

Aluminum in rocks: Optimized microwave-assisted acid digestion and UV-Vis spectrophotometric measurement

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(Received July 2, 2023; Revised August 12, 2023; Accepted August 17, 2023)

Abstract: Aluminium (Al) is one of the major elements in rocks and its concentration can be varied, depending on different rock types as well as sources. The present study aimed to propose an analytical method based on the UV-Vis as a cheap, simple, and common instrument equipped in most laboratories for Al quantification in rocks after the microwave assisted acid digestion. The aluminone and 8-hydroxyquinoline were investigated for the colorimetric assay. The results show that the 8-hydroxyquinoline reagent was more favorable in terms of the minimized affects of the potential interferences present in the digested solutions, i.e., Fe³⁺, Si⁴⁺ and F⁻. The calibration curve was constructed from 0.10 mg/L to 3.00 mg/L with the goodness of linearity ($R^2 = 0.9996$). The limits of detection and quantification (LOD and LOQ) were estimated, i.e., 0.029 mg/L and 0.087 mg/L, respectively. The 8-hydroxyquinoline was applied to real rock samples, demonstrating favorable precision (RSD = 0.34 %-1.8 %) and no remarkable differences were found compared to the inductively coupled plasma-mass spectrometry (ICP-MS) as a reference measurement approach.

Key words: Al, rocks, aluminone, 8-hydroxyquinoline, colorimetric

1. Introduction

Understanding the chemical compositions of rocks plays an important role in geological and geographical studies. To explore and investigate the minerals present in rocks, it is necessary to understand the components, distribution patterns, and geological conditions of rocks in nature. The study of the chemical compositions

of rocks also helps to know their origins and classification. Therefore, there is a certain demand for the analysis of the basic chemical components of rocks.

Most types of rocks in nature are composed of inorganic minerals, defined as inorganic solid substances with a specific crystalline structure and chemical compositions. The differences in structure,

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composition, and proportions of these inorganic minerals contribute to the diversity of rocks on the Earth's surface. By studying the chemical compositions of thousands of rock samples globally, scientists have identified eight major elements that play a role in the formation of inorganic minerals, i.e., O (46.6 %), Si (27.72 %), Al (8.13 %), Fe (5 %), Ca (3.63 %), Na (2.83 %), K (2.59 %), and Mg (2.09 %).¹

There are various types of rocks formed on the Earth. For instance, igneous rock is formed by the solidification and crystallization of molten silicate solution. Due to its primary composition of SiO₂, this type of rock exhibits a relatively high chemical resistance, and it serves as the source material for certain types of sedimentary and metamorphic rocks. The SiO₂ content in igneous rock varies between approximately 40 % and 75 %, depending on the formation conditions, and the content of Al₂O₃ ranges from around 12 % to 19 %.² Sedimentary rock is formed through the sedimentation of fragmented pieces of igneous rock, organic matters, or chemically precipitated materials. This is followed by the cementation of specific substances and lithification. The process of cementation can occur at or near the Earth's surface, especially in the case of carbonate-rich sediments.^{3,4} Another type is metamorphic rock, formed through the alteration of any type of rock (igneous, sedimentary, or pre-existing metamorphic rocks) under changing environmental conditions, such as temperature and pressure, compared to the original conditions those rocks were formed. These temperature and pressure conditions are always higher than their counterparts at the Earth's surface and must be sufficiently high to alter the original minerals into different mineral forms or into different forms of the same mineral, e.g., through recrystallization. Metamorphic rocks constitute approximately 15 % of the Earth's crust.³

As mentioned earlier, aluminum (Al) is one of the main compositions in rocks, accounting for approximately 8 % of the Earth's crust. To determine the Al contents in rocks, both destructive methods, e.g., acid digestion and measured by atomic absorption spectroscopy (AAS), inductively coupled plasma and

optical emission spectroscopy (ICP-OES) or mass spectrometry (ICP-MS), UV-Vis spectrophotometry,^{3,5,6} and the non-destructive methods, e.g., X-ray Fluorescence (XRF) and X-ray Diffraction (XRD).^{7,8} Regarding destructive methods, microwave-assisted acid digestion (with pressure) demonstrates several advantages compared to other conventional digestion approaches, such as a decrease in digestion time, the amount of reagents and samples used, then resulting in the improved repeatability and a low risk of sample contamination.^{9,10} The sample liquid after digestion is then proceeded to the measurement step using different instrumental analytical methods as mentioned earlier.

