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**DETERMINATION OF GOLD IN ARSENIC-ANTIMONY ORE
 BY FLAMELESS ATOMIC ABSORPTION SPECTROMETRY**

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ABSTRACT

Gold was determined in complex ore by flameless atomic absorption spectrometry. Iron interferes with the determination of gold by furnace atomic absorption even after extraction with methyl isobutyl ketone. Separation of iron is based on precipitation by ammonium hydroxide before extraction. The detection limit of the method is sufficient for geochemical prospecting for gold.

INTRODUCTION

Atomic absorption determination of gold in complex samples by aqua regia digestion and extraction by different organic solvents has been investigated extensively and is well-documented: methyl isobutyl ketone (1-9), organic sulfide (10-12), dithiocarbamates (10).

Flame atomic absorption determination of gold by extraction with methyl isobutyl ketone from aqueous hydrochloric acid has been described by several workers (1, 5, 6). Usually, the first step in determining gold is extraction with methyl isobutyl ketone which results in removal at an early stage of determination. However, it is a well-known fact that gold determination by atomic absorption spectroscopy is seriously affected by interferences from different components in complex materials (13-16). The same effects are observed when gold is determined by furnace atomic absorption spectroscopy. Results and explanations of the possible factors affecting the determination of gold in complex samples have been published (2, 3).

However, the determination of gold in arsenic-antimony ore has not been presented. In this work, influences of all elements present in the solution of arsenic-antimony ore which affect the determination of gold, using furnace atomic absorption spectroscopy with or without extraction by methyl isobutyl ketone, are investigated.

EXPERIMENTAL

Instrumentation

A Perkin-Elmer[®] Model 703 atomic absorption spectrophotometer equipped with a deuterium background corrector, an HGA[®]-400 graphite furnace, and a Model 056 strip-chart recorder was used. A gold hollow cathode lamp was used as a source. Background correction was applied throughout the course of analysis. Pyrolytically coated graphite tubes were used.

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Operating conditions for gold determination were established by extensive testing and are given in Table I.

TABLE I
Instrumental Parameters

<i>Model 703</i>	
Wavelength	242.8 nm
Spectral slit width	0.7 nm
Calibration mode	Absorbance, peak height
Background correction	Deuterium arc lamp
<i>HGA-400</i>	
Dry	120 °C, 20 s, ramp 2 s
Char	600 °C, 20 s, ramp 1 s
Atomize	2400 °C, 5 s, Maximum Power mode and Mini Flow mode
Cleaning	2700 °C, 3 s

Reagent and Samples

All reagents and standards were of analytical grade. Stock solution of gold was prepared by dissolving the metal samples of pure gold with aqua regia. The concentration of gold was 1000 mg/L and from this solution the other diluted solutions were prepared.

The ore samples are from the Alshar arsenic-antimony mine near Kavadarci, SR Macedonia, Yugoslavia.

Sample Preparation and Procedure

For better homogeneity, ore samples were crushed on a jaw crusher to 10 mm and on a cone crusher to 2-5 mm. After that a 100-g sample was ground on a vibratory mill to 0.074-mm particle size.

A 10-g finely powdered ore sample was transferred to a glass beaker and heated for 2 hr at 550 °C to eliminate sulfur (16). After heating, the sample was dissolved by the method proposed by Tindall (6, 7). After dissolution, the filtrate was collected in a 100-mL volumetric flask and diluted to volume. From this solution, 10 mL (or more, depending on the gold concentration) was taken and diluted with 10 mL deionized water; Fe(III) was precipitated by adding ammonium hydroxide, 0.2 g ammonium chloride, and 5 mL hydrogen peroxide. After heating one hour on a hot plate, the solution was filtered through filter paper. The filtrate was acidified and

made to volume with 2 M HCl. Then the solution was treated with 10 mL (or less, depending on the gold concentration) methyl isobutyl ketone to extract gold. This solution was ready for injection.

A series of standard solutions was prepared from the stock solution of gold. All standard samples were also treated according to the previous procedure. A calibration curve was constructed in the range of 0.1–5.0 ng gold.

RESULTS AND DISCUSSIONS

There are a number of papers on the determination of gold by flameless atomic absorption spectroscopy (2–4, 11, 17–22), but only a few authors investigated matrix interferences (2, 3). A series of different elements (As, Sb, Ca, Mg, Al, Fe, Tl, Cu, Zn, Pb) was tested to see if they caused any interference in the determination of gold. These influences were investigated by measuring the absorbance of gold in a series of samples with varying concentrations of added potential interferent.

All measurements were performed with and without background correction. The results showed that background absorption was present and that it was necessary to use a background corrector.

In most cases, especially in samples with higher concentrations of interfering element, the absorbance of gold decreased. When the interfering element was arsenic, calcium, magnesium, or iron, absorbance decreased rapidly (Figures 1 and 2). The situation was the same when the determinations were performed at higher preheating and atomization temperatures.

These influences make the direct determination of gold by furnace atomic absorption spectroscopy impossible. For

this reason, gold was extracted with one of the better extractors, methyl isobutyl ketone.

