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IMPROVED PHOSPHORIC–NITRIC ACID DIGESTION METHOD FOR ACCURATE DETERMINATION OF TUNGSTEN IN SOIL MATRICES

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Abstract

Accurate determination of tungsten in soil matrices is essential for understanding its environmental behavior and mobility. Conventional acid digestion procedures often result in low tungsten recovery due to the formation of insoluble species under acidic conditions. This study evaluates a modified phosphoric–nitric acid digestion method designed to enhance tungsten solubilization through the formation of stable phosphotungstate complexes. The modified procedure was compared with standard nitric and nitric–hydrochloric acid methods using reference materials and test soils. Results demonstrate significantly improved recovery efficiency and acceptable matrix spike performance. The method also allows simultaneous determination of other metals, ensuring analytical reliability and broader environmental applicability.

Keywords

tungsten, acid digestion, phosphoric acid modification, soil analysis, ICP-AES, metal recovery

Introduction

In recent years, interest in tungsten geochemistry and its environmental behavior has significantly increased due to the expanding industrial, military, and civilian applications of tungsten-containing materials. Tungsten is widely used in carbide tools, ammunition, mining equipment, and various industrial products, which increases its potential release into environmental systems. Although metallic tungsten does not occur naturally, it is commonly present in the environment in the form of oxyanion species, primarily as tungstate (WO_4^{2-}), which is thermodynamically stable under most environmental conditions.

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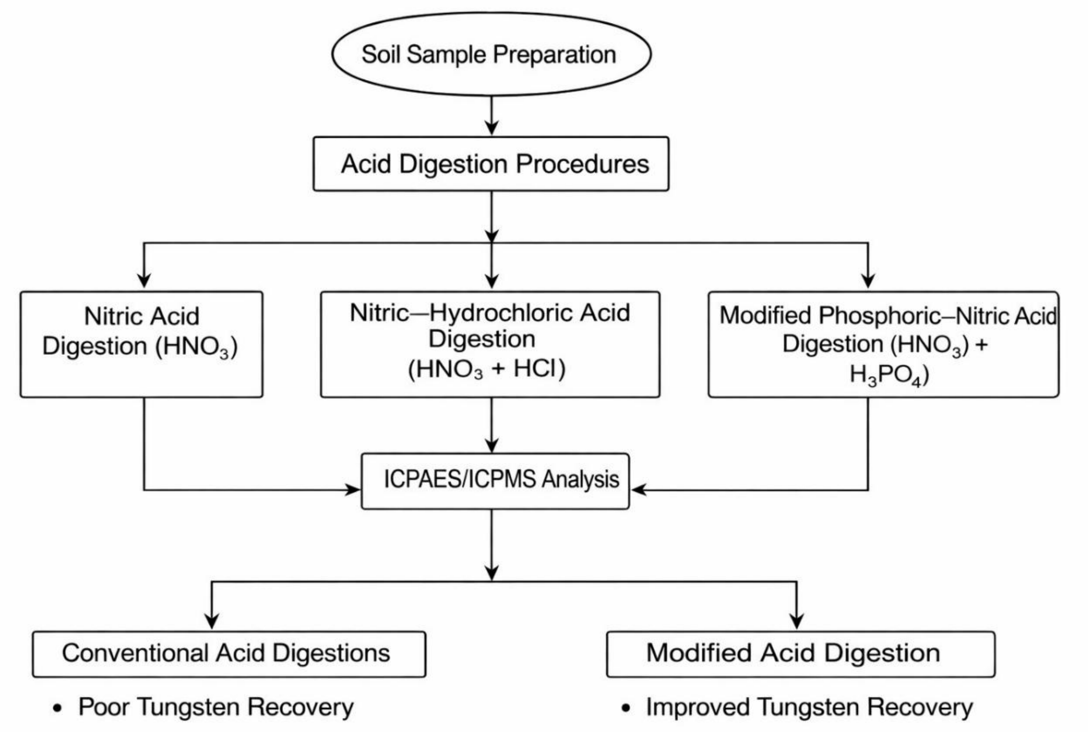

Figure 1. Schematic flow diagram of conventional and modified acid digestion procedures for tungsten extraction from soil samples

Unlike many trace metals that exist as cations, tungsten predominantly occurs as anionic species in soils and water, resulting in distinct geochemical behavior. Tungstate can polymerize and form poly- and heteropoly-tungstate species through interactions with other oxyanions such as molybdate, phosphate, and silicate, leading to complex speciation and variable mobility. Under acidic conditions, tungsten may form insoluble species or precipitates, while under alkaline conditions it tends to be more mobile, which complicates its environmental assessment and quantification.

Accurate determination of tungsten concentrations in soil matrices is essential for understanding its geochemical distribution, mobility, and potential environmental relevance. However, conventional acid digestion procedures commonly used for metal analysis, typically based on nitric and hydrochloric acids with hydrogen peroxide, often result in low tungsten recovery due to the formation of insoluble tungstic or polytungstic species during digestion. This analytical limitation can lead to significant underestimation of tungsten concentrations in environmental samples.

