

Rapid determination of Ferrum, Manganese, Strontium and Barium in geothermal water by ICP-OES

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Abstract: Developing a rapid and precise method for trace element analysis in geothermal water is crucial due to its high total dissolved solids and salinity, which can impact element determination. In this study, we optimized the determination of ferrum, manganese, strontium and barium in geothermal water samples collected from different regions. A matrix matching method was established for accurate quantification using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). Instrumental conditions and experimental parameters were optimized, and the influence of storage medium and storage duration on measurement accuracy were evaluated. The results demonstrated that storing geothermal water samples in 1% nitric acid had no significant impact on measurement results over an eight-week period. Calibration curve correlation coefficients exceeded 0.9998 for all target elements. The detection limits of this method ranged from 0.0002 mg/L to 0.0031 mg/L, with Relative Standard Deviations (RSD) were 0.07%–2.33%, and spike recovery rates were from 96.8% to 103.2%. The obtained data were consistent with results from the standard addition method and dilution method, demonstrating the reliability of this approach. This method offers low detection limits, high precision and excellent recovery rate, providing a robust reference for the accurate determination of ferrum, manganese, strontium and barium in geothermal water, thereby laying a solid foundation for the development and utilization of geothermal resources.

Keywords: Geothermal water; Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES); Matrix matching method; Trace element analysis

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Introduction

Geothermal energy, as a green, low-carbon, renewable and clean energy source, has gained global attention (Chen, 2014; Liu et al. 2023; Banks, 2022; Shang et al. 2024). China is rich in geothermal resources, which are widely distributed across provinces such as Yunnan, Tibet, Hebei, Shanxi, Qinghai, Chongqing, Guangdong and other provinces (cities) (Hao et al. 2014; Li et al. 2015; Guo et al. 2015; Zhang, 2017; Zhang et al. 2024; Ta et al.

2018; Shi et al. 2024). Geothermal energy systems that use groundwater as a carrier have significant potential for development and utilization. High-temperature geothermal resources are primarily used for power generation, while low-temperature geothermal resources are widely applied in heating, bathing, physical therapy, greenhouse agriculture, and many other fields. Given its extensive applications and promising future prospects, geothermal energy is of great economic and environmental value (Feng, 2017; Jia et al. 2024; Wang et al. 2024).

Analyzing the element composition of geothermal water is essential for understanding its chemical properties, determining its potential applications, elucidating its origin, estimating deep reservoir temperatures and optimizing resource utilization. The chemistry of geothermal water is complex, containing beneficial minerals and micronutrients, as well as potentially harmful

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components. Therefore, detailed studies on geothermal water composition are crucial for its sustainable and scientific development. Ferrum, manganese, strontium and barium are common trace components found in geothermal water at relatively high concentrations. Their rapid and accurate determination is critical for evaluating water quality and ensuring the safe and effective use of water quality.

Currently, the primary methods for determining trace elements in water include Ultraviolet and Visible Spectrophotometry (UV-Vis), Atomic Absorption Spectrometry (AAS), Atomic Fluorescence Spectrometry (AFS), and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) and so on (Zhang et al. 2022; Kassim et al. 2022; Kadhim et al. 2024; Ibrahim et al. 2024; Han et al. 2024). However, UV-Vis, AAS and AFS are time-consuming, complex to operate, and unsuitable for the simultaneous determination of multiple elements. Additionally, these methods often require the use of organic reagents, which can be harmful to both the environment and operators (Yu et al. 2018). While ICP-MS offers high sensitivity and precision, but it is an expensive technique that demands stringent environmental conditions and a high level of technical expertise of operators (Qiao et al. 2022).

Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) has been widely applied in the analysis of surface water, mineral water, groundwater, brine and other samples (Ashok et al. 2023; Chen, 2016; Zou et al. 2017; Liu et al. 2022; Farhadiyan et al. 2024). This technique is characterized by its low detection limit, high analytical speed, capability for simultaneous multi-element determination, and broad linear range (Deng, 2013; Zhao et al. 2020; Zhou et al. 2020). However, geothermal water typically contains elevated concentrations of Na^+ and Ca^{2+} , which can cause significant matrix effects and spectral interferences, potentially compromising the accuracy of trace element quantification, particularly for ferrum, manganese, strontium and barium.

