## Modern Research Methodologies for the Determination of the Heavy Metals Accumulation in the Soil

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## ABSTRACT

The present paper approaches a usual problem in the industrialised area, respectively Zlatna Area in Romania. The aim of this research is to establish the heavy metal soil contamination degree using modern research methods namely spectrometric and stripping methods. The results pointed out very high exceeding values, especially in the case of lead and copper, in the studied area. Moreover, the mobility and the availability of heavy metals decreases when pH increases. Findings suggest that the use of modern technologies have good results regarding the determination of the heavy metals in the soil.

#### **KEYWORDS**

Heavy Metals, Polarographic Methods, Soil Pollution, Spectrometric Methods

## INTRODUCTION

The soil is a dynamic system where short-term fluctuations occur, such as variations in humidity and pH levels, in redox conditions; it is also the place where the organic matter gradually decomposes as a consequence of changes in nature. These changes alter the shape and availability of metal ions and therefore they must be taken into account when making a decision on soil pollution or waste storage. Through field and laboratory tests, soils can be characterized from a physical, chemical and biological perspective.

The total metal content of soils is the result of varied metal input – parental material, atmospheric deposits, chemical fertilizers and improvements, organic fertilizers and other organic and inorganic polluting substances – minus metal output resulted from cropping or from leaching and volatilisation. All these factors can be quantified in relation (Lăcătaşu, 1995):

$$\boldsymbol{M}_{\textit{total}} = \left(\boldsymbol{M}_{p} + \boldsymbol{M}_{a} + \boldsymbol{M}_{f} + \boldsymbol{M}_{ac} + \boldsymbol{M}_{ow} + \boldsymbol{M}_{ip}\right) - \left(\boldsymbol{M}_{cr} + \boldsymbol{M}_{l}\right)$$

where:

M- represents heavy metals, p-parental material, a-atmospheric deposits, f-fertilizers, acagricultural chemical products, ow-organic waste, ip- other inorganic polluting substances, Cr-cropping losses, l- leaching and volatilisation losses.

Apart from the total heavy metal content of soils, the available concentrations depend on the chemical elements in the soil that control metal species; they also depend on elements of vegetation.

While these polluting substances are harmless at low concentrations, the soil can turn into a polluting element for water or vegetation if a certain value is exceeded (Pacyna, 1987).

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## **EXPERIMENTAL PART**

The soil samples were gathered from different depths depending on the vegetation of the respective soil: 0-20 cm for arable land and grass, 20-40 cm for orchards and vineyards.

For each material studied, we used the specific methods mentioned in our literature and complied with the experimental parameters and requirements of our study procedures (Popa, 2003).

We gave special attention to calibration. In order to determine metallic cations found in soil and vegetation samples by using atomic absorption spectrophotometric methods, we used the standards provided by AnalytikJena (which came with the equipment). Through proper dilution, we arrived at the optimum concentration (100 ppb) for GFAAS and 100 ppm for FAAS (see Table 1).

## METHODS AND EQUIPMENT

Through the electrochemical method (stripping), determinations were conducted on mercury drop electrode using the computerized system BAS 100W. It is equipped with a classical cell, composed of three electrodes (EL- Hg drop; ER-Ag/AgCl; CE- Pt high surface spiral). All the samples were deaerated for 15 minutes before each determination. The measurements took place in the electrolyte mentioned in the specialty literature (HME): 117 g NaCl, 35.5 g ascorbic acid, 7.7 g NaOH, all dissolved in 500 mL ultrapure water. Water used for determination was obtained with the equipment for ultrapure water Mili Q, produced by USF Elga. The experimental parameters for HMDE analysis are as follows: Ei=-400mV; Ef=-100mV; sensitivity  $100\mu A/V$ ; scanning speed=20mV/s; accumulation time=400s; mercury drop size =10.

By using spectrometric methods, we intended both to obtain results close to the specialty literature and to compare the two methods. We used an AAS absorption spectrophotometer, model 6.

The analytical data on which we worked for Cu in the soil are: spectral line  $\lambda = 324,7$ nm;  $\Phi$  (slit) = 0,5; strength of current = 10mA; flame type= air-acetylene; equipment sensitivity = 0,07 ppm. For Pb: spectral line  $\lambda = 217$  nm;  $\Phi$  (slit) = 1; strength of current = 5 mA; flame type= air-acetylene; equipment sensitivity = 0,12 ppm; For Cd: spectral line  $\lambda = 228,8$  nm;  $\Phi$  (slit) = 1; strength of current = 10mA; flame type = air-propane; equipment sensitivity = 0,03 ppm;

METHOD OF ANALYSIS	EQUIPMENT USED	MEASURED ELEMENTS	Sample preparing methods
Atomic absorption spectrophotometry <i>applied to soil</i> <i>samples</i>	AAS vario2 FL, AAS vario5 GF, Germany	Cu <sup>2+</sup> , Pb <sup>2+</sup> , Cd <sup>2+</sup>	Wet mineralization
Accumulation – dissolution methods, <i>applied to soil samples</i>	PTEA-Wagtech Computerized Electrochemical SystemBAS 100W;	Cu <sup>2+</sup> , Pb <sup>2+</sup> , Cd <sup>2+</sup>	Wet mineralization
Titrimetry, applied to soil samples	Automatic burette SCHELLBACH, Germany	organic C (humus)	Wet oxidation
Colorimetry, applied to soil samples	SPEKOLL UV-VIS, Analytik Jena, Germany	Phosphorus (P)	Ammonium lactate acetate extraction
Flame photometry, <i>applied to soil</i> samples	FLAMFO 4, AnalytikJena, Germany	Potassium (K)	Ammonium lactate acetate extraction
Instrumental pH determination, applied to soil samples	INNOLAB, Germany	рН	In watery suspension

#### Table 1. Methods used for the analysis of soil heavy metal contamination

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