

ICP-AES techniques applied to analysis of precious metals in ores

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Abstract A method for the simultaneous determination of precious metals and matrix elements in Fe and Cu ores is studied. The instrument used is ICAP-9000 polychromator and its accessory monochromator(N+1). Interferences of matrix are eliminated by matrix matching method, i.e. the influence of matrix was corrected by minus equivalent concentration method. The experiments show that the method is simple, sensitive and reliable; especially, suitable for determination of Au in Cu-Au ores.

Keywords Multi-element simultaneous analysis, Precious metals, Fe-Cu Ores, ICP-AES

1 Introduction

The purpose for this paper is in order to study whether there are values to develop precious metals in Fe and Cu ores as well as to tap potentialities of ICP-AES in precious metals analysis of ores, such as Au ores with Cu, Zn and Pb. ICP-AES is well suited for the analysis of precious metals. It has many advantages, such as wide concentration range, five to six orders of magnitude of linear dynamic range; small matrix effect; highspeed; the data of the concentration can be directly readed out; multi-element simultaneous analysis and low detection limits. So that the method is better than atomic absorption spectrometry and spectrophotometer. Because their interferences of matrix are too serious, so that the precious metals must be prior separated from matrix.

When mass fractions of Fe and Cu in matrix are less than 0.02. For the determination of trace precious metals, such as Au, Pt, Rh, Pd and Ag, they do not need to separate from matrix. When the content of matrix element is too high. They must be separated from matrix up to an extent.

2 Experimental

2.1 Apparatus

Spectrometer: Jarrell-Ash model 9000 and a 0.5m Ebert scanning monochromator(N+1). For polychromator, entrance and exit slits widths are 25 and 50 μm , respectively. For monochromator, entrance and exit slit widths

all are 50 μm .

Excitation source: 2.5 kW RF generator operating at 27 MHz with automatic power control and automatic tuning.

Plasma torch: Conventional small quartz torch.

Nebulizer: Cross flow pneumatic nebulizer.

Detectors: Photomultiplier tube (PMT) R427, R300 for polychromator; R889 for monochromator. Apple II microcomputer, Epson MX 80 printer.

2.2 Operating parameters

Output power: 1.1 kW

Argon flow rates: Coolent 18 L/min, plasma 0.5 L/min, carrier gas 0.5 L/min at 22 psi(pounds/square inch) = 1.54 kg/cm² determined by curve of detection signal vs pressure of carrier gas.

Observation height: 14 mm above load coil determined by curve of detection signal vs observation height.

Integration time: 10 s.

2.3 Standard solution preparation

In order to prepare respective standard solutions of 1 mg/ml, 0.100 g gold powder and 0.100 g silver wire are dissolved in aqua regia and in hot 4 mol/L nitric acid; 0.2275 g(NH₄)₂[PtCl₆], 0.2675 g (NH₄)₂[PtCl₄], 0.3856 g(NH₄)₃[RhCl₆] · 1.5H₂O, 0.3441 g(NH₄)₂[RuCl₆] and 0.2290 g(NH₄)₂[IrCl₆] are dissolved in 1 mol/L HCl; respectively.

Then, these solutions are diluted to 100 ml using deionized water.

2.4 Test solution

The solutions were prepared containing Fe or Cu of 0~5000 $\mu\text{g/ml}$ without precious metals. Effect of Fe or Cu on precious metals is observed.

2.5 Sample preparation

In a teflon beaker, 0.600 g well ground (~100 mesh) sample and 10 ml HF are placed. When solution is nearly dry, 20 ml aqua regia is added and heated at 100 °C for 4 h until complete dissolution. At a lower temperature it is evaporated again to near dryness. Finally, the solution is diluted to 5 ml using a dilute acid and to be determined.

2.6 Procedure

The instrument was profiled and calibrated using standard solutions of precious metals. After this, test solution, samples and blank were analyzed for three times

3 Results and discussion

3.1 Selecting condition for analysis^[1,2]

3.1.1 Wavelength selection

Precious metals are elements of complex. They have many spectrum lines e.g. Pd has 85 lines, Pt 113 lines, Ag 17 lines, Au 37 lines. Therefore, spectrum overlap interference is serious in this method, so that appropriate analyzed lines must be selected. In our ICAP 9000, Au, Ag, Pt, Ru, Fe and Cu all have fixed channels. Pd, Rh and Ir lines can be selected

using N+1 (see Table 1). Rh 249.1 nm is interfered by Fe. So that Rh 343.5 nm was selected. Pd 324.3 nm is interfered by Cu and Fe; Pd 363.5 nm interfered by Ar, so that Pd 340.5 nm was selected. Sensitive lines of Ir have 313.3 nm, 322.1 nm and 393.5 nm, line 393.5 nm is the best among these lines, so that Ir 393.5 nm was selected.

