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Minimizing Matrix Interferences in the Analytical Determination of Gold

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Abstract

In the effort to minimize the interference in gold analysis, standard addition, solvent extraction and Fe (III) precipitation were conducted for samples digested with aqua regia at approximately 80°C. Standard addition was unsuitable to be used to obtain the accurate concentration for gold because the solution was depressed by the matrix in the aliquot sample. Solvent extraction and Fe (III) precipitation with addition of NH₃ assisted in minimizing the interference by eliminating Fe and other gangue minerals. Before solvent extraction and Fe (III) precipitation, the FAAS gold analysis result for HG sample was 3.18 g/t and gold was not detected for LG sample. After solvent extraction and Fe (III) precipitation, the gold readings for HG sample were enhanced to 3.88 g/t and 3.90 g/t respectively. Whereas, the gold in LG sample were detected with 0.54 g/t and 0.65 g/t by using solvent extraction and Fe (III) precipitation correspondingly. Fe (III) precipitation was found to be the suitable method in minimizing the interference from gangue minerals which suppressed the gold reading.

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1. Introduction

When developing or choosing an analytical method, the selectivity of the method is important. Selectivity refers to the ability of the method to determine the analyte without interference from other substances present in the analyte matrix.^{1,2} The major specie believed to interfere with the accuracy of gold analysis is Fe.^{3,4,5}

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Interferences can be avoided by the correct choice of standards and conditions for a particular analysis which is termed as matrix matching of standard.^{6,7} Matrix effects coming from other constituents of complex samples usually lead to the declining of sensitivity and detectability of the methods used.⁸When the matrix is highly complex, the method of standard addition would probably be preferred. The advantages of standard addition methods over normal standardization techniques are the bias and flaws can be detected in this method and better precision will be obtained with multiple additions of inclining concentration added to the sample rather than replicates of a single addition.⁹

Direct determination of gold from geological sample is not possible due to severe spectroscopic interferences such as interfering gangue elements. Therefore, separation of gold from the gangue elements containing matrix prior to analysis is essential. In this research, the solvent extraction is conducted to purify the leached solution in order to minimize the interference¹⁰ caused by the gangue elements prior to gold content analysis.

Interferences from gangue elements especially Fe, could exist as sample of banded Fe formation at different facies (sulphides, oxides, silicate and carbonate) and quartz vein containing refractory.¹¹ Fe (III) precipitation using ammonia (NH₃), was selected because NH₃ system is inexpensive; require minimum equipment and labor requirement.¹² Bulut, et al. (2011)¹³ suggested that addition of NH₃ can mask the matrix effect of Fe. Three hydroxyl ions (Eq. 1) will react with Fe³⁺ to form a precipitate of Fe (III) hydroxide, Fe(OH)₃ with reference to Pourbaix diagram of Fe – H₂O system at 25°C (refer Fig.1). The overall chemical reaction equation is shown in Eq. 2. The masking agent such as NH₃, is suitable to reduce the Fe (III) content due to simple and rapid.