To ensure accurate trace element analysis within complex matrices, the standard addition method, internal standard method and matrix matching technique are frequently used to mitigate interference (Srikritsawong et al. 2024). However, the standard addition method is labor-intensive and unsuitable for the rapid quantitative analysis of large sample batches. But in contrast, the internal standard method and matrix matching method offer advantages such as simplified sample preparation, reduced reagent consumption, and improved effi-

ciency in batch sample analysis (Han et al. 2020; Ngumba et al. 2016).

Geothermal water contains high levels of dissolved solids, necessitating the mitigation of matrix effects caused by high salinity. In this study, the concentrations of ferrum, manganese, strontium and barium in geothermal water were determined using ICP-OES after optimizing the instrument conditions and experimental methods. Geothermal water samples may undergo compositional changes between collection and analysis due to various factors, potentially leading to unrepresentative results. To minimize such alterations, it is crucial to implement appropriate sample preservation techniques immediately upon collection. This study investigated the influence of storage medium and storage duration on the measured results. Additionally, the matrix matching method was applied to the determination of trace components in geothermal water, providing a reliable analytical approach that supports the sustainable development and utilization of geothermal resources.

1 Experiment

1.1 Instrument

The ICP-OES system used in this study is an Optima 8000 ICP-OES (PerkinElmer, USA), equipped with both radial and axial viewing capabilities and a cross-flow nebulizer. The operating parameters for ICP-OES are outlined in Table 1.

Table 1 Operating parameters for ICP-OES determination

Parameter	Value	Parameter	Value
Power	1,400 W	Auxiliary flow	0.20 L/min
Sample flow	1.5 mL/min	View dist	15.0 mm
Nebulizer flow	0.60 L/min	Plasma flow	12 L/min

High purity Argon ($\omega > 99.99\%$).

The analytical wavelengths for ferrum, manganese, strontium and barium were 238.2 nm, 257.6 nm, 407.7 nm and 233.5 nm, respectively.

1.2 Reagents

All reagents used were of guaranteed reagent grade unless otherwise specified. Standard solutions of ferrum and manganese (1,000 $\mu\text{g/mL}$) and strontium, barium (100 $\mu\text{g/mL}$) were purchased from China Metrology Academy of Sciences. Stock solutions were stored at 4°C in a refrigerator, and working standard solutions were freshly prepared

daily by serial dilution prior to use. Superior-grade nitric acid (HNO₃) was purchased from Beijing Reagents Co. Ltd. (Beijing, China). All experiments were conducted at a controlled temperature of 20°C–22°C, regulated by an air conditioning system. Ultrapure water (18.25 M-Ω) was obtained from a Milli-Q water purification system (Millipore, Bedford, USA) and used for all solution preparation.

1.3 Collection and pretreatment of water samples

The concentrations of ferrum, manganese, strontium and barium in geothermal water vary depending on the source of hydrochemical type. In this study, geothermal water samples were collected from geothermal wells in Gansu, Guangdong, Shandong, Hebei, Anhui, Liaoning and other places. The temperatures of the collected geothermal water samples ranged from 40°C to 70°C. The hydrochemical classifications of the seven geothermal water samples are summarized in Table 2.

Due to the high temperature and elevated Total Dissolved Solids (TDS) content of the samples, it was necessary to acidify the water with nitric acid (HNO₃) before analysis to ensure that ferrum, manganese, strontium and barium remained completely dissolved in solution.

All water samples were filtered through a 0.45 μm membrane filter before analysis. After discarding the initial filtrate, 100–150 mL of the filtrate was collected, and 50% nitric acid solution was added to adjust the pH to ≤2. The prepared samples were then analyzed under the optimized ICP-OES operating conditions.

2 Results and discussion

2.1 Selection of analytical wavelengths and viewing methods

To optimize the determination of ferrum, manganese, strontium and barium in geothermal water, three to four sensitive wavelengths for each element were initially selected from the periodic table and compared with the sub-sensitive wavelengths. The instrument's spectral interference function table was used to scan multiple geothermal water samples, wavelengths with significant spectral interference were discarded.