Due to high Al concentrations in rocks, the method sensitivity is not a priority, but the simplicity and reduced cost should be concerned. The UV-Vis instruments are commonly equipped in many laboratories, even for those not focusing on analytical activities, and demonstrate a good candidate as a measurement approach. However, before UV-Vis measurement, the colorimetric assay is necessary for Al detection and several reagents could be used, e.g., chrome azurol S, 8-hydroxyquinoline, and aurintricarboxylic acid (aluminone).¹¹ Although Al is present in high concentrations, rocks also contain many different other metallic and non-metallic components, resulting in the potential interferences during the colorimetric reactions. Therefore, proper investigations should be carried out for the colorimetric assays to ensure the reliability of the obtained analytical results. In the present study, two different colorimetric reagents, including aluminone and 8-hydroxyquinoline, were used and the potentially interfering ions (Fe³⁺, Si⁴⁺ and F⁻) were investigated to discover the suitable colorimetric assay for Al quantification in rocks by UV-Vis after the microwave-assisted acid digestion. The analytical method performance, including limits of detection and quantification (LOD and LOQ), calibration curve, precision, and recoveries, was then evaluated with the help of AOAC guidelines.¹² The method was also applied to determine the contents of Al in real rock samples and ICP-MS was used as a reference method for comparison.

2. Experimental

2.1. Sample collection and pre-treatment

A total number of seven rock samples were collected, including:

- Three surface rock samples of SR-1, SR-2, and SR-3 were directly collected from the surface and existed in a monolithic state. These rocks were reported to mainly compose of inorganic minerals, low content of organic components, and no moisture.

- Four drilling rock samples of DR-1, DR-2, DR-3, and DR-4 were drilled from the ground and compressed into a cake-shaped block, 10-cm diameter, and 3-4-cm thickness. These rocks were reported to have more complicated compositions compared to the surface rocks, including soils, sand, and certain organic impurities. Moreover, the rocks were relatively wet, i.e., the moisture content of 20-30 % w/w.

For pretreatment, the rocks were washed by deionized water (DIW), dried at 105 °C to their constant mass, crushed, and finely ground, then sieved through a 140-mesh membrane.

2.2. Sample digestion procedure

The samples were dissolved by the microwave-

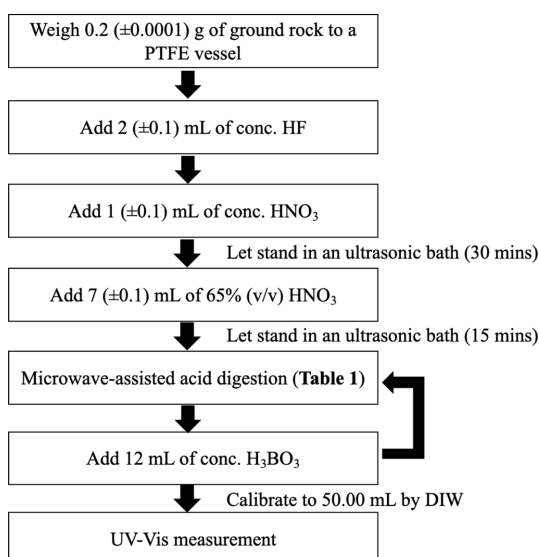


Fig. 1. Scheme of the microwave-assisted acid digestion for Al determination in rocks (conc.: concentrated).

assisted acid digestion¹³ as presented in Fig. 1, in which all the chemicals and reagents were purchased from Merck (Germany). The microwave digestion system was Anton Paar Multiwave 3000.

