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Atomic Absorption Spectroscopic Method for the Determination of Sediments and Core Samples of Gold Collected from Erigi Gold Mining Site Minna, Niger State

Umar Isah Ibrahim[1]; Odukunle Abdulazeez Olatunji[2]; & Shamsuddeen Mohammed[3]

[1]Department of Pure and Industrial Chemistry, Bayero University, Kano, Nigeria. [2]Department of Integrated Science, Federal College of Education Ilawe-Ekiti, Ekiti State, Nigeria. [3]Department of Chemistry, Aminu Saleh College of Education Azare, Bauchi, Nigeria.

Correspondence author: umarisah239@gmail.com

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Abstract

The ease and speed at which precise and accurate determinations can be made with this technique have made atomic absorption spectroscopy one of the most popular methods for the determination of metals. In this paper sediment and core samples of gold were quantified in ppm using AAS machine. 10g of the crushed and pulverized sediment and core samples were digested using aqua regia (3/1) (v/v) of concentrated HCl and HNO_3 respectively and heated in an electric hot plate at 200 ^{0}C for 45 minutes to have a complete digestion process. The digested samples were allowed to cool and polyacrylamides (flocculant) is added and then filtered in 250mL volumetric flasks. 5mL of Di-Isobutyl ketone (DIBK) mixed with methyl trioctyl ammonium chloride (aliquat 336) in 1% volume by volume solution to bring out gold extracts. Standards of 0.1, 0.5, 2, 5 and 10 ppm were prepared for calibration and DIBK as blank sample. The gold extracts of the samples were analyzed and the results obtained showed different appreciated concentrations which were then compared with the concentrations of the certified reference materials (CRM) and quality controls (QC) of the gold samples being analyzed at the same time for validation.

Keywords: Atomic Absorption Spectroscopy, Aqua Regia, Core Samples, Sediment Samples, Gold Determination

Introduction

The quality of analytical results is essential across many scientific disciplines that rely on chemical measurements. In mining and geochemical analysis, the transparency and reliability of these results are critical, particularly when determining precious metal content. Accurate measurements with clear traceability are necessary for informed decisions in exploration and valuation (Zhang et al., 2019).

Instrumental methods play a key role in quantifying gold content. Techniques such as inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma atomic emission spectrometry (ICP-AES), neutron activation analysis (NAA), atomic absorption spectrophotometry (AAS), and graphite furnace atomic absorption spectrometry (GF-AAS) have been widely applied (Nor et al., 2019; Fouad et al., 2015; Singh, 2012). These instruments are typically found in advanced laboratories due to their cost and required expertise.

In addition, some of these techniques were combined with fire assay (FA) as a concentration pre-processing step in order to increase their sensibility and decrease their detection and quantification limits (Battaini et al., 2014). These modern analytical technologies have many additional advantages such as their high productivity and low detection and quantification limits, etc. However, the high costs of these types of equipment, their maintenance costs and the necessary assayer's skills can make them inaccessible for many laboratories (Singh, 2012).

To enhance sensitivity and lower detection limits, some of these methods are combined with fire assay (FA) as a pre-concentration step. This integration improves precision, especially for trace-level gold (ISO, 2020). Despite their advantages, the cost of equipment, maintenance, and skilled labor can make such methods inaccessible for many field-based or resource-limited laboratories (Marsden & House, 2006).

Among available methods, atomic absorption spectroscopy (AAS) remains a practical and economical option. It offers good precision, especially when paired with digestion techniques like aqua regia and extraction methods like DIBK/Aliquat 336. UV/VIS spectrophotometry has also been explored for gold analysis due to gold's ability to form colored complexes with halides and other reagents, allowing for colorimetric detection (Singh et al., 2013).

Also, the limits of determination (LOD) and quantification (LOQ) obtained with the UV/VIS methods are relatively low. Modern UV/VIS spectrophotometers have auto samplers indicating they can test a large number of samples in a short period

of time. In addition, other authors reported the gold content determination in different types of samples using the colorimetric method (Tang et al., 2004).

EXPERIMENTAL

Chemicals

All chemicals used in the experiment are of analytical grade. Hydrochloric acid, Nitric acid, DIBK, methyl trioctyl ammonium chloride, polyacrylamide, ortho phosphoric acid, water from reverse osmosis.

Materials

Filter paper, volumetric flasks (250 mL), measuring cylinders ($10cm^3$ and $100cm^3$), pipettes (1, 2, 5, 10 mL), crucibles, electric hot plates and glass rod. Three certified reference materials (CRM) obtained from Australia: G-317-1 (concentration range 0.45 – 0.65ppm) G-920-6 (concentration range 5.01-5.74ppm) and G-320-3 (concentration range 0.99 – 1.01ppm).