The preliminary selected analytical wavelengths for each element were as follows:

- Fe: 238.204 nm, 259.939 nm
- Mn: 257.610 nm, 259.372 nm
- Sr: 407.771 nm, 421.552 nm
- Ba: 233.527 nm, 455.403 nm

To further refine the selection, samples containing 1 mg/L of each element were scanned to evaluate the signal strength across different wavelengths. The signal strength of different elements at various wavelengths is illustrated in Fig. 1.

Based on signal strength, peak shape, background interference, and coexistence element interference, wavelengths with excessively high or low sensitivity were discarded. The final optimized analytical wavelengths and their corresponding viewing methods were determined as follows:

- Fe: 238.204 nm (Axial)
- Mn: 257.610 nm (Axial)
- Sr: 407.771 nm (Radial)
- Ba: 233.527 nm (Axial)

The selection ensures a wide linear range, minimal background interference, and reduced spectral overlap with coexisting elements, enhancing the accuracy and reliability of the method.

2.2 Selection of RF power

Radio Frequency (RF) power significantly influence analytical line intensity and the signal-to-background ratio. To assess this effect, the RF power was varied from 1,000 W to 1,500 W, while all other parameters remained constant. A 1.0 mg/L standard solution of ferrum, manganese,

Table 2 Hydrochemical characteristics of the seven geothermal water samples

Sample ID	Sample site	Hydrochemical type	Total Dissolved Solids/mg·L ⁻¹	Na ⁺ /mg·L ⁻¹	Ca ²⁺ /mg·L ⁻¹
DR1	Lanzhou, Gansu	Na ⁺ —Cl ⁻	17,379	5,173	311
DR2	Huizhou, Guangdong	Na ⁺ —HCO ₃ ⁻	1,336	279	76.4
DR3	Heze, Shandong	Na ⁺ —SO ₄ ²⁻	5,626	773	125
DR4	Langfang, Hebei	Na ⁺ —Cl ⁻	2,823	794	232
DR5	Zhangjiakou, Hebei	Ca ²⁺ —HCO ₃ ⁻	443	32.7	140
DR6	Lu'an, Anhui	Na ⁺ —HCO ₃ ⁻	541	110	62.8
DR7	Dandong, Liaoning	Na ⁺ —Cl ⁻	4,725	1,387	58.9

