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DETERMINATION OF PLATINUM AND PALADIUM WITH GF AAS

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ABSTRACT
The method has been developed for the determination of low content of platinum and palladium, ppb level, using GF AAS in the copper concentrate. The fire assay method with lead, using the large weight of samples (3x50g), were used for the exctracting of precious metals and the content of Au and Ag were determined. Subsequent to the dissolution of Ag with nitric acid and Au with aqua regia, the solution were transformed into the volumetric flask of 10 cm^3, and the content of Pt and Pd were determined. The results show the presence of Pt and Pd in the nitric acid solution and their presence at the traces level from the aqua regia solution.The content of Pt and Pd were determined with graphite furnace at the temperature of atomization of 2500°C. The atomization of Pd was on the L'vov platform, and Pt on the wall. The lower detection limit for platinum and palladium is 1 ppb. The obtained GF AAS results were compared with ICP-OES results and the good agreement . GF AAS technique is quite selective, precise and not so expensive, and could be used after the determination of the content of silver and gold with fire assay method. The developed method could be applied for the ore samples and other flotation copper products.
Keywords: platinum, palladium, fire assay, atomic spectroscopy with graphic furnace.

1. INTRODUCTION

Metod of cupellation is the oldest and most reliable method for the separation and determination of precious metals in samples where their content is of order ppm. Laboratory of the Institute of Mining and Metallurgy in Bor cupellation method applies to the determination of silver and gold in geological samples, products and copper flotation concentrate and all the gold-bearing materials. In this study of cupellation was combined with atomic absorption spectrophotometry with a graphite furnace (GF AAS) for the determination of platinum and palladium from these samples. Due to the use of large sample weight of the sample (150 g), limit of platinum and palladium on the order ppb.

2. EXPERIMENTAL

Reagents

- Batch melting: PbO, Na_2CO_3, $Na_2B_4O_7$, reducing agents.
- Acid solution of beads: HNO_3, diluted (1:2), aqua regia.
- Standard solutions of platinum and palladium. We used commercial standards for AAS 1000 μg / cm^3. Of them were made diluted standard solutions, always prepared on the day recording.

Apparatus and Equipment:

-Chamotte pots of 150 cm^3, coupe ϕ 60 mm wells furnace with a working temperature of 1070 - 1100 ^{0}C, furnace cupellation 20 kW.

- Atomic absorption spectrophotometer with a graphite furnace, automatic sampler, and a printer.
- The hollow cathode lamps for platinum and palladium. Graphite furnace with L 'vov platform for Pt and pyrolytic graphite tubes without platform for Pd.

- Micropipettes

3. PROCEDURE

The process of melting and cupellation. I selected sample mass of 50g. For the removal of sulfur in the melt affects the loss of the precious metals, the samples were baked at a temperature of 600 ^{0}C. Select the optimal composition of the batch that is well mixed with the samples and quantitatively transferred to solitary pots. Melting process was carried out at a temperature of 1070 ^{0}C for the duration of one hour. The molten mass is poured into iron cups. After cooling, the lead is separated from the slag blacksmithing. Separate lead was placed in a hot coupe and cupellation worked at a temperature 890-940 ^{0}C. Abstract grain at the bottom coupe for further proceedings is acidic disolved.

Dissolution balls. The resulting pellets - an alloy of silver, gold, platinum and palladium dissolved with dilute acid and nitrogen solution completed in volumetric flask of 10 cm^3. Zero sample was prepared in the same manner and with the same amount of reagents but without the sample.

Recording of platinum and palladium: All tests for Pt and Pd were carried out on the device Perkin Elmer 1100B AAS with graphite furnace HGA 70. Insert the sample cuvette was carried out with an automatic sampler AS-70 on pyrolytic and L ' vov platform. For the preparation of calibration curves were made solutions of 100, 200, 300 and 400 ngPt/cm^3 respectively ngPd/cm^3 . In this paper, we selected peak height measurements, using begraund corrections.

