

Determination of Molybdenum in Arsenic-Antimony Ore by Flameless Atomic Absorption Spectrometry*

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INTRODUCTION

For the determination of molybdenum in complex samples by flame atomic absorption spectrometry (FAAS), other elements present in higher concentrations have been found to reduce Mo absorbance (1-5). To eliminate the interference of these elements, addition of compounds such as ammonium chloride (3,6) or aluminum chloride and ammonium chloride (5) has been suggested. Other literature shows extraction of Mo directly from the solution (7,8).

Interferences on Mo determination in complex samples have also been reported for flameless AAS (9-14). To our knowledge, a more extensive investigation of these influences has not been reported. In this work, we investigated all elements present in arsenic-antimony ores by using flameless AAS. It was found that even small quantities of ascorbic acid helped to eliminate interferences.

EXPERIMENTAL

Instrumentation

A Perkin-Elmer® Model 703 atomic absorption spectrophotometer was used equipped with D₂ background corrector, HGA-400 graphite furnace, and Model 056

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stripchart recorder. A Mo hollow cathode lamp was used as the source. Background correction was used throughout the analysis together with pyrolytically coated graphite tubes. Instrumental parameters are given in Table I.

Reagents and Samples

All reagents and standards were of analytical reagent grade. The stock solution of Mo was prepared by dissolving ammonium paramolybdate with deionized water. The concentration of Mo was 1000 mg/L, from which all diluted solutions were prepared.

The ore samples were from the Alshar arsenic-antimony mine near Kavadaci, SR Macedonia, in Yugoslavia.

Procedure

Preparation and dissolution of samples was similar to the furnace AAS method used for the determination of gold in the same types of samples (15).

Samples of finely powdered ore (10 g) were put into a glass beaker and heated for two hours at 450°C to remove sulfur (16). Then, 25-mL concentrated HCl was added and heated for 15 minutes; 15 mL concentrated HNO₃ was added and heated about 30 minutes. To remove the nitrogen oxide vapors, 25 mL concentrated HCl was added and heated. Deionized water was then added. The selection was filtered, and the filtrate collected in a 100-mL volumetric flask. Finally, 2 g ascorbic acid was added to the filtrate and the flask filled to volume with deionized water.

TABLE I
Instrumental Parameters

Model 703			
Wavelength	313.3 nm		
Spectral slit width	0.7 nm		
Calibration mode	Absorbance, peak height		
Background correction	D ₂ arc lamp		
HGA 400			
	Dry	Char	Atomize
Temp (°C)	120	1800	2700
Hold time (s)	20	20	7
Ramp time (s)	2	1	Max. power mode
			Mini flow mode
Clean	2700	3	-

RESULTS AND DISCUSSION

A number of papers have been published on the determination of Mo by flameless AAS (9-14); however, no literature is available discussing a more detailed investigation of the influence of other elements in complex ore samples.

In this work, the absorbance of Mo was measured in a series of samples containing a constant concentration of Mo but varying the concentrations of added potential interferent, i.e., Fe, Al, Ca, Mg, As, and Sb (mass ratio of $m_{Mo}:m_M = 1:100\ 000$).

All measurements were performed with and without background correction. The results showed that background absorption was present and that use of background correction was necessary.

Figure 1 shows that interference from other elements present in the solution (especially with higher concentrations) decreases the absorbance of Mo. The concentration range of these interfering elements in the ore samples varied from 0.5 to 10%.

To possibly eliminate these interferences, addition of ascorbic acid was investigated. Ascorbic acid eliminates the Fe interference in the determination of Mo in steels (12), as well as in the determination of other elements in complex samples (12,14,17).

We investigated the effect of ascorbic acid (2% or 5%) on the absorbance of Mo in a series of samples by measuring Mo in the presence of different elements, with and without ascorbic acid. The results showed that the addition of ascorbic acid significantly reduces interferences. The same results were obtained when the concentration of ascorbic acid was 2% or 5%.

We also checked this procedure with the method of additions.

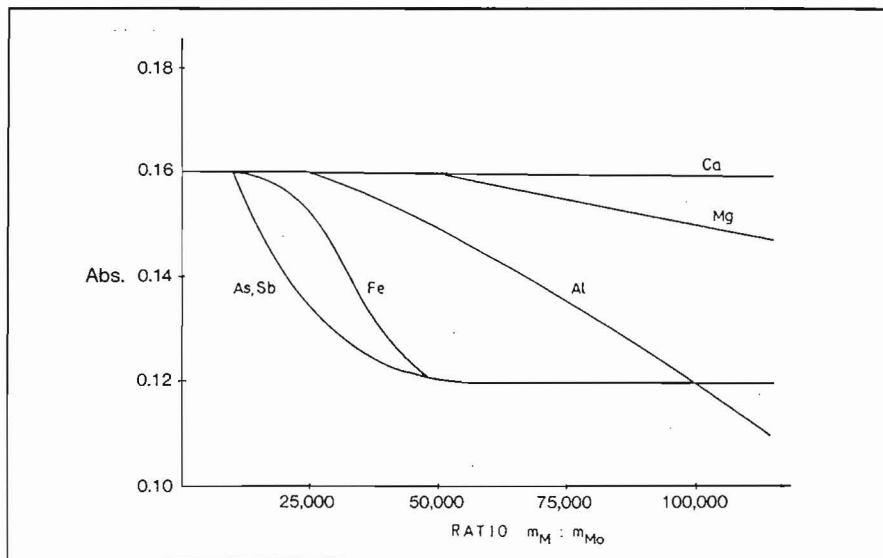


Fig. 1. Influence on absorbance of Mo by As, Sb, Fe, Al, Mg, and Ca.

TABLE II
Determination of Mo in Arsenic-Antimony Ore

Sample	Mo found ($\mu\text{g/g}$)	Mo added (μg)	Mo expected ($\mu\text{g/g}$)	Mo found ($\mu\text{g/g}$)	Recovery (%) With ^a	Without ^b
B 41	0.87	5.0	1.37	1.33	97.1	74.5
B 71	0.72	5.0	1.21	1.16	95.9	82.1
B 75	7.00	5.0	7.50	7.55	100.7	69.8

^a With ascorbic acid.

^b Without ascorbic acid.

Ascorbic acid and an exact amount of Mo standard solution were added to arsenic-antimony ore samples that had a known concentration of Mo. The results (with and without ascorbic acid) are given in Table II.

This method was then compared to the analysis of the same samples with inductively coupled plasma spectrometry as outlined by Markova (18,19). For results of this comparison see Table III.

A calibration curve was constructed for samples containing up to 5 ng Mo, with 2% ascorbic acid added. The relative standard deviation

for samples with a concentration around 1 $\mu\text{g/g}$ Mo was 2.3%.

TABLE III
Comparison of Proposed Method With ICP

Sample	Proposed method ($\mu\text{g/g Mo}$)	ICP ($\mu\text{g/g Mo}$)
ALM 1	5.8	6.0
ALM 2	5.1	5.0
ALM 3	2.7	3.0
ALM 4	9.7	10.0

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