Table 1 Analytical control parameters

No ⁽¹⁾	Elements	Polychromator			
		PCN	λ/nm	Standard low sig.	Conc. high sig./ $\mu\text{g.ml}^{-1}$
1	deionized water	—	—	—	—
2	Cu	13	324.7	0	100
3	Fe	40	259.9	0	100
4	Pt	12	265.9	0	10
	Au	39	267.5	0	10
	Pd	27**	340.5	0	10
5	Ru*	42	240.2	0	10
6	Ag*	17	328.0	0	10

No ⁽¹⁾	Elements	monochromator(N+1)			
		PCN	λ/nm	Standard low sig.	Conc. high sig./ $\mu\text{g.ml}^{-1}$
1	deionized water	—	—	—	—
2	Cu	13	324.7	0	100
3	Fe	40	259.9	0	100
4.a	Ir	27**	393.5	0	10
4.b	Rh	27**	343.5	0	10

Notes: ⁽¹⁾ Standard sample number; PCN—physical channel number.

* Ag and Ru are not stable. Therefore, leave them out of the standard. The shelf life of standard will be longer.

** PCN 27 is represented to use monochromator.

3.2 Detection limits(see Table 2)

Table 2 Detection limits of precious metals

Elements	λ/nm	D.L./ $\mu\text{g.ml}^{-1}$	Elements	λ/nm	D.L./ $\mu\text{g.ml}^{-1}$
		polychromator			monochromator
Au	267.5	0.008	Pd	340.5	0.08
Ag	328.0	0.004	Rh	343.5	0.08
Pt	265.9	0.05	Ir	393.5	0.05
Ru	240.2	0.04	Os		no determined

D.L. is defined as the concentration giving an intensity twice that of the standard deviation of the background.

3.3 Spectral interference of matrix elements^[3,4]

The effects of matrix on Au, Pd, Pt, Rh, Ru, Ag and Ir were studied. The extent of interference is Ir>Ru>Pt>Pd>Au>Rh>Ag in the presence of high content of iron. The extent of interference is Ir>Ag>Pd>Ru>Pt>Rh>Au in the presence of high content of copper (see

Figs.1,2). Though physical and chemical nature of precious metals are quite similar but trace precious metals are not interfered by each other. There are Pb and Zn in the ores by qualitative certification. Experiment showed that precious metals were not interfered by $\leq 100 \mu\text{g/ml}$ Pb and $\leq 300 \mu\text{g/ml}$ Zn, so that the method es-

tablished in the paper is useful especially for analysis of Au ores with Cu,Zn and Pb. Because Au often coexists with Cu, Zn and Pb.

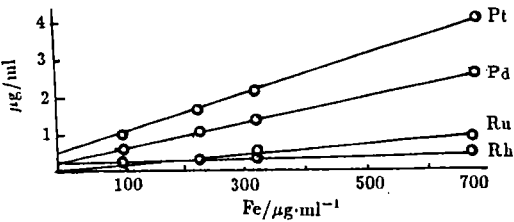


Fig.1 Effect of iron on precious metals

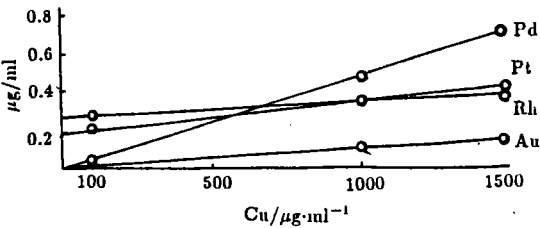


Fig.2 Effect of copper on precious metals

3.4 The recoveries of precious metals

The solution of 0.600 g 1# ore sample is diluted to 10 ml with a dilute acid as blank solution. Then 100µg Au, Pd, Pt and 150µg Rh are added to another solution of 0.600g 1# ore sample. Finally it is diluted to 10 ml with a diluted acid as sample solutions. After two kinds of solutions are determined simultaneously, the recoveries can be calculated by the following formula:
{sum-ore sample blank}/{added standard/10}

The recoveries of precious metals in 2# ore are similar to 1# ore. Tests of recoveries are made twice, i.e. each ore is dissolved for two times. The results are listed in Table 3.

Table 3 Recoveries of precious metals

Elements	1# Ore		2# Ore	
	1#-1	1#-2	2#-1	2#-2
Au	0.96	0.86	0.97	0.88
Pd	0.92	0.90	0.94	0.99
Pt	0.98	0.86	0.87	0.89
Rh	0.88	1.11	0.85	1.15

It can be seen from Table 3 that relative errors are 0.01 ~ 0.15.

3.5 Dtermination of ore samples

The content of each precious metal is obtained by ore sample value minus matrix interference value on corresponding precious metal PCN. Ore samples all come from Mei County of Guangdong Province. Results are listed in Table 4. Other methods also proved that contents

of precious metals in the two ores are indeed very low.

Table 4 Results of determination of ore samples

No.	1# Ore		2# Ore	
	µg/ml	µg/g	µg/ml	µg/g
Fe	117	1957	540	9000
Cu	744	12400	2.4	40
Au	0.419	7.0	< D.L.	< 0.13
Pd	<D.L.	< 1.3	<D.L.	< 0.13
Pt	<D.L.	< 0.83	<D.L.	< 0.83
Rh	<D.L.	< 1.3	<D.L.	< 1.3

4 Conclusions

a. ICP-AES technique can be used to determine the precious metals and the matrix elements simultaneously.

b. Detection limits attained with polychromator are better than those with the monochromator under similar conditions.

c. The results showed that the concentration of precious metals are very low in Fe-Cu ores. So the Fe-Cu ores are not worthy developing.

References

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