2.3. Aluminium measurement by UV-Vis spectrophotometry

2.3.1. The aluminon reagent assay

The colorimetric procedure followed the aluminon reagent assay. Briefly, the digested sample solution was added to a 50-mL volumetric flask. Then, 1.00 mL of 10 % w/v hydroxylamine (Merck, Germany) was added, and the solution was gently shaken and let stand for 15 minutes. After that, the reagents were added as follows, 1.00 mL of 5 % w/v gelatin (Merck, Germany), 5.00 mL of 1.0 mol/L acetate buffer, and 2.50 mL of 0.1 % w/w aluminon (Merck, Germany). The mixture was shaken well, calibrated to 50 mL by DIW, and let stand for 20 minutes. The UV-Vis measurement was carried out at 545 nm (UV-1800, Shimadzu, Japan) for quantification purposes.

2.3.2. The 8-hydroxyquinoline reagent assay

This colorimetric assay employed the 8-hydroxyquinoline reagent in combination with the liquid-liquid extraction, assisting in improving the selectivity for determining aluminum contents in rocks. In brief, the digested sample solution was pipetted to a 50-mL volumetric flask. After that, 5.00 mL of solution A (mixture of 10 % w/v hydroxylamine in DIW and 1 % w/w hydroxyquinoline in 2.5 % v/v acetic acid (Merck, Germany), volume ratio of 1:4) and 5.00 mL of solution B (dissolving 8.2 g of sodium acetate and 0.20 g of 1,10-phenanthroline in 2.5 % v/v acetic acid) were added. The mixture was gently shaken and let stand for 15 minutes. Then, the sample solution was added 5.00 mL of n-butyl acetate, thoroughly shaken for 30 second, and centrifuged at 6000 rpm for 5 minutes. The n-butyl acetate phase was collected and measured its UV-Vis absorbance at 396 nm for quantification purposes.

For both colorimetric assays, the potentially interfering ions for the two assays could be Fe³⁺, Si⁴⁺,

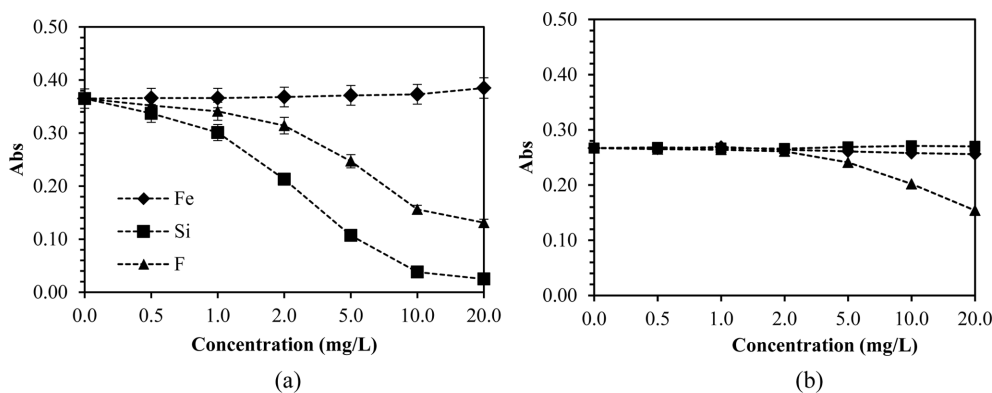


Fig. 2. Effects of potentially interfering ions of Fe^{3+} , Si^{4+} and F^- in the colorimetric assays using (a) aluminon and (b) 8-hydroxyquinoline reagents (error bar: standard deviation (SD) regarding three replicates).

and F^- , which are present in high contents in the rock sample solutions after the acid digestion. Therefore, the effects of these ions on the colorimetric procedure were investigated in the present study.

2.4. Determination of the aluminum content in rock samples

The quantification was conducted by establishing the linear calibration ($y = ax + b$), in which x and y stand for the standard concentrations (mg/L) and their absorbance values measured at the quantification wavelength, respectively. The limits of detection and quantification were calculated by the equations of $\text{LOD} = 3.3\text{SD}/a$ and $\text{LOQ} = 3\text{LOD}$, in which SD is the calculated standard deviation from the absorbance measurement of 10 blank samples (only containing the digestion and colorimetric reagents, without rock samples).

