

MATRIX INTERFERENCES IN THE DETERMINATION OF LEAD IN As-Sb-Tl  
ORES BY ELECTROTHERMAL ATOMIC ABSORPTION SPECTROMETRY

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The interferences of Tl, As, Sb, Ca, Mg, Al, Fe, Zn, Cu, and Ni in the determination of lead by electrothermal atomic absorption spectrometry, were studied. The interferences were investigated by measuring the absorbance of lead in series of samples with varying mass ratios of lead and potential interfering element. It can be concluded that with exception of Tl and Zn, all other elements influence to the absorbance of lead. The mass ratios, when the decreasing of the lead absorbance begins, were determined.

INTRODUCTION

According to the investigations of the project "Use of the thallium minerals as solar neutrino detectors", it is necessary to determine the content of  $^{205}\text{Pb}$ , which is accumulated in the thallium minerals of the Alshar mine, near Kavadarci, SR Macedonia, Yugoslavia. This isotope, with half-time of disintegration about 14 million years, is produced in the nuclear reaction between solar neutrino and  $^{205}\text{Pb}$ :



It is assumed that the accumulation of  $^{205}\text{Pb}$  lasts about 10 million years, inside the thallium minerals.

By separation of the most occurred thallium mineral in Alshar, lorandite ( $\text{TlAsS}_2$ ), and by determination of  $^{205}\text{Pb}$ , it can be found out the flux of the emitted solar neutrino.

Therefore, it is necessary exactly determination of the lead content in the thallium minerals, and in the other minerals, ores and rocks from Alshar. This have to be done, because of the possibility of being  $^{205}\text{Pb}$  which becomes from the lead in the other minerals and materials.

Until recently, there are little data about the content of lead in the minerals from Alshar. There are some data from Todt /1/, who has determined the content of lead in two samples of lorandite, one sample of orpiment, and one sample of realgar by mass spectrometry.

In this paper, we present the results from the investigation of the possibility for determination of lead in minerals and ores from Alshar by electrothermal atomic absorption spectrometry (ETAAS). Our point of interest is to determine the matrix interferences of the elements which are present in high concentrations in these materials, such as: Tl, As, Sb, Fe, Ca, Mg, Al, Cu, Zn, and Ni.

## 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 stripchart recorder was used. A lead electrode discharge lamp was used as a source. Operation conditions for lead determination were established by extensive testing, and are given in Table I.

### REAGENTS AND SAMPLES

All reagents and standards were of analytical grade. Stock solution of lead was prepared by dissolving of  $\text{Pb}(\text{NO}_3)_2$  in redistilled water. The mass concentration of lead was  $1000 \text{ mg}\cdot\text{dm}^{-3}$ , and from this solution the other diluted solutions were prepared.

Table I  
Instrumental Parameters

Model 703	
Wavelength	383,3 nm
Spectral slit width	0,7 nm
Calibration mode	Absorbance, peak height
Background correction	Deuterium arc lamp
Lamp power supply	10 W
HGA-400	
Dry	120 °C, 30 s, ramp 2 s
Char	550 °C, 20 s, ramp 2 s
Atomize	2100 °C, 5 s, maximum power mode, and miniflow mode (Argon)
Cleaning	2700 °C, 3 s

#### RESULTS AND DISCUSSION

Up to now, ETAAS is widely used for the determination of lead in a variety of complex materials, so there are number of papers about it. In a part of the presented papers, the results of the investigations of matrix interferences are given. However, in most cases these investigations are about some simple matrixes, such as: NaCl /2-6/, CaCl<sub>2</sub> /2, 4, 7-9/, sulfates and phosphates /5, 6, 10-13/. Vandegaus et al. /5/ studied the interferences of a large number of elements, such as: Ca, Cu, Zn, Fe, K, Na, and Mg in chloride and nitrate water solutions. Sedykh et al. /14/, performed the interferences of K, Ca, Mg, Al, Co, Ni, Fe, and Cu on the absorbance of lead. These investigations do not include all elements which are present in our samples (for example Tl, As, Sb), and also they are not in the same concentration ranges.

Therefore, the interferences of the elements which are present in the minerals and ores from Alshar are studied. For this purpose, series of samples with varying concentration of lead were prepared. The interference of the added element was determined

by measuring the absorbance of lead in the solution, without, and with addition. The investigations were done in solutions of  $2 \text{ mol} \cdot \text{dm}^{-3}$  hydrochloric acid.

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

In the most cases, especially in the samples with high concentrations of the interfering element, the absorbance of lead decreases (Fig. 1, 2, and 3). However, there is no decreasing of the absorbance of lead in such samples where the mass ratio of Pb within Mg is up to 1:500, 1:1000 for Ca, Fe, and Al (Fig. 1), 1:10000 for Sb (Fig. 2), and 1:10000 for Ni and Zn (Fig. 3). There are no interferences only in the cases when thallium is present in mass ratio up to 1:60000 (Fig. 2), and when zinc is present in mass ratio up to 1:20000 (Fig. 3).