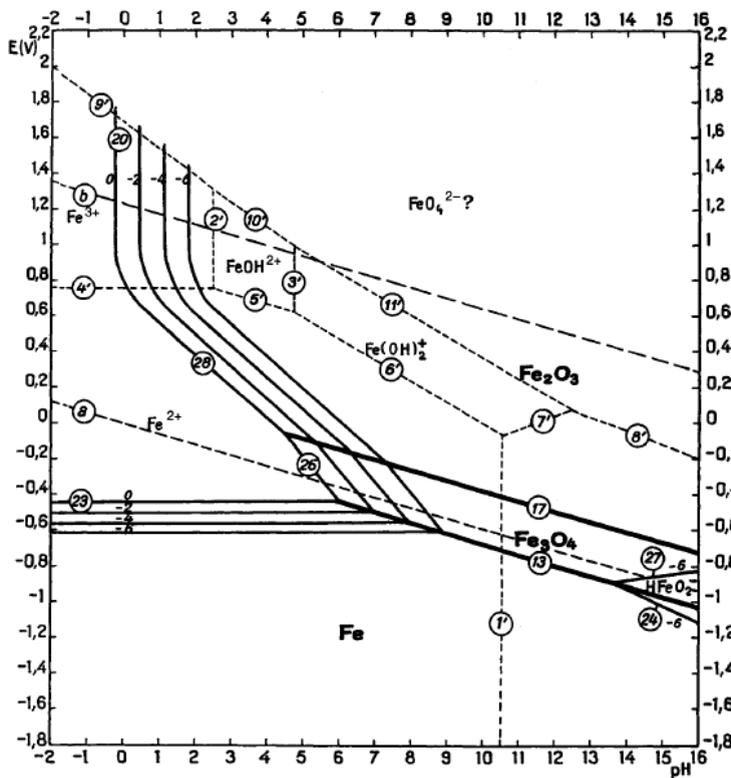


Fig. 1. Pourbaix diagram for the Fe – H₂O system at 25°C¹⁴.

2. Experimental

In this research, aqua regia leaching conducted in the condition of batch process within a few hours for high grade and low grade samples labeled as HG and LG respectively. The procedures of aqua regia leaching are almost similar to acid digestion. 60.0 g sample was digested in 300.0 ml aqua regia solution, heated to approximately 80°C for 7 hours. After agitation leaching in the reaction flask, the residues were removed and the solution was diluted to 500.0 ml by deionized water. Standard addition, solvent extraction and Fe (III) precipitation were carried out to reduce the matrix interference on gold analysis.

2.1. Standard Addition Method

The standard addition set sample was prepared by addition of known concentration of standard substance mixing together with unknown concentration of sample. This method is suitable for sample with complex background matrix. In this research, a ratio 1:1 of aliquot sample and standard solution were prepared for FAAS analysis. The absorbance result of mixed solution was re-plotted. If the linear line of standard addition is parallel to standard calibration line, there is no interference between aliquot sample and standard solution. Table 1 shows the amount of mixture volume of standard addition set.

Table 1. Standard solutions preparation for standard addition method

Set	Blank	1	2	3
Standard	25 ml sample + Deionized water	25 ml sample + Standard 1(1ppm)	25 ml sample + Standard 2 (2 ppm)	25 ml sample + Standard 3 (6 ppm)

2.2. Solvent Extraction

Solvent extraction using DIBK together with Aliquat 336 was conducted to extract the gold from diluted aurochloric acid into organic phase which followed by scrubbing of the organic phase using 7.0 M HCl acid for extracting the Fe into the aqueous phase.

2.3. Iron (III) Precipitation

20.0 ml aliquot of diluted chloroauric acid was mixed with 5.0 ml of ammonia, NH₃ and make up to 50 ml with 5% HCl. This mixture was shaken until reddish brown precipitate form. Then, the solution was left to settle down or filtration. The clear solution layer was formed at top. This clear solution was adjusted to pH 3-4 by HCl acid before FAAS analysis.

3. Results and Discussions

3.1. Standard Addition

Figure 2 and 3 shows that the matrix of HG and LG aliquot matched with the standard solution where the parallel line occurred. However, the absorbance reading of standard addition is different from the theoretical calculation (Table 2). This demonstrated that the standard addition solution was depressed by the matrix in the aliquot sample. Therefore, solvent extraction and Fe (III) precipitation were carried out to look into the most suitable method to minimize the matrix interference.