In previous papers (2, 4, 20) the influence of coextracted potentially interfering elements on the determination of gold has not been presented. This influence was determined by extracting gold in the presence of different elements in a proper concentration ratio. Examination of these results shows that the absorbance of gold is not changed except when iron is present (Table II).

TABLE II
Interference of Iron on the Absorbance of Gold in the Organic Phase

Sample no.	Mass ratio	Absorbance
	Au:Fe	
1	1:0	0.25
2	1:100	0.22
3	1:1000	0.06
4	1:2000	0.04
5	1:4000	0.04

To eliminate interference, iron was precipitated with ammonium hydroxide. After filtration, it was necessary to acidify the solution and extract it with methyl isobutyl ketone.

This method was checked by the method of additions. An exact amount of standard solution of gold was added to arsenic-antimony ore samples with a known concentration of gold. The results of gold determination and the recovery are given in Table III.

TABLE III
Determination of Gold in Arsenic-Antimony Ore

Sample	Au found ($\mu\text{g/g}$)	Au added (μg)	Au expected ($\mu\text{g/g}$)	Au found ($\mu\text{g/g}$)	Recovery (%)
AL1	0.38	4	0.78	0.77	98.7
AL2	0.49	4	0.89	0.87	97.7
AL3	1.63	4	2.03	1.98	97.5

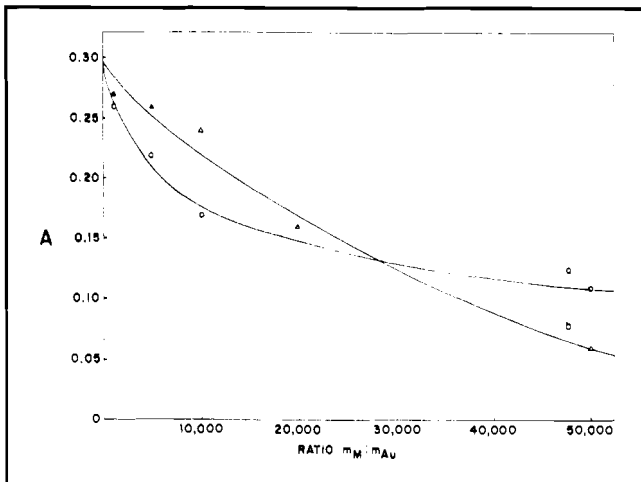


Fig. 1. Influence of gold absorbance by calcium (a) and magnesium (b).

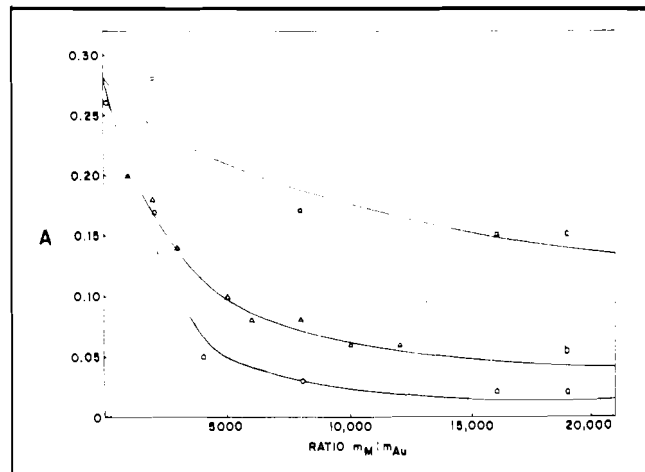


Fig. 2. Influence of gold absorbance by arsenic (a), antimony (b), and iron (c).

As a further check, determination of gold in similar standard sulfide ore samples, UM-1 (23) and SU-1a (24), was performed. The results are shown in Table IV.

A calibration curve was constructed by similar treatment of samples containing up to 5 ng gold (Figure 3). With this method or a modification of the procedure (larger mass of samples and/or smaller volume of ketone), it was possible to determine gold in complex matrix samples down to $1 \cdot 10^{-4}$ $\mu\text{g/g}$.

TABLE IV
Determination of Gold in Standard Sulfide Samples

Sample	Au concentration* ($\mu\text{g/g}$)	Au found ($\mu\text{g/g}$)
UM-1	0.08	0.082
SU-1a	0.15	0.148

*Ref. 23, 24.

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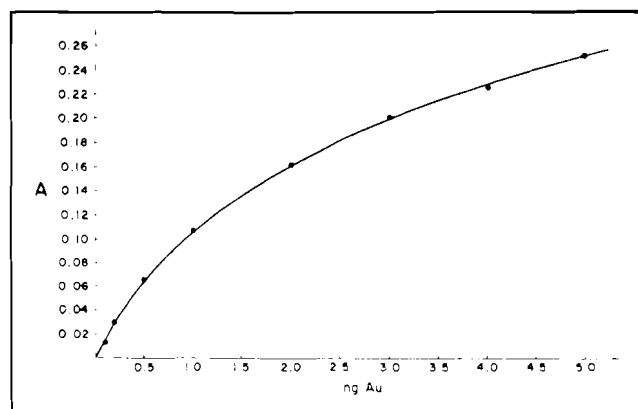


Fig. 3. Calibration curve for the determination of gold by graphite furnace.