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# Determination of Au and Ag from iron ores combining FA and ICP/AES methods

## ABSTRACT

*In this paper, the combination of FA (Flame Analysis of Noble Metals - Cupellation) and ICP/AES (Atomic Emission Spectrometry with Induction Coupled Plasma) methods for determination of Au and Ag in geological samples of iron ore-magnetite was presented.Au and Ag were concentrated with Pb from PbO after desulphurisation and melting process ($Pb^{2+}$→Pb). Regulus (Pb with noble metals) is then cupellated. The resulting bead-pril after cupellation was dissolved in $HNO_3$ and then in HCl (imperial water: $2HNO_3$ and 6HCl). After the preparation of standard solutions and a blank test, the ICP/AES recording is performed. The obtained results werecompared with those obtained by the classical method of cupellation. The advantages of ICP/AES are excellent detection limits and linear dynamic range as well as a stable and repeating signal which is particularly important for samples of iron tested due to the low content of noble metals.*

***Keywords:*** *geological samples, FA, ICP / AES,noble metals.*

## 1. INTRODUCTION

Gold is found in small amounts in magnetite, pyrite and almost all ores silver, copper, beryllium, lead, zinc, telure and antimony. Due to the very heterogeneous composition of geological samples and the content of gold and silver in them no matter which method is used, their determination is carried out in two stages. First, it is necessary to separate the noble metals, Au and Ag from the sample of the ore by cupellation. After that Au and Ag are dissolved [1-5]. FA (flame analysis) of noble metals-cupellation is the oldest and very reliable method used for the determination of Au and Ag from metal ores [6, 7]. In addition to the determination of Au and Ag from geological-mining samples wide application have extraction AAS analysis methods. This paper presents the possibility of determining Au and Ag by combining FA and ICP/AES methods. The ICP/AES method uses induced coupled plasma for the formation of excited atoms and ions that emit electromagnetic radiation at wavelengths characteristic in this case for Au and Ag.

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The method provides the ability to determine the Au and Ag concentration of ppb [6-8]. By using this combined method, ppb detection values, linear dynamic range, low chemical interference and also a stable and repeating signal were determined [9,10]. In relation to our previous research, [1] in this paper for the FA/ICP AESpearl analysis imperial water ($2HNO_3$: 6HCl)was used.The amount of PbO in the stream was increased on (40g) in order to obtain a higher regulusand higher accuracy. The amount of fluxes was reduced on ($Na_2CO_3$(20g), borax (10g)).

## 2. EXPERIMENTAL PART

### *2.1. Preparation of sample for analysis by combining FA and ICP/AES method*

Ore's pieces were dried at 120°C for 60 minutes (before and after crushing) and then crushed on a mining crusher under a diameter of less than 3mm and then into a mill with discs below 100nm.20g of the solid sample of iron ore was measured and placed in a furnace at a temperature of 600°C for 2h to desulfurize. The sample is cooled and mixed in a shamotte cup with 40g PbO, 20g $Na_2CO_3$, 10g borax, 3g $SiO_2$ and 5g starch. The prepared contents are placed in a melting furnace at 1070 °C for 1 hour. After the melting process is completed, the contents are poured into gray oil molds where, after cooling, a slag-rusty

resin is formed. Formed Pb regulus with noble metals weighs 30-34 g [1,2]. The slag is mechanically detached and the regulus is cleaned from mechanical impurities by forging. The cleaned regulus is subjected to the bathing process in bathing cups in charcoal baths first by heating up to the intake and putting them in a regulated position and at a temperature of 950 ° C, continuing from 25-30 minutes by constant ventilation of the furnace. The bath containers adsorb part of the Pb, other part evaporates so that after cooling, the beads of precious metals Ag and Au remain.

Since each PbO contains certain quantities of Au and Ag, it is necessary to perform a blanktest sample- all except the sample.