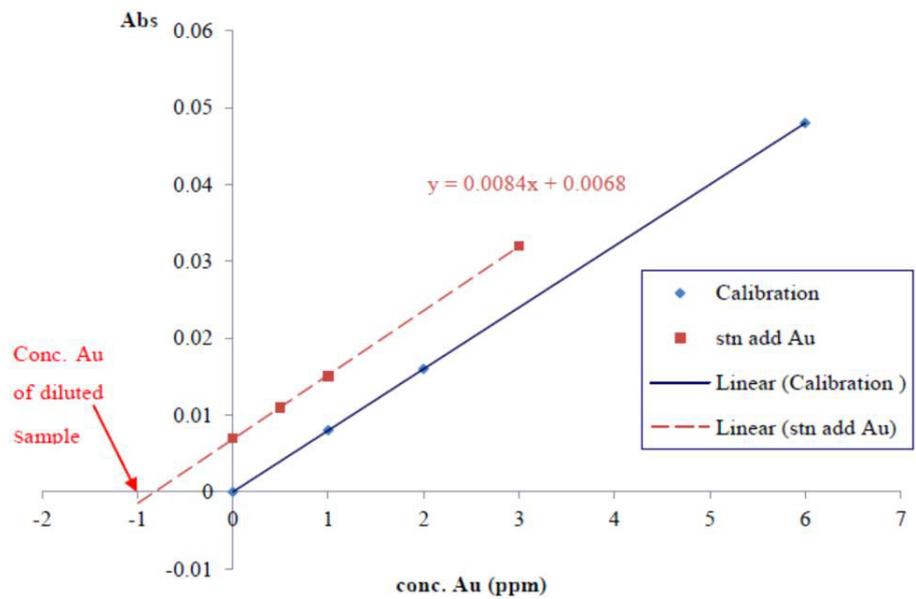


Fig. 2. Plot of Absorbance value against Au concentration from FAAS result for HG aliquot standard addition sets by aqua regia leaching.

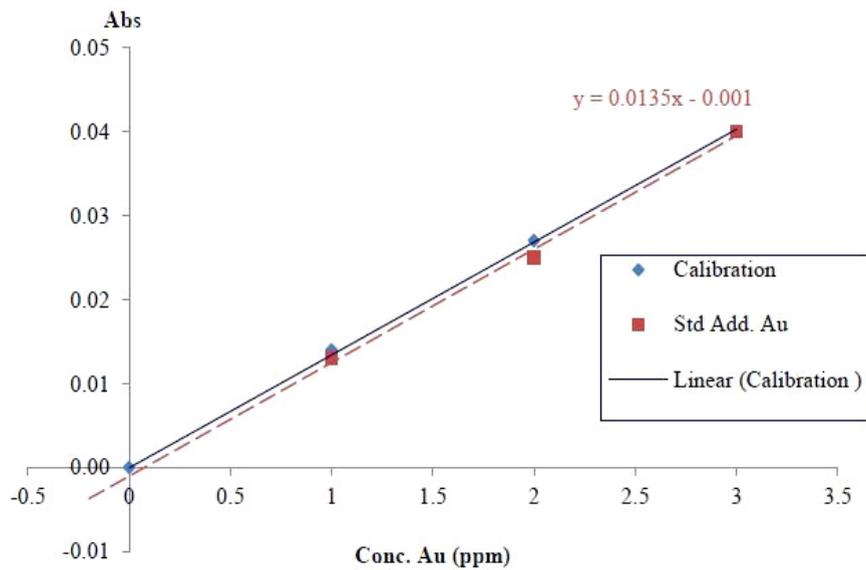


Fig. 3. Plot of Absorbance value against Au concentration from FAAS result for LG aliquot standard addition sets by aqua regia leaching.

Table 2. Absorbance reading of standard addition method for HG and LG sample.

Description	Abs reading	Theoretical abs reading
HG sample	0.008	-
*1 ppm Au + HG sample	0.015	0.016
LG sample	-	-
**1 ppm Au + LG sample	0.013	0.014 + x