To overcome this challenge, modified digestion approaches that incorporate phosphoric acid have been developed to promote the formation of soluble phosphotungstate complexes and enhance extraction efficiency. Such modifications aim to improve tungsten recovery while maintaining compatibility with simultaneous determination of other metals using ICP-AES or ICP-MS techniques.

Therefore, this study focuses on improving tungsten extraction from soil matrices through a modified acid digestion procedure and evaluating its effectiveness compared to conventional methods, with emphasis on analytical accuracy and multi-element determination capability.

Methodology

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The methodology of this study was designed to evaluate and compare different acid digestion procedures for the effective extraction and quantification of tungsten in soil matrices. The experimental approach included soil sample preparation, application of three digestion protocols, instrumental analysis, and quality control assessment.

Soil Samples

Two certified standard reference materials and three test soils with different physicochemical characteristics were selected for the study. The test soils represented varying mineralogical compositions, including silty loam, iron-rich clay, and sandy soil. All soil samples were air-dried, homogenized, ground, and sieved to ensure uniform particle size distribution prior to digestion.

Acid Digestion Procedures

Three acid digestion procedures were evaluated:

Nitric Acid Digestion. A standard acid digestion method using nitric acid and hydrogen peroxide was applied. Approximately 0.5 g of dried soil was digested with diluted nitric acid under controlled heating conditions, followed by sequential additions of concentrated nitric acid and hydrogen peroxide to oxidize organic matter and solubilize acid-extractable metals.

Nitric–Hydrochloric Acid Digestion. A modified standard method was conducted by adding concentrated hydrochloric acid after nitric acid digestion to enhance the solubility of certain metal species. This procedure aimed to evaluate whether chloride complexation improves tungsten recovery.

Phosphoric–Nitric Acid Digestion (Modified Procedure) The third method involved the addition of concentrated phosphoric acid to the initial nitric acid digestion mixture. The purpose of this modification was to promote the formation of soluble phosphotungstate complexes, thereby preventing precipitation of insoluble tungstic species under acidic conditions. The digestion was performed under controlled heating, followed by hydrogen peroxide treatment to ensure complete oxidation.

After digestion, all samples were cooled, filtered, and diluted to a fixed volume with dilute nitric acid prior to instrumental analysis.

Instrumental Analysis

Metal concentrations, including tungsten, were determined using Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). For samples with low tungsten concentrations, Inductively Coupled Plasma Mass Spectrometry (ICP-MS) was used to improve detection sensitivity. Calibration standards were prepared using traceable multi-element solutions, and internal standards were applied to correct for instrumental drift.

Quality Control and Data Evaluation

Quality control measures included digestion blanks, blank spikes, matrix spikes, and analysis of standard reference materials. Recovery percentages were calculated by comparing measured concentrations with known reference values. Matrix spike recoveries were used to evaluate extraction efficiency and potential interferences within different soil matrices.

Comparative analysis of the three digestion procedures was performed based on tungsten recovery efficiency, reproducibility, and compatibility with simultaneous determination of other metals. Statistical comparison of mean values and standard deviations was conducted to assess analytical performance and method reliability.

This methodological framework enabled a comprehensive evaluation of the modified phosphoric–nitric acid digestion procedure relative to conventional acid digestion methods for accurate determination of tungsten in soil samples.

Results

The comparative evaluation of the three acid digestion procedures demonstrated significant differences in tungsten recovery efficiency across all tested soil matrices. The standard nitric acid digestion method produced consistently low tungsten recoveries, generally below 50%, indicating incomplete solubilization of tungsten species. The addition of hydrochloric acid slightly improved extraction efficiency; however, recoveries remained moderate and highly dependent on soil composition.

Table 1. Comparative tungsten recovery by different digestion methods

Method	**Reference Soil Recovery (%)**	**Test Soil Range (mg/kg)**	**Spike Recovery (%)**
Nitric Acid	29–39	90–440	0–47
Nitric–Hydrochloric Acid	54	190–737	3–56
Phosphoric–Nitric Acid	86	483–1695	76–98

In contrast, the modified phosphoric–nitric acid digestion procedure showed substantially higher tungsten recoveries in both standard reference materials and test soils. Recovery values reached up to 80–98% in spiked samples, demonstrating improved solubilization of tungsten through the formation of soluble phosphotungstate complexes. The modified method minimized the formation of insoluble precipitates that were observed in the conventional acid digestion procedures, thereby preventing tungsten loss during filtration.