The proposed microwave-assisted acid digestion and UV-Vis measurement were applied to determine the aluminum content in real rock samples. Inductively coupled plasma mass spectrometry (ICP-MS) was used to compare with the results obtained from the UV-Vis. Spiked samples were used for recovery tests, in which the spiked Al concentration was equivalent to the estimated Al in each rock sample.

All the investigations were carried out in triplicates ($n = 3$) and expressed as mean value \pm standard deviation (SD).

3. Results and Discussion

3.1. Effects of Fe^{3+} , Si^{4+} and F^- as the potentially interfering ions on the colorimetry

In the present study, the spiked 1.00 mg/L Al^{3+} in DIW samples were used to investigate the effects of Fe^{3+} , Si^{4+} and F^- , ranging from 0 to 20.0 mg/L.

3.1.1. The aluminon reagent assay

Fig. 2(a) indicates that the influence of Fe^{3+} on the measured signals could be eliminated by the reducing agent of hydroxylamine,^{14,15} i.e., the Abs of 0.365 to 0.385 according to the rising Fe^{3+} content. Meanwhile, the interferences of Si^{4+} and F^- towards the measured signal were considerable, in which there was a remarkable drop in the measured Abs regarding the increased concentrations of these two ions, i.e., 0.365 to 0.025 and 0.365 to 0.131 for Si^{4+} and F^- , respectively. The reasons could be due to the complexation happened.¹⁶ For the digested rock sample solutions, due to the high content of Si^{4+} and F^- , it was disadvantageous to accurately determine the concentration of aluminum using the aluminon reagent assay.

3.1.2. The 8-hydroxyquinoline reagent assay

It is similar to the aluminon reagent assay, the interferences of Fe^{3+} could be eliminated by the addition of hydroxylamine in the reaction solution,^{14,15} i.e., only a slight Abs decrease was observed (0.267 to 0.256) (Fig. 2(b)). Moreover, it is highly noticeable that the

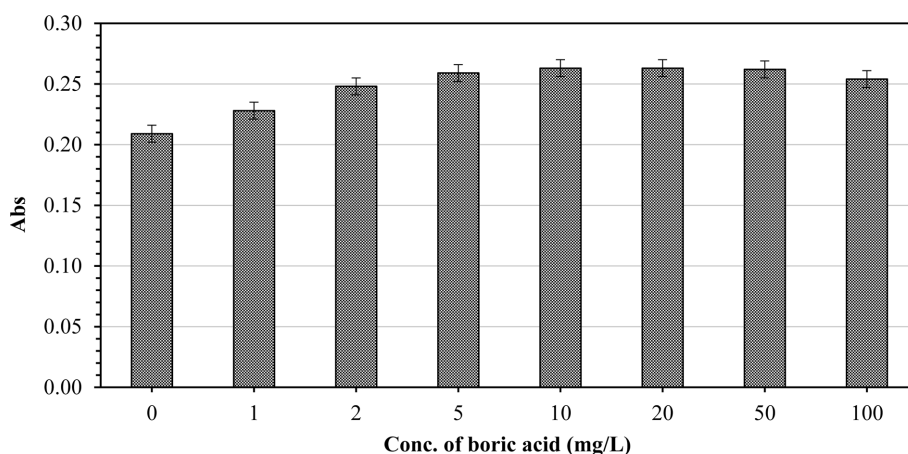


Fig. 3. Effects of boric acid concentrations on the elimination of F^- interference (error bar: standard deviation (SD) regarding three replicates).

effects of Si^{4+} on the Abs measurement were eliminated due to the application of the liquid extraction. In this procedure, the investigated Si^{4+} concentration was 20 times higher than that of aluminum in the context that Si is generally existed about 3 to 5 times higher than Al in real rocks, and the results demonstrated no effects on the Abs, ranging from 0.267 to 0.267.