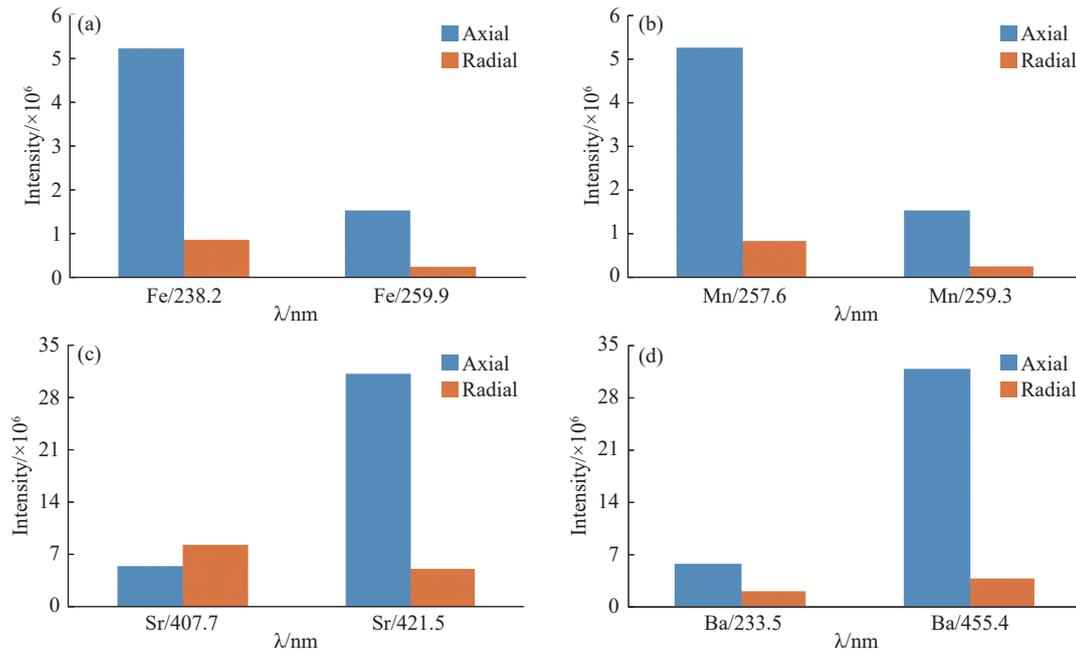


Fig. 1 Signal strength of different elements at various wavelengths

(a:Fe; b:Mn; c:Sr; d:Ba)

strontium and barium was used to measure changes in line intensity and signal-to-background ratio. The results indicated that higher RF power improved sensitivity. However, at 1,400 W, the signal-to-background ratio significantly decreased. Considering the excessive RF power may shorten the instrument's lifespan, a compromise power setting of 1,300 W was selected for this study.

2.3 Selection of atomizer flow

The atomizer gas flow rate plays a crucial role in determining the distribution of parameters within the central channel and the retention time of the sample. Therefore, selecting the optimal atomizer gas flow rate is critical when using ICP-OES for analysis. To evaluate the influence of the atomizer gas flow rate on analytical performance, the standard working solution of ferrum, manganese, strontium and barium (1.0 mg/L) was analyzed across a range of flow rates from 0.4 L/min to 0.9 L/min, while maintaining other experimental conditions constant.

The results revealed that an increase in the atomizer gas flow rate improved the signal-to-background ratio. However, when the flow rate reached 0.7 L/min, there was a marked increase in matrix effect. Therefore, an atomizer gas flow rate of 0.6 L/min was selected for this study.

2.4 Analysis of interference

The content of inorganic salts in geothermal water varies depending on the hydrochemical type, which leads to differences in sample composition compared to the standard solution. These variations affect the injection amount, atomization efficiency, and the number of excited atoms or ions, resulting in analytical errors. According to statistical data, most geothermal water contains high levels of sodium (Na^+) and calcium (Ca^{2+}). Specifically, Na^+ concentrations can reach up to 6,000 mg/L and Ca^{2+} concentrations can be as high as 400 mg/L. These elements are the primary sources of interference in the determination of ferrum, manganese, strontium, and barium. To assess the effects of Na^+ and Ca^{2+} on the analysis, aqueous solution of Na^+ (concentrations 400, 800, 1,200, 2,400, 4,000 and 6,000 mg/L) and Ca^{2+} (concentrations of 60, 120, 180, 240 and 360 mg/L) were prepared in the laboratory. The effects of these ions on the analysis of ferrum, manganese, strontium and barium (each at 1.00 mg/L) were then examined, as shown in Fig. 2.

From the data, it was observed that as the Na^+ concentration in the sample increased, the inhibitory effect on the measured elements became more pronounced. On the other hand, as the Ca^{2+} concentration increased, the results for ferrum, manganese and barium were slightly inhibited, while the results for strontium were sensitized to a certain extent. However, the recovery rates for all elements remained within 95%–105%, indicating that the influence of Ca^{2+} on the determination was

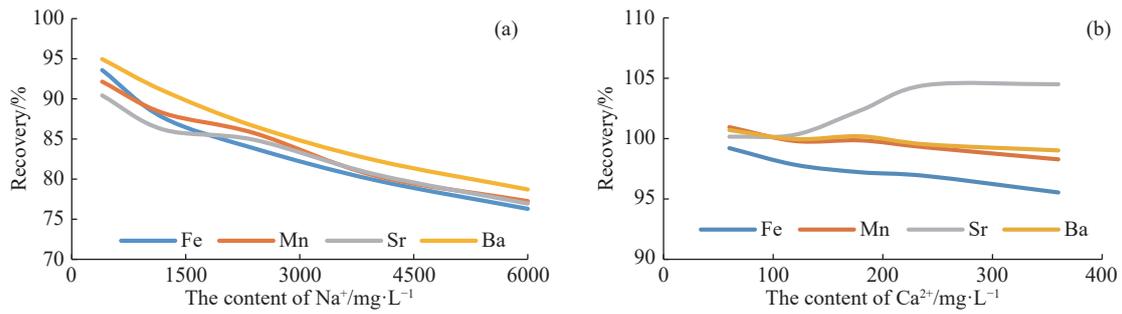


Fig. 2 Effects of co-existing ions on Natrium and Calcium determination

(a:Natrium; b:Calcium)

