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Comparative Evaluation of Aqua Regia and Tri-Acid Digestion Methods for the Determination of As, Cu, and Pb in an Ore-Grade Certified Reference Material by Flame Atomic Absorption Spectrometry

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ABSTRACT

Trace element analysis in ore grade samples is critical for mineral exploration and resource evaluation. The choice of digestion method greatly influences the accuracy of results, which form the basis for mining investment decisions. This study compares the performance of aqua regia (partial digestion) and tri-acid digestion (near-total digestion) methods for determining Arsenic (As), Copper (Cu), and Lead (Pb) concentrations. Measurements were conducted using Flame Atomic Absorption Spectrometry (AAS) with Certified Reference Material (CRM) as a validation standard, while statistical analysis was performed using the Wilcoxon Signed-Rank Test. Results show complementary performance between the two methods. Aqua regia provided the best accuracy for As (recovery 100.19%, bias 0.19%), but systematically overestimated Cu (bias 14.19%) and Pb (bias 14.14%). In contrast, tri-acid digestion delivered superior accuracy for Pb (recovery 101.27%, bias 1.27%) and better results for Cu (bias 4.83%), yet overestimated As (bias 12.25%). Statistical tests confirmed that only Pb by tri-acid ($p = 0.7213$) and As by aqua regia ($p = 0.8785$) showed no significant difference from CRM values. Operationally, aqua regia is more advantageous due to shorter digestion time (2 hours), lower cost, and better safety. Tri-acid digestion requires a longer time, higher cost, and strict safety protocols due to HF usage. This study concludes that digestion method selection should follow the fit-for-purpose principle based on target elements, sample matrix, and laboratory capabilities.

1. Introduction

Trace elements such as Arsenic (As), Copper (Cu), and Lead (Pb) play a vital role in modern geochemical exploration and ore grade characterization. These elements serve not only as pathfinders for valuable mineral deposits but also as critical factors influencing metallurgical recovery, economic valuation, and environmental compliance in mining operations [1]. In the context of increasing global demand for base metals driven by renewable energy technologies and electric vehicle batteries, accurate determination of trace element concentrations in ore grade samples has become increasingly important for reliable resource estimation and investment decision-making [2], [3].

Sample preparation, particularly the digestion stage, is widely recognized as one of the most critical steps in elemental analysis because it directly affects the efficiency of element release from complex mineral matrices [4]. Two commonly used acid digestion techniques in geochemical laboratories are aqua regia digestion (a mixture of HCl and HNO_3 in a 3:1 ratio) and tri-acid digestion (typically involving HF, HNO_3, and $HClO_4$) [5]. Aqua regia is generally classified as a partial digestion method that effectively dissolves sulfide minerals and some oxides, while tri-acid digestion approaches near-total digestion by breaking down resistant silicate and refractory phases [6], [7].

Recent studies have shown that the choice of digestion method can lead to significant variations in recovery rates, precision, and accuracy, especially when using Atomic Absorption Spectrometry (AAS) as the analytical instrument [8]. Aqua regia offers advantages in terms of operational simplicity, lower cost, and reduced safety risks, making it suitable for routine analysis. However, it often underperforms in dissolving silicate-bound elements. Conversely, tri-acid digestion provides more comprehensive matrix decomposition but requires specialized equipment and stringent safety protocols due to the highly hazardous nature of hydrofluoric acid (HF) [9], [10].

Despite the widespread use of both methods in commercial and research laboratories, comparative studies focusing specifically on ore-grade samples with AAS detection remain limited, particularly in the Indonesian context, where lateritic and porphyry deposits are dominant [11]. Many previous investigations have highlighted element-specific biases, such as overestimation or under-recovery, depending on the mineralogical association of the target elements [12], [13], [14].

This study aims to systematically compare the performance of aqua regia and tri-acid digestion methods for the determination of As, Cu, and Pb in Certified Reference Material (CRM) of ore grade samples using Flame Atomic Absorption Spectrometry (AAS). The research evaluates recovery, precision, accuracy, and operational aspects (time, cost, and safety) to provide practical recommendations for method selection based on the fit-for-purpose principle. By addressing these aspects, the study contributes to improved analytical reliability in mineral exploration and supports more sustainable laboratory practices in the mining industry.