For F^- , its effects were ignored in the concentration of lower than 2.00 mg/L; however, there was a drop in the measured Abs for the F^- of above 2.00 mg/L (0.261 to 0.154 regarding F^- of 2.00 mg/L and 20.0 mg/L). The explanation could be due to the formation of stable AlF_6^{3-} complex ($\log K = 21.4$, K is formation constant),¹⁷ especially in the presence of high F^- content compared to aluminum, e.g., in the present study, the concentration ratio $[F^-]/[Al^{3+}]$ of above 2, which resulted in the reduction in the formation of the desired complex between Al^{3+} and 8-hydroxyquinoline ($\log K = 5.81$).¹⁸ Therefore, further attempt was used to assist in the elimination of F^- interferences. In the 8-hydroxyquinoline, boric acid was added to the reaction solution to create the easily volatile BF_3 .¹⁹ In the present study, spiked samples containing 10.0 mg/L F^- and 1.00 Al^{3+} in DIW were used for the assessment of the effectiveness of boric acid in term of eliminating the interferences from F^- . Briefly, boric acid of different concentrations was added, and the reaction solution was heated to dryness. The residue was

dissolved by 1.0 mol/L nitric acid before the colorimetric procedure with 8-hydroxyquinoline. Fig. 3 demonstrate that the increase in boric acid resulted in the rising measured Abs from the initial of 0.209 (no boric acid), which reached the highest value (0.263) at 20 to 50 mg/L boric acid, then slightly decreased (0.254) at 100 mg/L boric acid. It could be concluded that the addition of boric acid effectively assisted in eliminating the interferences from F^- by competitively reacting with F^- to form BF_3 (easily volatile) instead of the formation of undesired complex of AlF_6^{3-} .

Compared to the aluminon reagent assay, the 8-hydroxyquinoline reagent assay with the help of boric acid could eliminate the effects from all three ions of Fe^{3+} , Si^{4+} and F^- on the colorimetry. Therefore, in the present study, the 8-hydroxyquinoline reagent assay would be applied for real rock samples after evaluating the analytical method performance.

3.2. Calibration curve

In the present study, linear relationship was applied to build up the calibration curve for quantification purposes (Fig. S1), in which the working range was from 0.10 mg/L to 3.00 mg/L. The goodness of linearity was obtained ($R^2 = 0.9996$). The regression equation, $y = 0.2136x + 0.0144$, was used for estimating the limits of detection and quantification in the later part.

3.3. Limit of detection and quantification

The limit of detection of the UV-Vis instrument was estimated by simultaneously analyzing 10 blanks employing the 8-hydroxyquinoline reagent assay. The absorbance signals of these blanks were recorded (see Table S1), then their standard deviation (SD) was calculated, in which $SD = 0.00190$.

$$LOD = 3 \times SD/a = 3.3 \times 0.00190/0.2136 = 0.029 \text{ mg/L}$$

$$LOQ = 3 \times LOD = 3 \times 0.029 = 0.087 \text{ mg/L}$$

The obtained LOD and LOQ are low, demonstrating favorable sensitivity for small rock sample weight used for the microwave-assisted acid digestion, which assures the microwave system safety due to the pressure during the operation and complete digestion of sample matrices.

3.4. Concentration of aluminum in rock samples, precision, and recoveries

Different acid mixtures could be applied in the microwave-assisted acid digestion, such as $\text{HNO}_3 + \text{HCl}$, $\text{HNO}_3 + \text{H}_2\text{O}_2$, $\text{HNO}_3 + \text{HF}$, etc. for various sample matrices. For rocks as a hard digested matrix, the composition mainly contains the inorganic minerals and oxides, two acid mixtures have been commonly used, i.e., $\text{HNO}_3 + \text{HCl}$ and $\text{HF} + \text{HNO}_3$.^{20,21} The first acid mixture employs the strong acidic property of HCl and the oxidation of HNO_3 to decompose several basic rock types, which typically compose of oxides and metallic salts. However, this acid mixture does

not completely digest the rock matrices, especially those with high content of silicon oxide. The combination of $\text{HF} + \text{HNO}_3$ is considered a better option to completely decompose the rock matrices due to the specific property of HF , which can dissolve SiO_2 , a main component of rocks (up to 50 % w/w for rock samples in the present study), besides the oxidation of HNO_3 employed for the digestion of other constituents. It should be noted that the HF residue must be eliminated in the sample solution after the digestion due to its potential to (i) erode the glass-containing labware and (ii) affect the colorimetric assay (forming the complex with aluminum). The HF residue elimination was carried out by the addition of boric acid to form the easily volatile BF_3 (discussed earlier).