3.2. Solvent Extraction and Fe (III) Precipitation

After solution purification to minimize the matrix interference, Au FAAS reading were enhanced as shown in Table 3. Before solvent extraction and Fe (III) precipitation, the FAAS gold analysis result for HG sample was 3.18 g/t and gold was not detected for LG sample. After solvent extraction and Fe (III) precipitation, the gold readings for HG sample were enhanced to 3.88 g/t and 3.90 g/t respectively. Gold was detected in LG sample with 0.54 g/t and 0.65 g/t by using solvent extraction and Fe (III) precipitation correspondingly. A little amount of Fe remained in the gold containing solution for HG and LG samples from the solvent extraction and Fe precipitation process. Large amount of Fe and gangue elements present in the ores were successfully precipitated out. Figure 4 shows SEM/EDX photomicrograph of precipitated residue from Fe (III) precipitation after filtration. The precipitates were reddish brown in colour which indicated the presence of Fe (III) hydroxide. The main composition of precipitated residue is iron oxide with some arsenic, aluminum and chlorine compound. It is believed that Fe was one of the gangue elements interfering with the Au signal, the Au (242.8 nm) and Fe (248.3 nm) are relatively close. Fe (III) precipitation was successful where from SEM/EDX residue analysis, Fe was detected.

Table 3. AAS results on Au and Fe content (g/t) extracted from the gold ores before and after solution purification processes.

Sample	Element	Elemental Concentration (g/t)		
		Before Purification	After Solvent Extraction	After Fe (III) Precipitation
HG	Au	3.180	3.883	3.900
	Fe	4111	14.24	2.95
LG	Au	not detectable	0.535	0.650
	Fe	5826	46.06	3.825

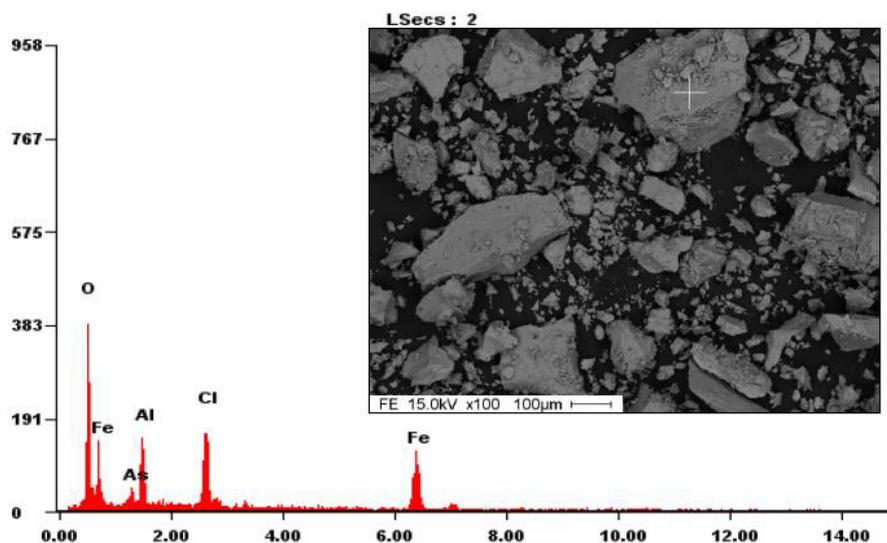


Fig. 4. SEM photomicrograph and EDX diffractogram of precipitated residue from Fe (III) precipitation.

4. Conclusions

Standard addition was not suitable to be used in obtaining the accurate concentration for gold because the solution was depressed by the matrix in the aliquot sample. Solvent extraction and Fe (III) precipitation by the addition of NH_3 assisted in purifying the gold bearing solution helps in minimizing the interference by eliminating Fe and other gangue minerals which affect the accuracy of the FAAS results obtained. Before solvent extraction and Fe (III) precipitation, the FAAS gold analysis result for HG sample was 3.18 g/t and gold was not detected for LG sample. After solvent extraction and Fe (III) precipitation, the gold readings for HG sample were enhanced to 3.88 g/t and 3.90 g/t respectively. Whereas, the gold in LG sample were detected with 0.54 g/t and 0.65 g/t by using solvent extraction and Fe (III) precipitation correspondingly. From the research work, Fe (III) precipitation was found to be the suitable method in minimizing the interference from gangue minerals which have suppressed the gold reading.

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