*2.2. The dissolution process*

The bead is mechanically separated, purified as far as it is mechanically possible and subjected to the dissolution process by placing a bead of 150 ml glass and adding 2 $cm^3$ concentrated $HNO_3$ followed by 6 $cm^3$ of HCl (imperial water $2HNO_3$ and 6HCl) adding the acid to multiple portions to complete dissolution (at warm). After dissolution, the contents is transferred to a normal 100 ml aliquot and supplemented with 20% v/v HCl. Aim is to leave silver in the form of complex salts of $AgCl_2$ and $AgCl_3$ remaining in solution. The precipitation is filtered through a teflonic filter of 0.45. Before testing, a blank test is performed with all the acids except the sample.

All measurements were performed on ICP AES (Shimadzu 9820).

*2.3. Determination of gold*

Solutions for the calibration curve of gold were also prepared in two sets, the first (1; 2; 4; 6; 8 ppb, 1 ppm Au) and the other (01; 02; 05; 0.8; 1.0 ppm Au). Gold was recorded at a wavelength of 242.795nm

*2.4. Determination of silver*

The calibration curves were made in two sets, the first (0.1; 0.5; 1.0 ppm Ag) and the other (1; 2; 4; 6; 8; 10; 15.20 ppm Ag). A sample for a blank test and a dissolved sample were introduced into the system and the silver was recorded at an infrared length of 328,068 nm.

## 3. RESULTS AND DISCUSSION

Experimental studies were carried out by determining the precious metal content (Au and Ag) in geological samples of iron ore from previously prepared solutions. The results of the study were obtained by combining FA and ICP / AES method with FA method, under the same laboratory conditions. The FA (Flame Method for Determination of Noble Metal - Cupellation) is the oldest and most reliable method of determining precious metals from the ore but is a very long and expensive process. Due to the fact that gold is in nature in the form of quartz vessels, it is nonhomogeneously distributed in the iron ore. For this reason, a minimum sample size of 20g is used according to our own experience and experience of other authors. Table 1 gives an overview of gold and silver content in (g/t) for seven samples and the standard deviation (s) obtained after five measurements using the FA method. Table 2 gives an overview of gold and silver content in (g/t) obtained by the FA / ICP AES method combination.

*Table 1. Determination of gold and silver by cupellation (FA) [1]*

*Tabela 1. Određivanje zlata i srebra kupelacijom (FA) [1]*

| Sample | FA Au (g/t) | standard deviation (s) | FA Ag (g/t) | standard deviation (s) |
|---|---|---|---|---|
| 1 | 0,68 | 0,041 | 78,0 | 0,98 |
| 2 | 0,50 | 0,032 | 61,0 | 0,87 |
| 3 | 0,57 | 0,045 | 57,4 | 0,76 |
| 4 | 0,46 | 0,023 | 49,8 | 0,34 |
| 5 | 0,47 | 0,090 | 25,3 | 0,42 |
| 6 | 0,18 | 0,003 | 20,2 | 0,28 |
| 7 | 0,14 | 0,012 | 10,9 | 0,31 |

*Table 2. Determination of gold and silver combined method (FA and ICP / AES)*

*Tabela 2. Određivanje zlata i srebra kombinovanom metodom (FA i ICP/AES)*

| Sample: | FA/ICP AES Au (g/t) | standard deviation (s) | FA/ICP AES Ag (g/t) | standard deviation (s) |
|---|---|---|---|---|
| 1 | 0,69 | 0,032 | 79,0 | 0,72 |
| 2 | 0,50 | 0,024 | 62,0 | 0,62 |
| 3 | 0,59 | 0,031 | 58,0 | 0,44 |
| 4 | 0,47 | 0,017 | 50,0 | 0,35 |
| 5 | 0,46 | 0,089 | 25,4 | 0,33 |
| 6 | 0,19 | 0,003 | 20,1 | 0,24 |
| 7 | 0,15 | 0,011 | 11,0 | 0,13 |

Figures 1 and 2 show the calibrating curves used for Au and Ag with correlation coefficients and wavelengths.

The recording screens Au and Ag are shown in Figures 3 and 4. On the recording screens in the right corner, you can see the interfering elements accompanying the precious metals.These elements are removed by process of preparation samples.