For real rock samples, the relative standard deviation or RSD, i.e., ratio of standard deviation (SD) to mean

Table 2. Aluminum concentrations of rock samples

No.	Sample code	Aluminum content \pm SD (% w/w)		Recovery (%)	
		UV-Vis	ICP-MS	UV-Vis	ICP-MS
1	SR-1	5.4 ± 0.03	5.3 ± 0.03	97	102
2	SR-2	5.0 ± 0.06	4.8 ± 0.05	98	101
3	SR-3	5.9 ± 0.02	5.8 ± 0.02	101	103
4	DR-1	4.3 ± 0.04	-	102	-
5	DR-2	2.2 ± 0.04	-	101	-
6	DR-3	2.5 ± 0.02	-	99	-
7	DR-4	1.8 ± 0.03	-	98	-

“-”: not measure by ICP-MS; SD: standard deviation for three replicates (n = 3)

Table 1. Microwave pressure and temperature program for the digestion of the rock samples

Temp. (°C)	Pressure (bar)	Ramp (min)	Hold (min)	Power (W)	Description
150	20	20	25	800	The temperature increases quickly while the pressure rises slowly. After about 9 to 10 minutes, the power, temperature, and pressure are around 650 W, 110-120 °C, and 9-10 bar, respectively. The acids start to evaporate, and chemical reactions begin to strongly happen, resulting in quickly increased pressure. The instrument power reaches the set-up value of 800 W. The chemical reactions happen vigorously and produce a lot of NO_x gas, resulting in increased pressure (around 12-20 bar). The temperature is ~140-150 °C.
40	20	15	0	0	The power gains its stability. The temperature and pressure also reach the highest values as set-up (about 20 minutes). Therefore, after 20 minutes, the reaction system achieves the desired condition, which is hold for another 25 minutes to completely decompose the sample matrix.

value (expressed as percentages), was calculated for each sample to evaluate the precision (Table 2). The results of RSD were ranged from 0.34 % to 1.8 %, indicating favorable precision according to the¹² for the concentrations from 1 % to 10 %. Besides, the recoveries were acceptable for this concentration range. The results from the ICP-MS as a comparison (applied for surface rocks) were similar to those obtained from the UV-Vis measurement, which indicates the application potential of the proposed UV-Vis method. The proposed method provides more opportunities for many laboratories, especially geology or environment laboratories, to self-test the Al contents in rocks without the equipment of specific analytical instruments, e.g., ICP-MS or ICP-OES.

4. Conclusions

The present study proposed an analytical method for Al in rocks based on the colorimetric reactions with 8-hydroxyquinoline reagent and UV-Vis measurement after microwave-assisted acid digestion. The potentially interfering ions in the digested solutions, such as Fe³⁺, Si⁴⁺, and F⁻, were investigated to minimize their effects. The LOD and LOQ were estimated as 0.029 mg/L and 0.087 mg/L, respectively. The calibration curve was built based on the linear relationship between concentrations and their corresponding absorbance values at xxx nm, demonstrating the goodness of linearity ($R^2 = 0.9996$, 0.10 mg/L to 3.00 mg/L). Several rock samples were analyzed their Al contents by the 8-hydroxyquinoline method. The results were compared to ICP-MS as a reference, indicating favorable precision and no remarkable differences between the UV-Vis and ICP-MS. The present study emphasizes the application potential of UV-Vis to detect Al in rocks (after acid digestion) despite their rich-in-ion matrices.

Acknowledgements

The authors would like to thank the Faculty of Environmental and Food Engineering, Nguyen Tat Thanh University, Ho Chi Minh City (Vietnam) and

the Faculty of Chemistry, University of Science, Vietnam National University, Ho Chi Minh City (Vietnam) for their assistance and support during this study.