minimal as long as the concentration did not exceed 400 mg/L. Therefore, the effect of Ca²⁺ on the result is negligible within this concentration range. To reduce the interference caused by Na⁺, alternative methods were explored for minimizing matrix interference.

2.5 Matrix matching method and Standard addition method

The matrix matching method is the primary technique to eliminate non-spectral interference caused by matrix effect (Shen et al. 2022; Liang et al. 2022). Based on the above interference analysis, Na⁺ was identified as the main interference factor in geothermal water analysis. Therefore, NaCl was added to the standard solution to increase the Na⁺ concentration to 6,000 mg/L. During sample analysis, the Na⁺ concentration was first measured, and an appropriate amount of NaCl was added to match the Na⁺ concentration to 6,000 mg/L. Subsequently, the contents of ferrum, manganese, strontium and barium were determined.

The standard addition method, also known as the linear extrapolation method, involves adding a known amount of standard solution to the sample

and then measuring the concentration of the sample both before and after the addition. This method is particularly useful for analyzing complex samples with unknown components, but it has the disadvantage of being relatively slow. In this study, the contents of ferrum, manganese, strontium and barium in geothermal water with different hydrochemical types were measured using both the matrix matching method and the standard addition method. The comparison results are shown in Table 3 (with the internal control sample IC1 and IC2 containing 0.50 mg/L and 1.00 mg/L of the elements, respectively).

As shown in Table 3, there was minimal deviation between the measured values of the two internal control samples and the true values. Both the matrix matching method and the standard addition method were employed to determine concentrations of the elements in the laboratory geothermal water samples, and the comparison of results from these two methods showed only slight deviation. During the experiment, the samples were progressively diluted, and the measured data from these diluted samples were consistent with the results obtained using the two methods. These experiments confirmed that both the matrix matching

Table 3 Analytical results of internal control and samples

ID	Analytical results /mg·L ⁻¹							
	Matrix matching method				Standard addition method			
	Ferrum	Manganese	Strontium	Barium	Ferrum	Manganese	Strontium	Barium
IC1	0.4995	0.4902	0.5169	0.4989	0.4983	0.5011	0.5076	0.5032
IC2	1.0101	0.9964	1.0098	1.0213	1.0028	0.9896	0.9976	1.0145
DR1	4.7688	1.2538	3.7658	0.8746	4.6944	1.2612	3.6212	0.8731
DR2	0.1345	0.2576	2.5127	0.2142	0.1296	0.2489	2.5312	0.2099
DR3	1.3795	0.3122	2.7611	0.3816	1.3628	0.3075	2.8234	0.3782
DR4	0.5134	0.2110	0.8712	0.4419	0.5096	0.2087	0.8548	0.4503
DR5	0.0985	0.1038	0.9984	0.0072	0.0964	0.9958	1.0274	0.0066
DR6	N.D.	0.0098	0.2964	0.0976	N.D.	0.0094	0.2951	0.1001
DR7	2.4982	1.2131	1.5873	0.5964	2.5214	1.1876	1.6249	0.6077

method and the standard addition method were reliable for determining the concentrations of Ferrum, Manganese, Strontium and Barium in geothermal water, following the optimization of instrument conditions. Considering the working efficiency, the matrix matching method was typically preferred.