2. Research and Methodology

2.1. Research Design

This study employed a quantitative experimental design to compare the analytical performance of two acid digestion methods, namely aqua regia digestion and tri-acid digestion (HF-HNO_3-$HClO_4$), for the determination of As, Cu, and Pb in geological samples. A Certified Reference Material (CRM) from the Geochem Base Metal (GBM) series was used to provide a homogeneous matrix and certified reference values for method evaluation [15].

The digestion method was treated as the independent variable, while analytical performance was evaluated based on elemental recovery, precision (RSD), accuracy (bias), digestion time, and reagent consumption. Each method was applied to ten replicate samples (0.5000 g), and all analyses were conducted under identical experimental conditions [16].

Method performance was assessed using quality control procedures involving CRM, reagent blanks, and replicate analyses. Differences between digestion methods were evaluated using the Wilcoxon signed-rank test ($\alpha = 0.05$) to determine statistically significant differences in analytical performance [9].

2.2. Materials and Equipment

A Certified Reference Material (CRM) from the Geochem Base Metal (GBM) series was used throughout this study to evaluate the performance of the acid digestion methods. The CRM contains certified concentrations of 3705 mg kg^{-1} Cu, 270 mg kg^{-1} Pb, and 32 mg kg^{-1} As, with a mineralogical matrix representative of base-metal ore samples containing sulfide and silicate phases. The use of a matrix-matched CRM ensured analytical consistency and provided a reliable basis for assessing digestion efficiency and elemental recovery.

All chemical reagents were of analytical reagent (AR) grade or higher purity. Concentrated hydrochloric acid (HCl, 37%), nitric acid (HNO_3, 65%), perchloric acid ($HClO_4$, 72%), and hydrofluoric acid

(HF, 40%) were used for sample digestion. Aqua regia was prepared from HCl and HNO_3 immediately before use, whereas the tri-acid digestion employed a mixture of HF, HNO_3, and $HClO_4$ to achieve complete decomposition of silicate-bearing matrices and quantitative release of target elements [5].

Samples were weighed using an analytical balance with a readability of 0.0001 g. Digestions were performed on a temperature-controlled hotplate using polytetrafluoroethylene (PTFE) digestion vessels to ensure chemical resistance against concentrated acids, particularly HF. Conventional laboratory glassware was used where compatible with the reagents employed.

Elemental concentrations of Cu, Pb, and As were determined using a Flame Atomic Absorption Spectrometer (FAAS) equipped with element-specific hollow cathode lamps. Instrument calibration was conducted using certified standard solutions according to the manufacturer's operating procedures. All laboratory materials and analytical procedures complied with internationally accepted practices for trace-element determination in geological materials

2.3. Sampling Procedure

The Certified Reference Material (CRM) was thoroughly homogenized by manual mixing followed by riffle splitting prior to subsampling. Ten replicate aliquots of 0.5000 ± 0.0001 g were prepared for each digestion method (aqua regia and tri-acid), resulting in a total of 20 analytical samples.

All subsampling was performed under clean laboratory conditions using acid-cleaned sampling tools and disposable weighing boats to minimize contamination. Each aliquot was transferred to a labeled PTFE digestion vessel and identified according to the digestion method and replicate number before digestion.

2.4. Sample Preparation

Prior to digestion, the CRM subsamples were oven-dried at 105 ^{0}C for 24 h to remove residual moisture. The dried material was ground using an agate mortar and pestle and sieved to obtain a particle size of <74 µm (200 mesh). The prepared samples were subsequently stored in sealed polyethylene containers and kept in a desiccator until acid digestion to prevent moisture uptake.

2.5. AAS Analysis Procedure

Following acid digestion, the solutions were cooled, filtered through Whatman filter paper, and diluted to the required volume with deionized water. Elemental concentrations of Cu, Pb, and As were determined in triplicate using a Flame Atomic Absorption Spectrometer (FAAS) under the manufacturer's recommended operating conditions for each element.

Quantification was performed using matrix-matched calibration standards prepared in the corresponding acid media. Reagent blanks and Certified Reference Material (CRM) analyses were included in each analytical batch for quality control. Instrument performance was verified through calibration linearity ($R^2 \geq 0.999$) and routine quality control measurements prior to sample analysis [17], [18].

