Fen Bilimleri Enstitüsü Dergisi, 21 (2009) 63-71
Marmara Üniversitesi

INTERFERENCE EFFECT OF FE ON THE SPECTROSCOPIC (FAAS) DETERMINATION OF AU AND AG IN THE ORE OF İZMİT REGION IN TURKEY

Halim AYRANCİ*, Ali Sadi BASAK, Hakan KARABULUT

Department of Inorganic Chemistry, Marmara University, TR-34722
Istanbul, Turkey

Alındığı Tarih: 17 Haziran 2008 *Kabul Tarihi: 26 Ekim 2009*

Summary: In this paper, interference and matrix effect of Fe on the spectroscopic (FAAS) determination of Au and Ag in the ore of İzmit Region in Turkey was investigated. Au and Ag contents of the samples were determined by the FAAS and Fe by the XRF and found to be about 12.7 % Fe. To determine the interference effect of Fe, standard solutions of $FeCl_3$ were added to 0.5-1 ppm standard solutions of Au increasingly and analyzed by FAAS. The minimum interference occured at 100 ppm Fe^{3+} in 0.5 ppm standard solution of Au and 500-2000 ppm Fe^{3+} in 1 ppm standard solution of Au on BC-ON (Background Correction-ON) mode and higher interference ranges were found on BC-OFF mode. As a result, increasing or decreasing the Fe^{3+} concentration increases the interference ranges remarkably. It has been concluded that the Fe^{3+} concentration should be optimized for the analysis of Au and Ag in the ore samples containing Fe as well.

Keywords: Interference, Analysis of Au, Digestion, FAAS

---

* E-posta: *hayranci@marmara.edu.tr* and Fax: +90 216 347 8783

INTRODUCTION

Presently XRD method is employed for qualitative analysis of ores[1] and XRF and AAS methods are used for the quantitative analysis. Atomic absorption spectrometry (AAS) is now probably the most preferred technique used for determination of metals in various samples. AAS determinations are usually made by flame-AAS (FAAS) when the concentration of the analyte is high enough or by graphite furnace (GFAAS) when the concentration is low[2,3].

The crushed and ground rock samples are calcined and then digested by microwave method using various mineral acids at suitable temperatures depending on the structures of the sample. The advantages of microwave method are high temperature, closed vessel, and acid digestion prepared samples in shorter time than other methods, less acid consumed and volatile elements available. In application, microwave sample preparation has been widely used for trace and ultra-trace metal analysis, such as atomic absorption spectrometry[4,5].

Matrix and interference effect must be minimized by studying on BC-ON (Background Correction) mode to obtain optimum results for metal determinations by AAS[6,7]. Because of the similarity of absorption wavelengths of Fe and Au (λ_{max} Au = 242.8 nm; λ_{max} Fe = 248.3 nm), in specific concentrations interference occurs even on BC-ON mode[6-8]. The interference effects of metals are minimum on BC-ON mode. Ag-because of the absorption wavelength value (λ_{max} (Ag) = 328.1 nm)-is not interfered with Fe and Au[8], on the contrary the matrix effect of other metals at specific concentrations occurs[9,10]. But this effect was minimized on BC-ON mode.

The rock samples which were scattered over the surface were collected. Fe content of the samples were determined by XRF[1-11] and Ag,Au contents by FAAS[12,13] and found to be containing about Fe 12.7 %, Ag 0.0631 %, Au 0.0855%. The minimum interference ranges based on Fe were established in Au and Ag analysis. It’s obvious that, the ores that contain high amounts of noble metals are convenient for exploiting for recovery of Au and Ag.

According to the analysis results, it's assigned that the metals except Au and Ag are not economically valuable[3].

EXPERIMENTAL

Equipment

Philips PW-2404 model with wavelength spectrum X-Ray Fluoresans Spectrometer (XRF), Varian SpectraAA-200 type Atomic Absorption Spectrometers (AAS), CEM MARS S type Closed Microwave Digestion System

Samples

Ore deposits of about 0.5-3 kg size were found scattered over 10,000 m^2 surface of mountainous area in the Izmit region of Turkey. About 150 kg of rocks of similar appearance and most abundantly found have been collected from nearly 500 m^2 of this area in polyethylene bags, which were then labeled.

Sample Preparation

The rock samples were ground in a ball-mill and followed by fine grinding in an agate mortar particle size of minus 150 mesh. In all stages during grinding, the samples were quartered and mixed thoroughly to ensure a homogeneous particle size distribution.

Standard Solutions

2500 ppm (2500µg mL^{-1}) of Fe^{3+} solution was prepared. Fe and Au standard solutions were prepared by using 2500 ppm of Fe^{3+} and 50 ppm of Au solutions.