2.6 Comparative experiments of different types of instruments

Currently, there is no unified standard for the detection of geothermal water, and there is a lack of reference materials for monitoring of geothermal water. Therefore, the ICP-MS method which based on a different test principle was used in this study to conduct comparative experiments and verify the accuracy of the ICP-OES method in determining ferrum, manganese, strontium and barium in geothermal water. The comparative experimental results were shown in Table 4. As seen from the results in Table 4, the data obtained by ICP-OES was generally consistent with that measured by ICP-MS. ICP-MS is a sophisticated and costly instrument that demands stringent requirements for both the experimental environment and the technical proficiency of its operators. While ICP-MS is more suitable for determining elements with very low concentrations and ICP-OES has a wider range of applications.

2.7 Indicators of analysis quality

2.7.1 Method detection limit and detection range

The detection limit for each element was determined 12 times using a 1% pure nitric acid and 6,000 mg/L Na⁺ blank matrix solution under the selected instrument conditions. The detection limit was defined as three times the standard deviation, and the detection limit for 3 times to 5 times the standard deviation was used as the lower limit. The highest point of the standard curve with a good linear correlation coefficient was used as the upper limit. The detection limit and detection range of the method were shown in Table 5. According to the results, the lower limit of detection for each element in the method was below the class I limit specified in the "Standard for groundwater quality".

2.7.2 The relative standard deviation and recovery rate

Following the experimental procedure, seven geothermal water samples were analyzed eight times. The relative standard deviation of the method was 0.21%–1.13% for ferrum, 0.34%–1.46% for manganese, 1.02%–2.33% for strontium and 0.07%–2.12% for barium. The recoveries for ferrum, manganese, strontium and barium were 97.4%–103.2%, 96.8%–99.7%, 97.7%–103.2% and 97.2%–102.5%, respectively.

2.8 Influence of storage medium and storage time on the measured results

During storage, the composition of natural water is

Table 4 Comparison of analytical results of ICP-OES and ICP-MS

ID	Analytical results/mg·L ⁻¹							
	ICP-OES				ICP-MS			
	Ferrum	Manganese	Strontium	Barium	Ferrum	Manganese	Strontium	Barium
DR1	4.7688	1.2538	3.7658	0.8746	4.9126	1.2544	3.8679	0.8633
DR2	0.1345	0.2576	2.5127	0.2142	0.1421	0.2713	2.5691	0.2228
DR3	1.3795	0.3122	2.7611	0.3816	1.4091	0.3085	2.7834	0.3992
DR4	0.5134	0.2110	0.8712	0.4419	0.5040	0.2276	0.8964	0.4622
DR5	0.0985	0.1038	0.9984	0.0072	0.0998	0.1064	1.0019	0.0078
DR6	N.D	0.0098	0.2964	0.0976	0.0011	0.0096	0.2993	0.1002
DR7	2.4982	1.2131	1.5873	0.5964	2.5096	1.2213	1.5966	0.6003

Table 5 Detection limit and measured range of the method

Element	Linearity	Detection limit/mg·L ⁻¹	Linear range/mg·L ⁻¹
Ferrum	0.9998	0.0013	0.005–10.0
Manganese	0.9999	0.0002	0.001–10.0
Strontium	0.9998	0.0003	0.001–10.0
Barium	0.9999	0.0002	0.001–10.0

prone to changes due to physical and chemical equilibrium conditions, biological actions, precipitation and container adsorption, leading to errors in the analysis results. The mineral composition in geothermal water can precipitate, adsorb and transform with changes in temperature and pressure, resulting in a decrease in solubility. To ensure the representativeness of the samples, this study investigate the effects of storage medium and storage time on the samples.

One sample (DR1) was selected to compare raw water, 1% nitric acid and 1% hydrochloric acid acidification over eight consecutive weeks. The concentrations of each element to be measured in geothermal water over the storage period were shown in Fig. 3.

The comparison of the test results indicated no significant difference between the original and acidified samples for strontium and barium over the eight-week period. However, the concentrations of ferrum and manganese in the original samples decreased with time over the eight weeks, and iron red precipitated in the sample bottle by day 2, reaching the lowest value at week 3. The

results showed that acidification could prevent precipitation or adsorption of the samples, thus avoiding element loss. No significant decrease in the concentration of any element was observed during the eight-week storage period.