Instrumentation

AAS Agilent technology 200 series AA model 240FS AA. Measurement time 1.5s, Pre-Read Delay 2s, Flame type: Acetylene/Air, Air flow 13.50lL/min, Acetylene flow 2.00L/min, wavelength 242.8nm, slit width 1nm, instrument detection limit 0.0100ppm, Nebulizer uptake rate 6mL/min, background correction (BC On)

Procedure

Preparation of Samples

Preparation of Core Samples

The core samples were washed and cut into smaller pieces and then dried in an oven at 200 ^{0}C for 3 hours. The dried samples were crushed and pulverized into powder and labeled according to each sample code. The samples are coded as BH24-01, BH24-02, BH24-03, BH24-04, BH24-05, BH24-06, BH24-07, BH24-08, BH24-09, BH24-10.

Preparation of Sediment Samples

The sediment samples were dried in an oven at 200 ^{0}C for 3 hours. The dried samples were crushed and pulverized into powder and labeled according to each sample code. The sediment samples are coded as SD24-01, SD24-02, SD24-03, SD24-04, SD24-05, SD24-06(1), SD24-06(2), SD24-07, SD24-08, SD24-09 and SD24-10.

Samples Digestion Method

10g of pulverized samples were weighed and transferred to clean beakers, which was labeled according to the sample code. Aqua regia method was used for the digestion of

the samples. 30 mL of concentrated HCl and 10 mL of concentrated HNO_3 were added to each sample. The samples were heated in electric hot plates for 1 hour at 200 ^{0}C for complete digestion. Little amount of polyacrylamide (flocculant) is added to the digested samples before filtration. The samples were filtered in 250 mL volumetric flasks and rinse severally with water. 5mL of DIBK mixed with aliquat were added each filtered sample and shook for 5 minutes to bring out the gold extract for analysis. Water is added above the mark to see the extracts. The gold content, in parts per million, of the sample is equal to:

$$= concentration\ (ppm)\ \times\ ppm\ DIBK\ \times\ 1000\ \div 10$$

Preparation of DIBK

1000mL of DIBK was transferred into amber reagent bottle and 10mL of methyl trioctyl ammonium chloride (aliquat 336) was added to make 1% volume by volume and the mixture was stirred using glass rod. The prepared DIBK was used as blank sample.

Preparation of Standards

1mL of pure gold solution was taken and placed in 100 mL volumetric flask and 10 mL concentrated HCl was added with little shake. Water was added to the mark to form a stock of 10,000 ppm, and another five different concentrations were prepared from the stock solutions: 0.1, 0.5, 2, 5 and 10 ppm. 10 mL of DIBK was added to each prepared five different concentrations to bring out the gold extract.

AAS Measurements and Calibration

Absorbance measurements were made at the 242.8 nm wavelength using D2 background correction. The operating parameters were optimized to get maximum absorbance for gold in the working range. The nebulizer spray chamber reservoir was filled with water before starting the analysis. The calibration was performed using the prepared standards and is presented in fig. 1.