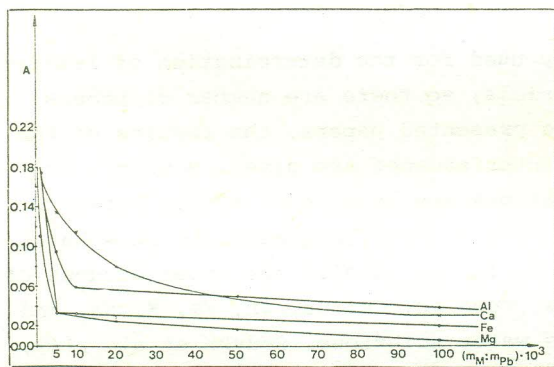


Fig. 1 Influence of lead absorbance by Al, Ca, Fe, and Mg.

It can be concluded that the interferences of all these elements (with exception of Tl and Zn), in the same concentration range as in the minerals and ores from Alshar, are very high, so the direct determination of lead by ETAAS is impossible. Therefore, for avoiding interferences, it is necessary to find some methods for lead separation from the matrix, such as: preconcentration, extraction, ionic exchange etc.

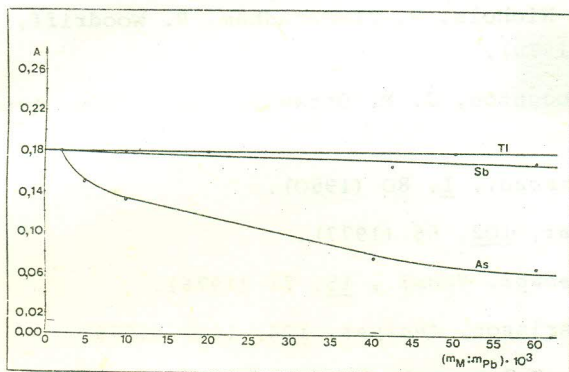


Fig. 2 Influence of lead absorbance by Tl, Sb, and As.

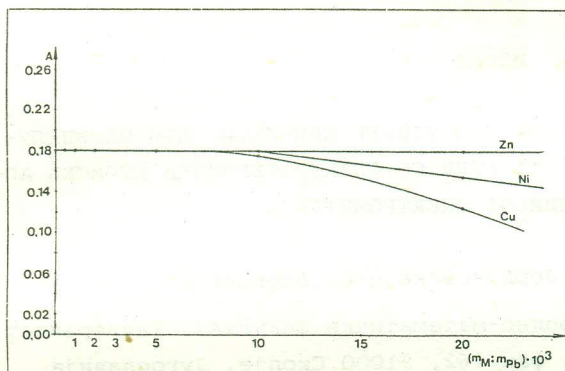


Fig. 3 Influence of lead absorbance by Zn, Ni, and Cu.

#### REFERENCES

1. W. Todt, *Nucl. Inst. and Meth. Phys. Res.* **A271**, 251 (1988).
2. D. Manning, W. Slavin, *At. Absorp. Newsl.* **17**, 43 (1978).
3. M. Tominaga, Y. Umezani, *Anal. Chim. Acta*, **139**, 279 (1982).
4. M. P. Bertenshaw, D. Geltshorpe, K. C. Wheatstone, *Analyst*, **107**, 163 (1982).
5. J. Vandegans, P. Rosseels, W. Verplancken, J. C. Huarez, *Anal. Chim. Acta*, **193**, 169 (1987).
6. W. Frech, A. Cedergin, *Anal. Chim. Acta*, **88**, 57 (1977).

7. J. G. T. Regan, J. Waren, *Analyst*, 101, 220 (1976).
8. L. R. Hageman, J. A. Nichols, P. Vismanadham, R. Woodriff, *Anal. Chem.*, 1406 (1979).
9. M. C. Halliday, C. Houghton, J. M. Ottaway, *Anal. Chim. Acta*, 119, 67 (1980).
10. S. Callio, *At. Spectrosc.*, 1, 80 (1980).
11. D. J. Hodges, *Analyst*, 102, 66 (1977).
12. A. Anderson, *At. Absorpt. Newsl.*, 15, 71 (1976).
13. S. Bacuman, R. W. Karlsson, *Analyst*, 104, 1017 (1979).
14. E. M. Sedykh, Yu. I. Belyaev, E. V. Sorokina, *Zh. Anal. Khim.*, 35, 2162 (1980).

## ИЗВОД

ИСПИТУВАЊЕ НА ВЛИЈАНИЕТО НА ПРИСУТНИТЕ ЕЛЕМЕНТИ ПРИ ОПРЕДЕЛУВАЊЕТО НА ОЛОВО ВО As-Sb-Tl РУДИ СО ЕЛЕКТРОТЕРМИЧКА АТОМСКА АПСОРПЦИОНА СПЕКТРОМЕТРИЈА

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Извршено е испитување на влијанието на Tl, As, Sb, Ca, Mg, Al, Fe, Zn, Cu и Ni на определувањето на олово со електротермичка атомска апсорпциона спектрометрија. Влијанијата беа испитувани со мерење на апсорбанцата на оловото, во серии од проби со различни масени односи на оловото и испитуваниот елемент. Покажано е дека освен талиум и цинк, сите други елементи влијаат на намалување на апсорбанцата на оловото. Утврдени се и граничните масени односи кога отпочнува намалувањето на апсорбанцата на оловото.