~200g/l) that results in high unselective absorption in ETAAS and high background emission in ICP-AES. Multielement preconcentration with separation of matrix is an effective solution of this problem.

Electrothermal atomic absorption (ETAAS), inductively coupled plasma atomic emission spectrometry (ICP-AES) and off-line sorption preconcentration have been used for the development of new sensitive and selective methods of heavy metals determination in the salts and waste brines. Measurements were carried out using atomic absorption spectrophotometer Perkin-Elmer Zeeman/3030 Model with HGA-600 electrothermal atomization system, for multielement measurements the ICP - AES polychromator Jarrell Ash ICAP 9000 was used. Device for flow injection sorption preconcentration BPI (Aquitia, Russia) with 4 micro columns packed with DETATA sorbent (aminocarboxylic on polysterene base) was used for separation and preconcentration of heavy metals.

There are many factors that influence effective FI preconcentration: content of salts, sorption and desorption flow rates, sorption and desorption times (ore volumes of the sample and eluent), pH, acid concentration in eluent, kind of eluent, etc. The parameters of simultaneous multi-element sorption, elution, and signal registration have been optimized. The parameters of flow sorption ICP - AES analysis of the samples with complex matrices were investigated and the analytical procedure for simultaneous determination of Cd, Zn, Pb, Cu, Ni, Mn, V, Ti, Al by ICP-AES in industrial brines and salts was developed.

For routine laboratories methods of ETAAS determination of elements in concentrate solutions are developed and proposed for validation as standard methods. Using reference brines the bias, precision and accuracy of methods were determined according to Standard Procedure for Certification of Measurement Methods. Sensitivity of proposed methods is better for the factor of 10 - 100 than that for the certified methods used in salt industry now.

**FRACTIONATION OF WATER SOLUBLE FORMS OF HEAVY METALS IN SLUDGE BY FLOW INJECTION SORPTION - ICP-AES**

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*Key words: fractionation, sludge, flow sorption, inductively coupled plasma atomic emission spectrometry*

The content of water - soluble and mobile species of heavy metals in wastewater and potable water sediments is the major criterion for its subsequent utilization. Determination of the species content is generally conducted by the techniques, which are similar to those applied to soils. It is well known that humulinic acids fix trace elements in soil extracts. Wastewater sediments significantly differ by chemical composition (especially by nature of organic matter) from soil ones. The purpose of long-term treatment of these residues is a transformation of organic substances to humus. Dynamics of alternation of the organic to inorganic species ratio can be chosen as an indicator of the treatment process.

A technique for the determination of organic and inorganic species of heavy metals in aqueous extracts of wastewater and potable water sediments has been developed for the monitoring. Aqueous extracts were prepared according to standard method. In order to separate the species a technique of flow sorption preconcentration and separation (dynamic sorption) was
applied. Organic species were isolated using Diapac C16 sorbent (cross-linked silica gel of 15 mM/g capacity). For separation and preconcentration of inorganic species DETATA (aminocarboxyl agent on polypropylene substrate) and Chelex-100 sorbents were used.

Determination of Cu, Zn, Pb, Cd, Ni, Co, Fe, Mn, V, Ti, Cr, as well as Na, K, Ca, Mg, Sr, P, Ba in the initial sediment samples, aqueous extracts, methanol and acid eluats was carried out by ICP - AES technique. The total content of elements in sediments was determined by ICP-AES after treatment with oxidizing mixture in autoclaves (T ~ 220 °C, P ≤ 200 ATM).

The content of organic matter was measured by COD test (Chemical Oxygen Demand). The oxygen equivalent of the organic matter that can be oxidized was measured by using solid electrolyte analyzer, developed and patented in GEOKHI. Method is based on controlled high temperature oxidation of organic by the oxygen with inert gas addition. This method is very useful because the COD can be determined in twenty minutes, compared with three hours for the potassium dichromate method.

The method developed was used for the monitoring of organic and inorganic species for the sediment samples with different storage time. The initial treated sludge consists of suspension of organic and mineral substances; the organic part contains carbohydrates, bacterial biomass, lipids, lignin, and petroleum and humidic matters. For comparison the samples of portable water sediments obtained in the process of purification of natural using aluminum hydroxide coagulation were investigated. These sediments consist of organic matter (50%), mainly of humus. Special sample of sediment, which was certified as reference material in scope of the IMEP-14 program, has been analyzed using the elaborated technique.

DIRECT ATOMIC ABSORPTION SPECTROMETRY ANALYSIS OF SOLIDS AND POWDERED MATERIALS. SOME VIEWS ON QUESTION OF METHODOLOGY AND STANDARDIZATION

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Key words: ET AAS, solids, evaporation, atomization, refractory metal oxides, certified referents materials.

The main part of papers devoted to direct electrothermal atomic absorption spectrophotometry (ET AAS) analysis of solid and powdered materials are based on the usage of commercial versions of graphite atomizers (HGA, CRA) and introduction on its no representative mass (several mg) of analytes to be investigated or its suspensions. Simultaneously with it in the most cases there was marked a problems of matrix interference on the results of micro impurities determination. At the same time in several papers which dedicated to direct ET AAS analysis some of solids: refractory metals and its oxides, ores, concentrates, soils and etc. it has been propose to previously diluted analyzing samples by high-purity graphite and use a powdered carbon with certificated contents of elements as standard reference materials. The authors of these work have assumed that at direct ET AAS determination of micro quantities of highly- and medium-volatility elements in small mass (several mg) of low-volatility, refractory materials there are the fractional and its full vaporization takes place and thus matrix components does not interfered on value and process of their analytical signal formation.