2.6. Elemental Content Analysis Using AAS

The concentrations of Arsenic (As), Copper (Cu), and Lead (Pb) were quantified using Flame Atomic Absorption Spectrometry based on the Beer-Lambert law. Each element was measured at its specific wavelength using appropriate hollow cathode lamps. Calibration was performed with matrix-matched standards, and all calibration curves showed excellent linearity ($R^2 \geq 0.999$).

Percent recovery (%R) was calculated by comparing the measured concentration with the certified CRM value. Bias and relative standard deviation (RSD) were also determined to evaluate accuracy and

precision. This comprehensive approach allowed for a thorough assessment of the extraction efficiency of both digestion methods. All procedures followed standard quality control protocols to ensure the reliability of the results [19], [20].

3. Results and Discussion

3.1. Dependent Distribution of As, Cu, and Pb

The analytical results obtained from this study clearly demonstrate significant differences in the performance of aqua regia and tri-acid digestion methods when applied to ore-grade Certified Reference Material. The Certified Reference Material used in this research has the following certified values and statistical parameters as shown in Table 1.

Table 1. Certified Values and Statistical Parameters of the Geochem Base Metal CRM

Element	Grade (ppm)	Standard Deviation	Num of Analyses	Confidence Interval
Nickel (Ni)	229	19	86	± 4.1
Copper (Cu)	3705	194	111	± 36.6
Zinc (Zn)	795	61	101	± 12.2
Lead (Pb)	270	28	99	± 5.7
Arsenic (Ar)	32	6	78	± 1.3
Cobalt (Co)	34	7	90	± 1.4
Silver (Ag)	7.4	1.5	93	± 0.32

Table 2 presents the measured concentrations of Cu, Pb, and As obtained using the aqua regia and tri-acid digestion methods, compared with their certified CRM values. The aqua regia digestion produced higher measured concentrations for Cu and Pb than the certified values, whereas the tri-acid method yielded results that were closer to the certified concentrations for these elements. For As, both digestion methods produced values comparable to the certified concentration, although the tri-acid method showed a slightly higher mean value. Overall, the results indicate differences in analytical response between the two digestion methods, particularly for Cu and Pb.

Table 2. Measured Concentrations by Digestion Method Compared to Certified Values (ppm)

Element	Certified Value	Aqua Regia (Mean ± SD)	Tri-Acid (Mean ± SD)
Cu	3705	4230.70 ± 234.14	3883.77 ± 110.33
Pb	270	308.19 ± 15.60	273.43 ± 21.70
As	32	32.06 ± 3.13	35.92 ± 1.94

The two digestion methods exhibited complementary analytical performance depending on the geochemical association of the target elements within the ore matrix. Aqua regia produced systematically higher concentrations of Cu and Pb than the certified values, indicating a positive bias of approximately 14%, whereas tri-acid digestion yielded results that were closer to the certified concentrations (Cu: 4.83%; Pb: 1.27% bias). The improved agreement obtained with tri-acid digestion is consistent with the enhanced dissolution of silicate and refractory phases by HF-containing acid mixtures, resulting in more complete elemental recovery from complex geological matrices [5], [9]. In contrast, aqua regia provided the closest agreement with the certified value for As, while tri-acid digestion slightly overestimated its concentration. These findings support the *fit-for-purpose* principle, highlighting that the selection of an appropriate digestion method should consider the mineralogical

characteristics of the sample and the elemental host phases to achieve optimal analytical accuracy [6], [16], [18].

The observed differences between the two digestion methods have important implications for routine geochemical analysis of ore-grade materials. Aqua regia is suitable for rapid screening applications due to its operational simplicity, lower reagent requirements, and reduced safety concerns, whereas HF-containing tri-acid digestion is more appropriate for samples with significant silicate or refractory mineral phases, where improved recovery of Cu and Pb is required [21], [22]. Regardless of the digestion procedure employed, analytical quality should be verified through the routine use of Certified Reference Materials (CRMs), reagent blanks, and replicate analyses to ensure measurement accuracy and precision. Where analytical results may influence resource estimation or environmental assessment, cross-validation using alternative digestion methods is recommended to minimize systematic bias and improve confidence in the reported data [5], [23].

As presented in Table 2 and visualized in Figure 1, the two digestion methods showed distinctly complementary performance. Aqua regia digestion consistently produced higher measured values for Copper (4230.70 mg/L) and Lead (308.19 mg/L), indicating systematic overestimation of approximately 14.19% for Cu and 14.14% for Pb compared to the certified values [9]. However, for Arsenic, aqua regia delivered highly accurate results (32.06 mg/L), with recovery reaching 100.19% and very minimal bias (0.19%) [11].