4. ANALYTICAL SHOOTING CONDITIONS:

a) The instrumental conditions

For platinum
Wavelength ..... 260 nm
Slot width ..... 0.7 nm
Hollow cathode ..... 20 mA.
The integration time 5s

For palladium
Wavelength ..... 247.6 nm
Slot width ..... 0.7 nm
Hollow catode..... 30mA
The integration time 4s

b) HGA conditions for Pt and Pd

Table 1. HGA conditions for Pt and Pd

Steps number	Temperature furance (°C)	Time ramp (s)	Time hold (s)	Internal gas flow ml/min
1	120	10	15	300
2	1300 / 1100	10	15	300
3	2200	0	3	0
4	2650	1	3	300

Sample volume V = 15µ l

5. RESULTS AND DISCUSION

At the end of the cupellation of Cu concentrate sample was done in triplicate, separated beads were collected and dissolved successively. In order to separate silver from other precious metals, beads were first dissolved with a dilute nitric acid and the resulting solution was integrated in volumetric flsk 10cm^3. Insoluble part (gold) diluted the aqua regia solution completed in the second volumetric flask. For both platinum and palladium solution were determined by the proposed method. The results of the analysis are shown in Table 2.

Table 2. The content of precious metal in copper concentrate in ppm

Test methods					
CUPELLATION		GF AAS			
		Dissolved HNO_3		Aqua regia	
Ag	**Au**	**Pd**	**Pt**	**Pd**	**Pt**
29.4	4.4	0.33	0.02	0.003	<0.001

To experimentally verify this result, the influence of Ag, Au determination of platinum and palladium by the proposed method. Artificially prepared sample of silver bullion, gold bullion, $PdCl_2$, $PtCl_2$, in that respect what is in our pooled sample. All components are wrapped in lead foil and copellation worked at t = 1100 ° C. The resulting pellet – the precious metals, dissolved by the same procedure as the pellet sample. The nitric acid solution was obtained 98% Pd and 95% Pt were taken from the amount of artificial preparation of the sample. The content of palladium was expected, but the question of dissolution and finding platinum in nitrogen-acid solution?

The ratio of silver and platinum (Table 2) is 1000: 1. In terms of cupellation, at high temperature occurred alloying Ag and Pt. Diluted solution of nitric acid dissolved in the platinum balls - noble alloy elements, building probably platinum nitrate. This is very important for the determination of precious metals using cupellation, the platinum in this relationship dissolved by nitric acid with silver and palladium. Platinum in aqua regia solution is not registered, and palladium, less than 1%.

Tests were carried out on the nitrogen acid solutions on samples of copper concentrate, domestic and imported, which are processed in Bor. (Table 3)

Table 3 The content of palladium and platinum in the copper concentrate

The sample label	Pd (ppm)	Pt (ppm)
Bor flotation	0.052	0.004
Flotation V.Krivelj	0.090	0.005
Flotation Majdanpek	0.500	0.063
Imported concentrate	0.250	0.014

The content of palladium in Table 3, which is approximately ten times higher than that of platinum is expected, because such a relationship occurs when testing these elements in samples of intermediate obtaining pure silver and gold from copper concentrates, which our lab regularly monitored. The proposed method can be applied to samples from the copper flotation process (Table 4). Nitrogen acid solution obtained after cupellation, was shot in the furnace and plasma. Comparative analysis of the results of the two techniques are shown in Table 4.

Table 4 Comparative results of Pd and Pt in ppm, a GFAAS and ICP-OES

	Palladium		Platinum	
The sample label	GFAAS	ICP-OES	GFAAS	ICP-OES
Experience tailings J1	0.005	0.006	0.005	0.007
Experience tailings J2	0.003	0.007	0.002	0.006
Experience tailings Jdef.	0.006	0.004	0.006	0.004
gained entrance	0.006	0.008	0.005	0.005
Experience K def.	0.034	0.014	0.016	0.018
sample 1	0.009	0.011	0.015	0.011
sample 2	0.009	0.011	0.007	0.009
sample 3	0.008	0.010	0.006	0.008
Concentrate	0.250	0.150	0.021	0.018

6. CONCLUSION

A method for the relatively rapid and accurate determination of palladium and platinum in concentrate copper by atomic absorption spectrophotometry with a graphite furnace. GF-AAS is selective, precise, and not so expensive method that is applied after determining the Ag and Au using cupellation method. Important effect of this work is dissolution (separation) from nitric acid platinum beads of precious metal that is obtained after cupellation. The results of determining Pt and Pd by the proposed method are compared with the results of ICP-OES, and show good agreement.

The proposed method can be applied to samples of ore and produce copper flotation. Because of the potential large sample weight, the lower limit of the Pd and Pt is 1 ppb.

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