Digestion Procedure

0.5030 g, 0.5037 g and 0.5036 g were weighed from the grounded sample respectively and labeled. 15 mL of aqua regia and 2.0 mL of H_2O_2 (%30) were added to first weight, 9.0 mL of HNO_3 (%65), 4.0 mL of HF (%40), 3.0 mL of HCl (%37), 1.5 mL of H_2O_2 (%30) and 30 mL of H_3BO_3 (saturated) were added to second weight, 9.0 mL of HNO_3 (%65), 4.0 mL of HF (%40), 3.0 mL of $HClO_4$ (%60), 1.5 mL of H_2O_2 (%30) and 30 mL of H_3BO_3 (saturated) were added to third weight respectively and digested in CEM MARS S type Closed Microwave Digestion System under following conditions;

Power (Watt)	Power %	Pressure (psi)	Temperature (0C)	Hold Time (min)	Ramp Time (min)
1200	100	180	210	20	30

After digestion procedure, sample 1, 2 and 3 were diluted to volume of 100 mL and analyzed by FAAS respectively[10]. Powder samples were analyzed by XRF.

RESULTS AND DISCUSSION

Various concentrations of Fe^{3+} solutions were added to standard solutions of Au and gold was determined by FAAS. As seen on Table 1 it has been concluded that the minimum interference occurs at the 100 ppm Fe^{3+} in 0.5-ppm standard solution of Au and of 500-2000 ppm Fe^{3+} in the 1 ppm standard solution of Au on BC-ON mode. On the other hand higher interference ranges were found at BC-OFF mode. The results that obtained from the determination of Au and Ag in the digested ore samples by FAAS on BC-ON and BC-OFF modes were shown in Table 2. Due to the high Fe content of the ore sample of İzmit region, accurate determination was carried out on BC-ON mode (Table 2). And also higher interference ranges were obtained on BC-OFF mode as shown in Table 2.

Ag determinations were carried out in three samples (N-1, N-2, N-3) by FAAS on BC-ON and BC-OFF modes. Matrix effect was minimum in N-1 on BC-ON mode (Table 2).

Table 1. Determination of Au in the Standard Solutions by FAAS at BC-ON and BC-OFF Mode (λ = 242.8 nm, L.C. = 4.0 mA)

Standard Solution	BC-ON Au Conc. (ppm)	BC-OFF Au Conc. (ppm)
0.5ppm Au + 50ppm Fe	0.523± 0.014	-
0.5ppm Au	0.513± 0.029	-
0.5ppm Au + 100ppm Fe	0.520± 0.025	0.560± 0.000
0.5ppm Au	0.520± 0.000	0.523± 0.014
0.5ppm Au + 200ppm Fe	0.527± 0.014	0.580± 0.000
0.5ppm Au	0.510± 0.000	0.523± 0.014
0.5ppm Au + 500ppm Fe	0.527± 0.014	0.623± 0.014
0.5ppm Au	0.533± 0.029	0.540± 0.000
0.5ppm Au + 1000ppm Fe	0.503± 0.014	0.717± 0.014
0.5ppm Au	0.480± 0.000	0.523± 0.014
0.5ppm Au + 2000ppm Fe	0.493± 0.029	0.880± 0.000
0.5ppm Au	0.470± 0.000	0.523± 0.014
0.2ppm Au + 50ppm Fe	0.230± 0.025	-
0.2ppm Au	0.210± 0.025	-
1.0ppm Au + 50ppm Fe	1.067± 0.038	-
1.0ppm Au	1.030± 0.052	-
1.0ppm Au + 500ppm Fe	1.030± 0.043	1.047± 0.014
1.0ppm Au	1.020± 0.014	0.963 0.014
1.0ppm Au + 1000ppm Fe	1.013± 0.038	1.133± 0.014
1.0ppm Au	1.020± 0.014	0.963± 0.014
1.0ppm Au + 2000ppm Fe	1.013± 0.057	1.293± 0.029
1.0ppm Au	1.020± 0.014	0.963± 0.014

Table 2 Determination of Au and Ag in the Digested Ore Samples by FAAS at BC-ON Mode and BC-OFF Mode (λ = 242.8 nm, L.C. = 4.0 mA)

Metal/Standard Solution	N-1 (ppm)	N-2 (ppm)	N-3 (ppm)
BC-ON			
Au	3.360± 0.043	4.307± 0.014	3.313± 0.062
2.0 ppm Standard Au	2.000± 0.000	2.083± 0.072	2.047± 0.100
Ag	3.175± 0.007	1.063± 0.017	0.104± 0.006
1.0 ppm Standard Ag	0.922± 0.125	0.972± 0.103	1.037± 0.094
BC-OFF			
Au	3.453± 0.014	4.727± 0.062	3.650± 0.124
5.0 ppm Standard Au	5.017± 0.072	5.110± 0.043	5.107± 0.574
Ag	3.249± 0.015	1.083± 0.012	0.110± 0.007
1.0 ppm Standard Ag	1.003± 0.060	0.993± 0.064	1.019± 0.054