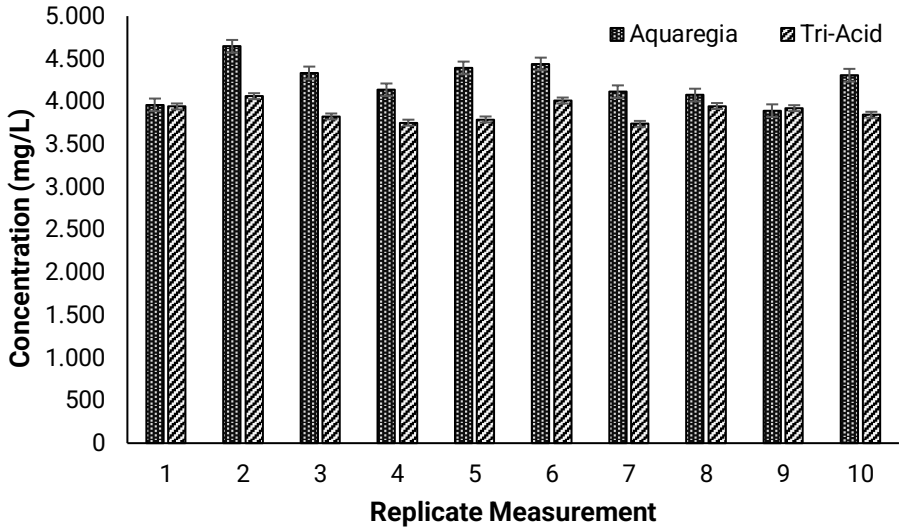

Figure 1. Graph of Copper (Cu) concentration obtained from the aqua regia and tri-acid digestion methods.

Based on Figure 1, the aqua regia digestion method produced systematically overestimated Cu concentrations, with an average concentration of 4230.697 mg/L, a bias of 14.19%, and a recovery of 114.19%. This positive bias may be attributed to matrix and spectral interferences that enhance the analytical signal, as well as the non-selective co-dissolution of matrix components due to the strong oxidizing power of aqua regia, which can result in spectral overlap and elevated Cu. In contrast, the tri-acid digestion method yielded more accurate results, with an average concentration of 3883.770 mg/L, a bias of 4.83%, and a recovery of 104.83%, indicating acceptable analytical performance. The superior performance of the tri-acid method is associated with its ability to achieve near-total digestion of Cu-bearing sulfide and silicate matrices, where HNO_3 oxidizes sulfide minerals and HF effectively decomposes silicate structures. Therefore, tri-acid digestion is more suitable for total Cu determination

in ore samples, while results obtained using aqua regia should be verified with Certified Reference Materials (CRMs) to minimize systematic bias.

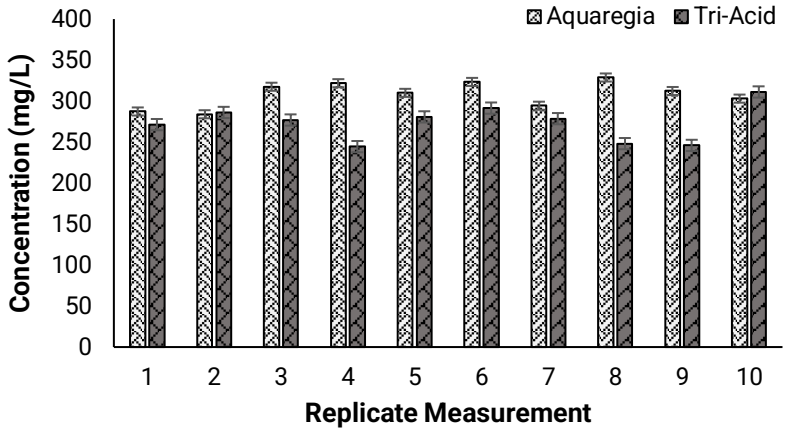

Figure 2. Graph of Lead (Pb) concentration obtained from the aqua regia and tri-acid digestion methods.