References

1. M. Pidwirny, 'Composition of rocks. Fundamentals of physical geography', 2nd Ed., Date Viewed, 2006.
2. S. V. Khromykh, P. D. Kotler, A. V. Kulikova, D. V. Semenova, K. R. Minnebaev, B. I. Gareev, G. A. Batalin, T. N. Antsiferova, E. A. Il'icheva, and A. S. Volosov, *Minerals*, **12**(1), 1-21 (2022).
3. Z. A. Alhawdar, 'Method Development for the Analysis of Aluminum, Calcium, Magnesium and Iron in Sedimentary Rocks' Master Degree Thesis, Pittsburg State University, Pittsburg, Kansas, 2014.
4. J. A. Haggerty, *Geophys. Monogr. Ser.*, **43**, 175-185 (1987).
5. V. Balaram, R. Mathur, M. Satyanarayanan, S. S. Sawant, P. Roy, K. S. V. Subramanyam, C. T. Kamala, K. V. Anjiah, S. L. Ramesh, and B. Dasaram, *Mapan-J. Metrol. Soc. India*, **27**, 87-95 (2012).
6. S. K. Pradhan and B. Ambade, *J. Anal. At. Spectrom.*, **35**, 1395-1404 (2020). <https://doi.org/10.1039/D0JA00190B>
7. P. Ildefonse, D. Cabaret, P. Saintavit, G. Calas, A. M. Flank, and P. Lagarde, *Phys. Chem. Miner.*, **25**, 112-121 (1998). <https://doi.org/10.1007/s002690050093>
8. M. H. Özkan and M. Akçay, *Turkish J. Chem.*, **26**, 711-724 (2002).
9. I. A. Alhagri and M. Albeshry, *J. Chem.*, **2023**, 2349838 (2023). <https://doi.org/10.1155/2023/2349838>
10. R. Camilleri, C. Stark, A. J. Vella, R. M. Harrison, and N. J. Aquilina, *Heliyon*, **9**, e12844 (2023). <https://doi.org/10.1016/j.heliyon.2023.e12844>
11. Z. Marczenko and M. Balcerzak, 'Separation, preconcentration and spectrophotometry in inorganic analysis', 1st Ed., Elsevier, 2000.
12. Appendix F of AOAC: Guidelines for Standard Method Performance Requirements (2016).
13. Environmental Protection Agency (EPA)-Method 3052: Microwave Assisted Acid Digestion Of Siliceous And Organically Based Matrices (2007).
14. G. Bengtsson, S. Fronæus, and L. Bengtsson-Kloo, *J. Chem. Soc. Dalton Trans.*, **2002**, 2548-2552 (2002). <https://doi.org/10.1039/C2JD00000A>

- doi.org/10.1039/B201602H
15. J. Zou, J. Ma, L. Chen, X. Li, Y. Guan, P. Xie, and C. Pan, *Environ. Sci. Technol.*, **47**, 11685-11691 (2013). <https://doi.org/10.1021/es4019145>
16. V. Malyshev, D. Shakhnin, A. Gab, I. Astrelin, L. Molotovska, V. Soare, C. Donath, E. I. Neacsu, V. Constantin, and A. M. Popescu, *Rev. Chim.*, **67**, 2490-2500 (2016)
17. J. Zolghamein, A. Shahrjerdi, G. Azimi, and J. Ghasemi, *Anal. Sci.*, **25**, 1249-1253 (2009). <https://doi.org/10.2116/analsci.25.1249>
18. Y. Zhao, Z. Lin, H. Liao, C. Duan, and Q. Meng, *Inorg. Chem. Commun.*, **9**, 966-968 (2006). <https://doi.org/10.1016/j.inoche.2006.05.030>
19. R. E. Zeebe and J. W. B. Rae, *Chem. Geol.*, **550**, 119693 (2020). <https://doi.org/10.1016/j.chemgeo.2020.119693>
20. D. E. Güven and G. Akinci, *Gazi Univ. J. Sci.*, **24**, 29-34 (2011).
21. C. J. Warren, B. Xing, and M. J. Dudas, *Can. J. Soil Sci.*, **70**, 617-620 (1990).

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