N-1: 0.5030g Ore Sample+15ml Aqua regia+2ml H_2O_2/100ml

N-2: 0.5037g Ore Sample+9ml HNO_3+3ml HCl+4ml HF+1.5 ml H_2O_2+30 ml H_3BO_3/100ml

N-3: 0.5036g Ore Sample+9ml HNO_3+3ml $HClO_4$+4ml HF+1.5ml H_2O_2+30 ml H_3BO_3/100ml

Table 3 Mean Concentrations of Au in the Digested Ore Samples by FAAS at BC-ON Mode (λ = 242.8 nm, L.C. = 4.0 mA)

Sample No	Mean Au Conc. (ppm)	Mean Au Conc. (µg/g)
N-1	3.36	667.99
N-2	4.31	855.01
N-3	3.31	657.93

Table 4 Mean Concentrations of Au in the Digested Ore Samples by FAAS at BC-OFF Mode (λ = 242.8 nm, L.C. = 4.0 mA)

Sample No	Mean Au Conc. (ppm)	Mean Au Conc. (μg/g)
N-1	3.45	686.55
N-2	4.73	938.39
N-3	3.68	730.74

Table 5 Mean Concentrations of Ag in the Digested Ore Samples by FAAS at BC-ON Mode (λ = 328.1 nm, L.C. = 2.5 mA)

Sample No	Mean Ag Conc. (ppm)	Mean Ag Conc. (μg/g)
N-1	3.18	631.15
N-2	1.06	211.04
N-3	0.1	20.65

Table 6 Mean Concentrations of Ag in the Digested Ore Samples by FAAS at BC-OFF Mode (λ = 328.1 nm, L.C. = 2.5 mA)

Sample No	Mean Ag Conc. (ppm)	Mean Ag Conc. (μg/g)
N-1	3.25	645.19
N-2	1.08	215.08
N-3	0.11	21.98

CONCLUSION

Au determination was carried out on BC-ON and BC-OFF modes by adding various concentrations of Fe^{3+} solutions into the standard Au solutions according to find out minimum interference ranges based on Fe. %1-4 interference range has been found on BC-ON (Background Correction) mode, on the other hand higher interference ranges has been found on BC-OFF mode. Fe had not interfered with Ag on FAAS analysis. Here only the matrix effect has to be taken into account and this effect was minimized on BC-ON mode. In this study, it was concluded that interference effect of Fe should be definitely be minimized by using the instrument on BC-ON mode, so that the accurate Au analysis can be done by FAAS for Fe containing Au ores. To exploit an ore economically, its gold content should be not less than 3 ppm[14]. For this reason, recovery of Au and Ag from this ore would be convenient and very economical.

REFERENCES

[1] C.Ruiz, M.del; Rodriguez, M. H.; Perino, E.; Olsina, R.A.: *Mineral Engineering*, 15 (**2002**) 373.

[2] Canfranc, E.; Abarca, A.; Sierra, I.; Marina, M.L.: *J. Pharm. and Biomed. Anal.*, 25 (**2001**) 103-108.

[3] Lupu, R.; Nat, A.; Ene, A.: *Nuclear Instruments and Methods in Physics Research B,* 217 (**2004**) 123–135.

[4] Melaku, S.; Dams, R.; Moens, L.: *Analytica Chimica Acta,* 543 (**2005**) 117–123.

[5] Chen, Chieng-Ming; Chen, Mi; Peng, Yong-Wang; Lin, Cheng-Hsiung; Chang, Li-Wei; Chen, Chia-Fu: *Diamond & Related Materials,* 14 (**2005**) 798– 803.

[6] Ko´scielniak, P.; Kozak, M.: *Analytica Chimica Acta*, 438 (**2001**) 187–194.

[7] Ozcan, M.; Akman, S.: *Spectrochimica Acta Part B,* 55 (**2000**) 509-515.

[8] El-Gohary, Z.: *Journal of Quantitative Spectroscopy & Radiative Transfer* 75 (**2002**) 389–396.

[9] Grotti, M.; Leardi, R.; Gnecco, C.; Frache, R.: *Spectrochimica Acta Part B,* 54 (**1999**) 845-851.

[10] Gong, B.; Liu, Y.; Li, J.; Lin, T.: *Analytica Chimica Acta,* 362 (**1998**) 247-251.

[11] Muia, L.; Van Grieken, R.: *Anal. Chim. Acta*, 251 (**1991**) 177-181.

[12] Rowland, J.: *Varian Techtron Pt. Limited*, Springvale, Victoria, Australia, **1982**.

[13] Chakrapani, G.; Mahanta, P.; Murty, D.S.R.; Gomahy, B.: *Talanta*, 53 (**2001**) 1139-1147.

[14] Kirk-Othmer, *Encyclopedia of Chemical Technology*, 3rd Edition, Volume 11, A Wiley-Interscience Publication, New York, **1980**.