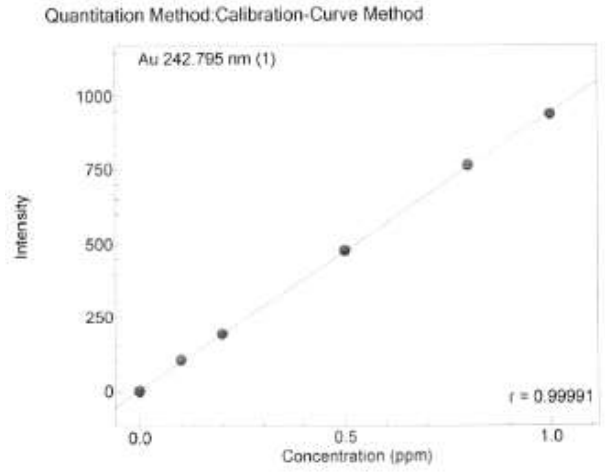


*Figure 1. Calibration curve for gold*

*Slika 1. Kalibraciona kriva za zlato*

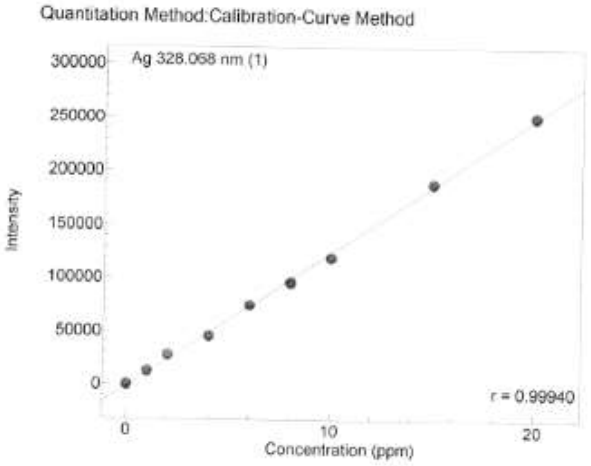


*Figure 2. Calibration curve for silver*

*Slika 2. Kalibraciona kriva za srebro*

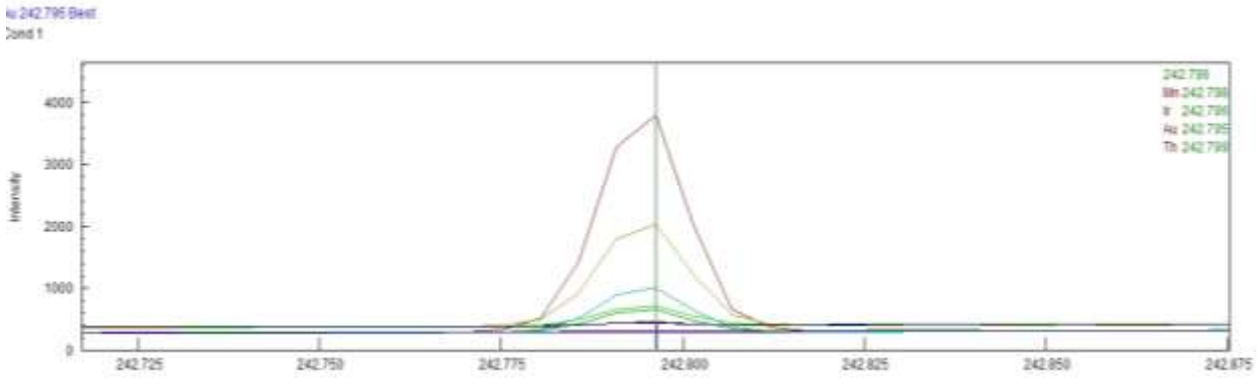


*Figure 3. Spectrum of gold*

*Slika 3. Spektri zlata*

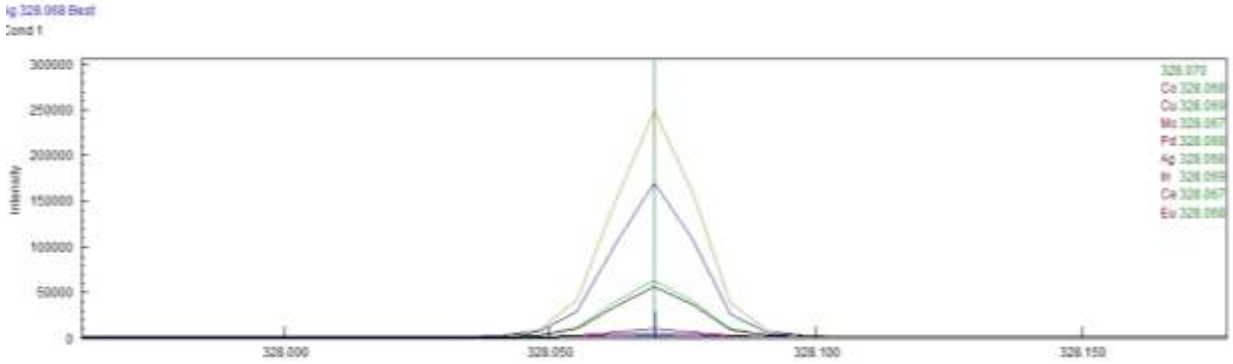


*Figure 4. Spectrum of silver*

*Slika 4. Spektri srebra*

Table 3 gives a summary of results obtained by comparing FA and FA / ICP AES methods.

*Table 3. Summary view of obtained results with differences*

*Tabela 3. Zbirni prikaz dobijenih rezultata sa prikazanom razlikom*

| Sample: | FA | | FA/ICP/AES | | difference: | difference: |
|---|---|---|---|---|---|---|
| | Au (g/t) | Ag (g/t) | Au (g/t) | Ag (g/t) | Au (g/t) | Ag (g/t) |
| 1 | 0,68 | 78,0 | 0,69 | 79,0 | +0,01 | +1,0 |
| 2 | 0,50 | 61,0 | 0,50 | 62,0 | 0,00 | +1,0 |
| 3 | 0,57 | 57,4 | 0,59 | 58,0 | +0,02 | +0,6 |
| 4 | 0,46 | 49,8 | 0,47 | 50,0 | +0,01 | +0,2 |
| 5 | 0,47 | 25,3 | 0,46 | 25,4 | +0,01 | +0,1 |
| 6 | 0,18 | 20,2 | 0,19 | 20,1 | +0,01 | -0,1 |
| 7 | 0,14 | 10,9 | 0,15 | 11,0 | +0,01 | +0,1 |

## 4. CONCLUSION

A method for the rapid and accurate determination of Ag and Au was elaborated in geological samples of iron ore by atomic emission spectrometry with induced coupled plasma in combination with the cupellation. The bubbling method was efficiently separated and concentrated noble metals from the total mass of the sample, which allowed further dissolution processes in the respective acids to reliably determine their contents on ICP AES.