Table 2. Comparative Extraction of Other Metals by Different Digestion Methods

Element	**Nitric Acid**	**Nitric Acid**	**Nitric–HCl**	**Nitric–HCl**	**Phosphoric–Nitric**	**Phosphoric–Nitric**
Al	70	80	71	78	116	109
Ba	86	81	86	78	94	82
Co	90	101	93	100	105	102
Cr	75	81	77	80	100	106
Cu	75	91	83	92	89	99
Fe	86	87	86	86	98	100
Mn	88	92	89	87	93	94
Ni	83	97	83	95	91	117
Pb	NA	90	NA	86	NA	87
Zn	83	85	86	80	91	87

For standard reference materials, the phosphoric–nitric method yielded recoveries significantly closer to reported reference concentrations compared to the two standard methods. Although the method was not designed as a total dissolution technique, it achieved acceptable extraction efficiency for environmentally relevant tungsten fractions.

In the three test soils, tungsten concentrations determined using the modified procedure were markedly higher than those obtained by conventional digestion methods. The greatest improvement was observed in sandy and iron-rich soils, where standard methods exhibited particularly poor matrix spike recoveries. The modified method consistently produced acceptable matrix spike recoveries, generally within the 75–98% range, confirming improved analytical reliability.

In addition to tungsten, the determination of other metals showed comparable results among the three digestion procedures. For most major and trace elements, recoveries remained within acceptable analytical limits, indicating that the addition of phosphoric acid did not negatively affect multi-element analysis. In some cases, slightly enhanced extraction efficiencies were observed for certain elements, suggesting improved digestion performance.

Conclusion

The results of this study demonstrate that the modified phosphoric–nitric acid digestion procedure significantly improves tungsten extraction from soil matrices compared to conventional nitric and nitric–hydrochloric acid methods. The enhanced recovery is attributed to the formation of soluble phosphotungstate complexes, which prevent precipitation of insoluble tungsten species during digestion. The modified method consistently achieved higher recovery rates and improved matrix spike performance across different soil types, while maintaining reliable determination of other metals. Therefore, the phosphoric–nitric acid digestion procedure provides a more accurate and analytically robust approach for tungsten quantification in environmental soil samples.

References

1. Shen, L.; Li, X.; Lindberg, D.; Taskinen, P. Tungsten extractive metallurgy: A review of processes and their challenges for sustainability. *Minerals Engineering*, 2019, 142, 105934.
2. Han, Z.; Golev, A.; Edraki, M. A review of tungsten resources and potential extraction from mine waste. *Minerals*, 2021, 11(7), 701.
3. Liu, H.; Liu, H.; Nie, C.; Zhang, J.; Steenari, B.M.; Ekberg, C. Comprehensive treatments of tungsten slags: A critical review. *Journal of Environmental Management*, 2020, 270, 110927.
4. Dvořáček, J.; Sousedíková, R.; Vrátný, T.; Jureková, Z. Global tungsten demand and supply forecast. *Archives of Mining Sciences*, 2017, 62(1), 3–12.
5. Koutsospyros, A.; Braida, W.; Christodoulatos, C.; Dermatas, D.; Strigul, N. A review of tungsten: From environmental obscurity to scrutiny. *Journal of Hazardous Materials*, 2006, 136(1), 1–19.
6. Calvo, G.; Valero, A.; Valero, A. How can strategic metals drive the economy? Tungsten and tin production during periods of war. *The Extractive Industries and Society*, 2019, 6(1), 8–14.
7. Абдисамиевич С.А., Мамарасулович Р.У. и Азаматугли К.О. (2025). РАЗРАБОТКА ТЕХНОЛОГИИ ПОЛУЧЕНИЯ ГЛИНОЗЕМИЯ ИЗ МЕСТНОГО СЫРЬЯ. *Sanoatda raqamli technologiyalar/Цифровые технологии в промышленности* , *3* (2), 105-111.
8. Grey, I.E.; Birch, W.D.; Bougerol, C.; Mills, S.J. Unit-cell intergrowth of pyrochlore and hexagonal tungsten bronze structures in secondary tungsten minerals. *Journal of Solid State Chemistry*, 2006, 179, 3860–3869.
9. Lassner, E.; Schubert, W.D. *Tungsten: Properties, Chemistry, Technology of the Element, Alloys, and Chemical Compounds*. Springer, Boston, 1999.
10. Хужакулов, Н. Б., Рузиев, У. М., & Насирова, Н. Р. (2021). ИССЛЕДОВАНИЯ ВЛИЯНИЯ КАЧЕСТВА БИОКЕКА НА ПОКАЗАТЕЛИ СОРБЦИОННОГО ВЫЩЕЛАЧИВАНИЯ. *Universum: технические науки*, (5-2 (86)), 20-23.

11. 4U.S. Geological Survey. *Mineral Commodity Summaries: Tungsten*. Reston, VA, 2024.
12. Martins, F.; Castro, H. Significance ranking method applied to critical raw materials in a circular economy. *Procedia CIRP*, 2019, 84, 1059–1062.