The test results for the two acidified samples were not notably different. Considering the interference of hydrochloric acid in the determination of other elements, especially when using the ICP-OES method, 1% nitric acid was chosen as the storage medium for geothermal water samples.

3 Conclusion

After determining the analytical spectral lines for ferrum, manganese, strontium and barium, optimizing the working conditions of the instrument, and eliminating the interference factors, the detection limit, minimum quantitative detection limit, and linear range of the method all exceed the recommended values in the Chinese Geological and Mineral Industry Standards for groundwater quality analysis (DZ/T 0064.42–2021). The method offers the advantages of a low detection

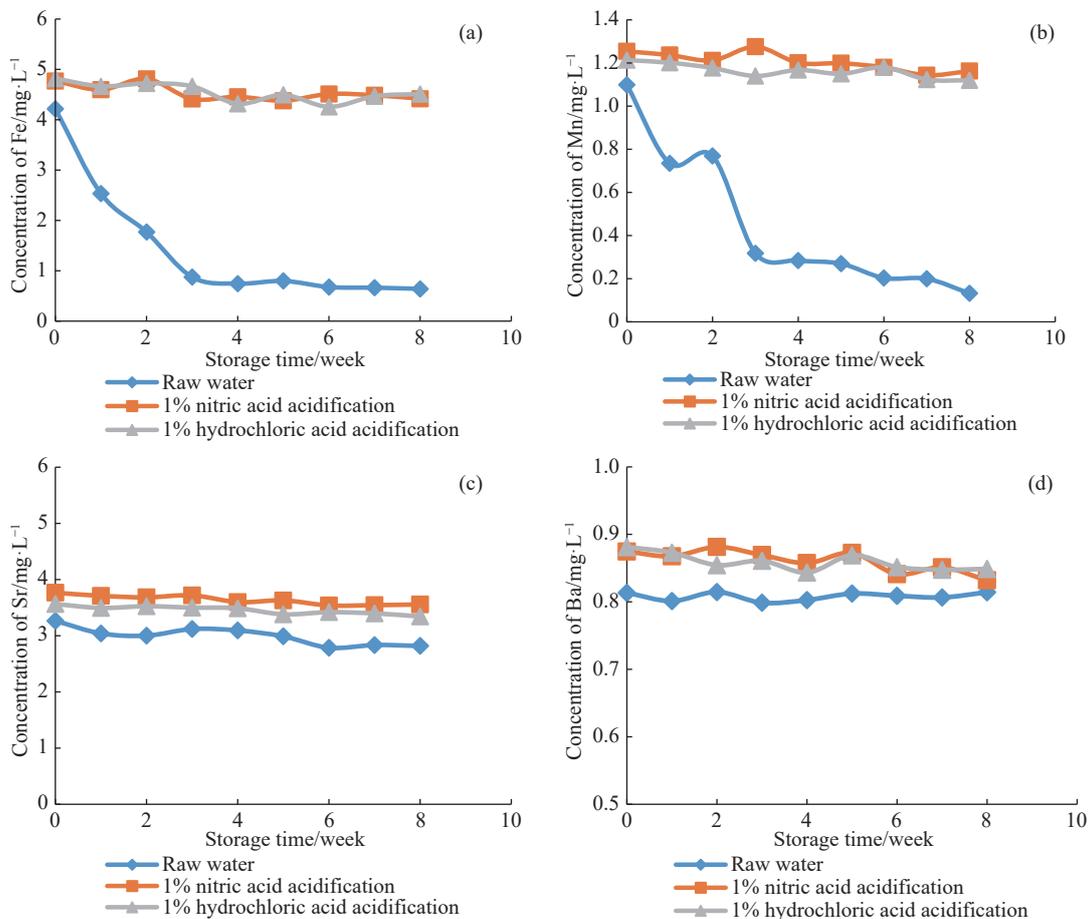


Fig. 3 The concentration of each element to be measured in geothermal water as a function of storage time (a:Fe; b:Mn; c:Sr; d:Ba)

limit, good precision and high recovery rate. It enables the continuous determination of ferrum, manganese, strontium and barium in large quantities of geothermal water samples and provides a theoretical foundation and data support for the further research and development of geothermal water.

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