5

Note: Zirconium powder is generally not added to bursting charges.

As shown in Table 2, the relative standard deviation (RSD) ranged from 1.64% to 2.71%, and the recovery rate was between 98.5% and 101%, indicating that this method possesses high precision and accuracy, which fully meets the analytical requirements for detecting prohibited components (arsenic, mercury, lead and zirconium) in pyrotechnic compositions

4. Conclusions

The ICP-OES method uses the analytical lines As 189.042nm, Hg 194.227nm, Pb 220.353nm and Zr 343.823nm for the simultaneous determination of prohibited components—arsenic, lead, mercury and zirconium—in pretreated pyrotechnic compositions of fireworks, The method demonstrates relative standard deviations (RSD) of 1.64%–2.71% and recoveries ranging from 98.5% to 101%. Featuring a simple and rapid procedure, low detection limits, and minimal interference, this approach delivers accurate and reliable results.

Abbreviations

ICP - OES	Inductively Coupled Plasma Optical Emission Spectrometry
HNO3	Nitric acid
As	Arsenic
Hg	Mercury
Pb	Llead
Zr	Zirconium
Cu	Copper
Al	Aluminum
Mg	Magnesium
K	Potassium
RSD	Relative Standard Deviations

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Author Contributions

Yang Lin: Conceptualization, Project administration, Writing–original draft

Chen Jie: Data curation, Writing–review & editing

Zeng Xu: Investigation, Resources

Zhu Yuping: Funding acquisition, Supervision

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Data Availability Statement

The data is available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare no conflicts of interest.

References

[1] Liu N, Yang L. Pyrotechnic Materials and Compositions. Changsha: Hunan Science and Technology Press; 2015, pp. 171-172.

[2] GB 10631-2013, Safety and Quality for Fireworks [S].

[3] GB/T 21242-2019, Qualitative Detection Methods for Prohibited Agents in Fireworks [S].

[4] YANG Lin, ZOU Guoqing, ZHOU Wuquan, etal. Determinationof W, Sn, Mo, Cu, Pb, Zn, S and As in tungsten-tin ore by inductively coupled plasma optical emission spectrometry (ICP-OES) with alkali fusion [J]. Chinese Journal of Inorganic Analytical Chemistry, 2023, 13(11): 1191-1196

[5] Al-Juhaimi F, Kulluk D A, Mohamed Ahmed I A, et al. Quantitative determination of macro and micro elements and heavy metals accumulated in wild fruits analyzed by ICP-OES method [J]. Environmental Monitoring and Assessment, 2023, 195(11): 1370.

[6] WANG Li, XU Yue, SUN Haifeng, et al. Determination of Zr and Hf in zirconium sand by inductively coupled plasma atomic emission spectroscopy [J]. Chinese Journal of Analysis Laboratory, 2025, 44(5): 757-761.

[7] ZHENG Yan-ping. Determination of Lead, Arsenic, Cadmium, Chromium and Mercury in Organic Fertilizers by Inductively Coupled Plasma Emission Spectrometry [J]. Guangzhou Chemical Industry, 2020, 50(20): 144-148.

[8] Li Y N. Application of ICP-OES method to determine the content of valuable rare earth elements in soil of mining areas [J]. Mineral Exploration, 2024, 15(7): 1245−1253.

[9] ZHANG Yijun, ZHANG Yuhang, CHEN Yan. Determination of Zirconium and Titanium in Marine Placer Deposits by ICP-OES with Alkali Fusion of Lithium Metaborate-Lithium Tetraborate Composite Flux [J]. Rock and Mineral Analysis, 2024, 43(6): 858-865.

[10] Khan S R, Sharma B, Chawla P A, et al. Inductively coupled plasma optical emission spectrometry (ICP-OES): A powerful analytical technique for elemental analysis [J]. Food Analytical Methods, 2022: 1-23.

[11] Teng G Q, Wang B G. Determination of 8 components in limestone by inductively coupled plasma atomic emission spectrometry with alkali fusion [J]. Metallurgical Analysis, 2024, 44(7): 88−94.

[12] Xi X L, An T T. Determination of 8 components in kaolin by inductively coupled plasma emission spectrometry [J]. Analytical Instrumentation, 2024(3): 25−30.

[13] Lu X F. Determination of 9 elements in molybdenum chromium alloy by inductively coupled plasma emission spectroscopy [J]. Ferro-Alloys, 2024, 55(3): 51−54.

[14] BIAN Dayong, LIU Shuxiang, YAO Xu. Determination of 9 micro elements in antimony ingot by inductively coupled plasma atomic emission spectrometry [J]. Metallurgical Analysis, 2024, 44(3): 65.

[15] AHMADZ, KHAN S M, PAGE S E, etal. Environmental sustainability and resilience in a polluted ecosystem via phytoremediation of heavy metals and plant physiological adaptations [J]. Journal of Cleaner Production, 2023, 385: 135733. https://doi.org/10.1016/j.jclepro. 2022.135733

[16] XU Qing, ZHANG Xiao, HONG Xuejiao, et al. Determination of rhenium content in tungsten-rhenium alloys by inductively coupled plasma emission spectrometry [J]. Chinese Journal of Analysis Laboratory, 2024, 43(10): 1484.

Biography

Yang Lin is a director of Standardization & Quality Management, China Quality Testingand Inspection Center for Fireworks, China, Senior Engineer. He completed his master of Chemical Engineering from Ocean University of China in 2007. Recognized for his exceptional contributions, Mr. Yang has been honored with core expert by national and international Standardization Technical Committee (SAC/TC149 and ISO/TC 264). He has participated in multiple national and international research collaboration projects in recent years: drafting 2 ISO standards, 4 National Standards (e.g., GB/T 35760-2017, GB/T 35030-2018) and 1 Provincial Standard, directed 5 provincial R&D projects including Key Component Detection Platform for Pyrotechnic Compositions and managed critical risk monitoring programs. In addition, he holds a Power Certified National Registered Safety Engineer certification.

Research Field

Yang Lin: Fireworks and Firecrackers Safety Testing, Pyrotechnic Composition Analysis Technology, Fireworks' Standardization Research and Development, Fireworks Product Performance Evaluation Methods, Fireworks Production Risk Assessment and Management.

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