For lead (Pb), the aqua regia digestion method produced an average concentration of 308.190 mg/L, with a bias of 14.14% and a recovery of 114.14%, indicating systematic overestimation (Figure 1). This positive bias is likely associated with matrix and spectral interferences [23], [24]. Residual HCl can promote the formation of $PbCl_2$, which exhibits different atomization characteristics from the calibration standards, thereby enhancing the analytical signal. In addition, oxidation of PbS in the presence of HNO_3 may produce sparingly soluble $PbSO_4$, which can interfere with Pb determination. The strong oxidizing nature of aqua regia may also dissolve additional matrix components, increasing background absorbance and contributing to spectral interference. In contrast, the tri-acid digestion method yielded an average Pb concentration of 273.434 mg/L, with a bias of 1.27% and a recovery of 101.27%, indicating superior analytical accuracy. This improved performance is attributed to the absence of HCl, preventing $PbCl_2$ formation, while $HClO_4$ facilitates the dissolution of $PbSO_4$ and other refractory Pb-bearing minerals, and HF effectively decomposes silicate matrices, resulting in more complete digestion and more reliable Pb quantification.

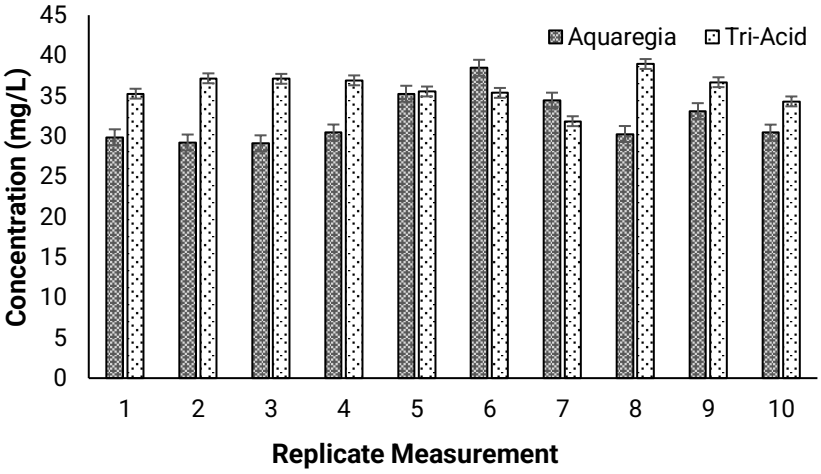

Figure 3. Graph of Arsenic (As) concentration obtained from the aqua regia and tri-acid digestion methods.

In figure 3, arsenic (As) showed the best analytical performance with the aqua regia digestion method, yielding an average concentration of 32.062 mg/L, a bias of 0.19%, and a recovery of 100.19%. This high accuracy is attributed to the ability of aqua regia to effectively dissolve As-bearing sulfide minerals, such as arsenopyrite (FeAsS), through the oxidizing action of Cl_2 and NOCl, which break As–S bonds. In addition, the highly acidic conditions (pH < 1) and the presence of chloride ions stabilize arsenic as soluble chloro-complexes, minimizing re-precipitation and improving analytical recovery (Singhania et al., 2006). In contrast, the tri-acid digestion method produced a higher average As concentration (35.919 mg/L), with a bias of 12.25% and a recovery of 112.25%, indicating systematic overestimation. This may result from the dissolution of silicate matrices, which introduces elements such as Al, Si, and Ca that can contribute to spectral interference near the As analytical wavelength (193.7 nm), leading to elevated absorbance [7], [10]. Furthermore, the absence of HCl reduces the formation of stable arsenic chloro-complexes, while the strong oxidizing action of $HClO_4$ may promote excessive oxidation of As^{3+} to As^{5+}, affecting measurement sensitivity and overall analytical accuracy.

3.2. Analysis Results of Other Components

The statistical validation using the Wilcoxon Signed-Rank Test (Table 3) confirms these observations. Only Pb measured by tri-acid digestion (p = 0.7213) and As measured by aqua regia (p = 0.8785) showed no significant difference from the CRM values. Both methods showed significant bias for Cu, while aqua regia was inaccurate for Pb and tri-acid was inaccurate for As.