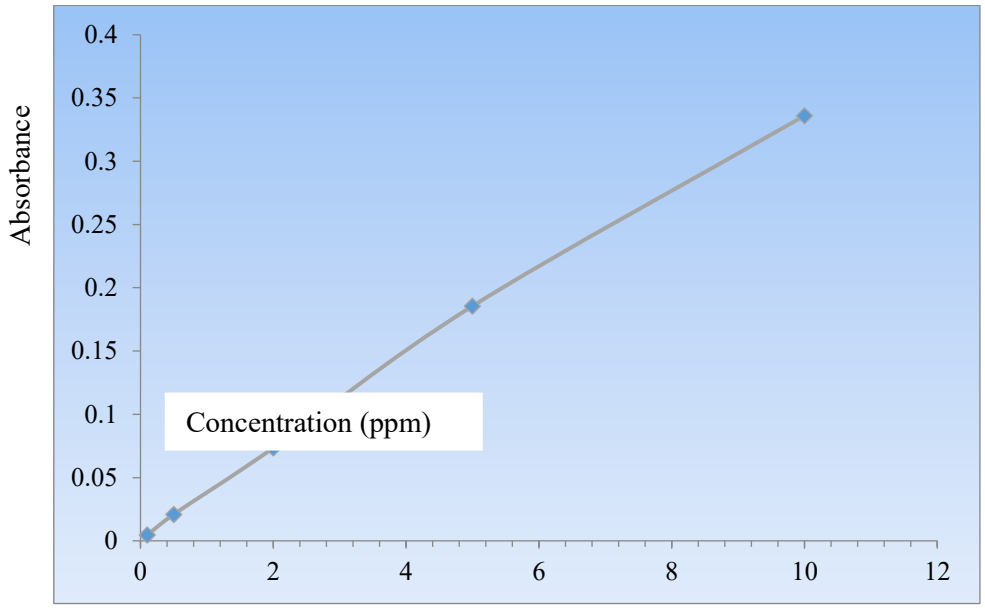


Fig. 1: Calibration Curve

RESULTS AND DISCUSSION

Sample Digestion

Aqua regia (HCl: HNO3, 3:1) attacks are the oxidizing acid leaches that are in common use for the extraction of Au from geological materials: However, there have been several published reports [15–18] that aqua regia attack is virtually completely efficient in solubilizing Au from geological samples, particularly when the attack is made after roasting the sample at 650 ^{o}C.

DIBK Extraction Method

The organic medium in the extraction of gold consists of Aliquat 336 in DIBK. Aliquat 336 is a long chain in quaternary amine (methyl trioctyl ammonium chloride). Aliquat 336 forms a stable complex with gold and 100 % of gold content will be extracted into the organic phase The gold content of the organic phase can be accurately determined by atomic absorption spectrometry.

Comparison between the Gold Samples, QC Samples and CRM Samples

The analytical results for international gold certified reference materials (CRM) measured by the proposed method were in close agreement with the QC and both the core samples and sediment samples of gold. Table 1 shows their concentrations in ppm.

CONCLUSION

The developed method using aqua regia digestion followed by DIBK extraction and flame atomic absorption spectrometry was shown to be effective and reproducible for determining gold content in geological samples. The results were consistent with CRM and QC samples, supporting the accuracy of the method. Compared to more complex or cost-prohibitive approaches like ICP-MS or NAA, this approach offers a viable alternative for laboratories with limited access to high-end instrumentation. Its reliability, relatively low cost, and compatibility with field workflows make it well-suited for routine gold analysis in exploration settings.

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REFERENCES

Caporali, S., Fossati, A., Lavacchi, A., & Perra, A. (2010). Potentiometric titration of gold in alloys as an alternative to fire assay. Gold Bulletin, 43(2), 122–130. https://doi.org/10.1007/BF03214977

Fouad, H. K., Elrakaiby, R. M., & Hashim, M. D. (2015). The application of flame atomic absorption spectrometry for gold determination in some of its bearing rocks. American Journal of Analytical Chemistry, 6(5), 411–421. https://doi.org/10.4236/ajac.2015.65040

International Organization for Standardization. (2020). ISO 15093:2020 - Jewellery and precious metals — Determination of high purity gold, platinum and palladium — Difference method using ICP-OES. https://www.iso.org/standard/69794.html

Marsden, J., & House, I. (2006). The chemistry of gold extraction (2nd ed.). Society for Mining, Metallurgy, and Exploration.

Nor, F. M., Tamuri, A. R., & Ismail, A. K. (2019). Non-destructive method for gold purity measurement to detect fake gold. International Journal of Engineering and Technology, 8(11), 165–172. https://doi.org/10.14419/ijet.v8i11.29371

Singh, N. (2012). A rugged, precise and accurate new gravimetry method for the determination of gold: An alternative to fire assay method. SpringerPlus, 1(1), 14. https://doi.org/10.1186/2193-1801-1-14

Singh, N., Bansal, A., & Shukla, S. K. (2013). Determination of purity of high-purity lead by conventional and electro-gravimetric methods: Estimation of measurement uncertainty. Chemistry Central Journal, 7, 108. https://doi.org/10.1186/1752-153X-7-108

Zhang, Y., Zhang, S., Zhao, Y., & Li, Y. (2019). Preconcentration and determination of ultra-trace gold in geological samples: A review. Molecules, 24(9), 1778. https://doi.org/10.3390/molecules24091778

APPENDIX:

Table 1: Comparison between the Gold Samples, QC Samples and CRM Samples

Core Samples	Concentration(ppm)	Sediment Samples	Concentration(ppm)	QC	Concentration(ppm)	CRM	Concentration(ppm)
BH24-01	0.34	SD24-01	0.09	GN01	1.45	G317	0.46
BH24-02	0.24	SD24-02	0.025	GN02	0.15	G920	5.30
BH24-03	0.14	SD24-03	0.21	GN03	0.48	G320	0.49
BH24-04	0.36	SD24-04	0.42	GN04	5.19		
BH24-05	0.37	SD24-05	0.53				
BH24-06(1)	5.04	SD24-06(1)	0.195				
BH24-06(2)	5.74	SD24-06(2)	0.165				
BH24-07	1.07	SD24-07	0.215				
BH24-08	0.5	SD24-08	0.12				
BH24-09	0.46	SD24-09	0.005				
BH24-10	-0.01	SD24-10	-0.015				