Correlation coefficients of calibration curves: Au r = 0.99991, Ag r = 0.99940. Thickness of recording length: λ (Au) = 242,795nm; λ (Ag) = 328.068nm. The results obtained by the proposed method were compared to the results of analysis of the same samples by cupellation (FA) using 100g PbO in the flux and for the FA / ICP analysis 40g was used. The results are compatible, reliable and renewable as found in a number of experiments.

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## IZVOD

### ODREĐIVANJE Au I Ag IZ RUDE GVOŽĐA KOMBINOVANJEM FA I ICP/AES METODA

*U ovom radu je prikazana kombinacija FA (plamena analiza plemenitih metala-kupelacija) i ICP/AES (atomska emisiona spektrometrija sa indukovanom kuplovanom plazmom) metoda za određivanje Au i Ag u geološkim uzorcima rude gvožđa-magnetit. Au i Agse koncentišu sa Pb iz PbOnakon procesa odsumporavanja i topljenja ($Pb^{2+}$→Pb).Regulus (Pb sa Au i Ag)se kupelira. Dobijena perla-pril nakon kupelacije se rastvara u $HNO_3$ a zatim u HCl(carska voda $2HNO_3$ : 6HCl). Nakon pripreme standardnih rastvora i blank probe vrši se snimanje na ICP/AES. Dobijeni rezultati su poređeni sa rezultatima dobijenim klasičnom metodom kupelacije. Prednosti ICP/AES su odlične granice detekcije i linearni dinamički opseg kao i stabilan i ponovljiv signal što je posebno bitno za uzorke ispitivane rude gvožđa zbog malog sadržaja plemenitih metala.*

***Ključne riječi:*** *geološki uzorci, FA, ICP/AES, plemeniti metali*

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