Table 3. Wilcoxon Signed-Rank Test Results Comparing Tri-Acid Digestion and Aqua Regia Methods Against CRM Values

Element/Method	CRM Value	W^+	W^-	T Calculated	T Critical	Z Calculated	p-Value
Cu - Tri-Acid	3705	55	0	0	8	-2.803	0.0051
Pb - Tri-Acid	270	31	24	24	8	-0.357	0.7213
As - Tri-Acid	32	54	1	1	8	-2.701	0.0069
Cu - Aqua Regia	3705	55	0	0	8	-2.803	0.0051

The analytical performance of the digestion methods varied according to the mineralogical association of the target elements. Tri-acid digestion consistently produced Cu and Pb concentrations that were closer to the certified values than those obtained by aqua regia, indicating improved recovery from the CRM matrix. This behavior is consistent with the ability of HF-containing acid mixtures to decompose silicate and refractory minerals that retain Cu and Pb, thereby providing more complete elemental liberation [25]. In contrast, the higher Cu and Pb concentrations obtained by aqua regia indicate a systematic positive bias, likely associated with partial digestion, matrix effects, and spectral interferences.

A different trend was observed for As, where aqua regia produced the closest agreement with the certified value, whereas tri-acid digestion resulted in a moderate positive bias. This finding suggests that the predominant As-bearing phases in the CRM were effectively dissolved under aqua regia conditions, while the more aggressive tri-acid digestion may have increased matrix-derived interferences during FAAS determination [16], [23], [26]. Similar observations have been reported for geological materials, where As determination is influenced not only by digestion efficiency but also by solution chemistry and instrumental matrix effects.

Overall, these findings demonstrate that the two digestion methods exhibit complementary analytical performance rather than a universally superior capability. Tri-acid digestion is more suitable for the determination of Cu and Pb in silicate-rich or refractory matrices because it achieves more complete digestion, whereas aqua regia provides more reliable results for As by preserving favorable

solution chemistry and minimizing analytical interferences. Therefore, the selection of a digestion procedure should follow the fit-for-purpose principle, considering both the mineralogical characteristics of the sample and the target analytes to achieve optimum analytical accuracy [1], [10], [11], [27], [28].

3.3 Operational Aspects

From an operational perspective, notable differences exist between the two digestion methods. Aqua regia digestion requires approximately 2 hours at 95°C, offering relatively fast processing time. In contrast, tri-acid digestion demands longer processing time, typically 3 to 4 hours, with temperatures reaching up to 180°C.

In terms of cost, aqua regia is more economical due to the lower price of HCl and HNO_3 compared to the more expensive HF and $HClO_4$ required in tri-acid digestion. Regarding safety, aqua regia can be performed using standard laboratory glassware and conventional fume hoods. Tri-acid digestion, however, requires specialized equipment such as PTFE vessels and enhanced fume hood systems with wash-down capabilities to handle the highly corrosive and hazardous nature of hydrofluoric acid (HF) and perchloric acid ($HClO_4$). Strict safety protocols, including the availability of calcium gluconate gel for immediate treatment of HF skin exposure, are mandatory [7], [26].

Table 3. Operational Comparison between Aqua Regia and Tri-Acid Digestion

Aspect	Aqua Regia	Tri-Acid Digestion
Time	2 hours, 95°C	3–4 hours, up to 180°C
Cost	Lower	Higher
Safety	Safer (standard equipment)	High risk (corrosive, requires special equipment)

The operational advantages of aqua regia make it more practical and suitable for routine laboratory analysis, especially in laboratories with limited resources or standard facilities. On the other hand, tri-acid digestion is preferred when maximum recovery of base metals from complex silicate-rich matrices is required, despite its higher operational demands and safety considerations.

4. Conclusion

This study demonstrated that the analytical performance of acid digestion methods depends on the geochemical characteristics of the target elements and the sample matrix. Aqua regia provided the most accurate determination of As, whereas tri-acid digestion yielded better agreement with the certified values for Cu and Pb, reflecting its greater ability to decompose silicate and refractory mineral phases. Statistical evaluation further confirmed that the suitability of each digestion method is element-dependent rather than universally applicable.

From a practical perspective, aqua regia is appropriate for routine analyses requiring rapid, cost-effective, and safer sample preparation, while tri-acid digestion is preferable for the accurate determination of base metals in complex geological matrices where near-total digestion is required. These findings support the *fit-for-purpose* approach in analytical geochemistry, emphasizing that digestion procedures should be selected according to the analytical objective, mineralogical characteristics of the sample, and laboratory capabilities to achieve reliable and accurate trace-element determination.

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