

## ANALYTICAL CHEMISTRY FOR ENVIRONMENTAL DIAGNOSIS AND TO ASSIST THE NUCLEAR FUEL CYCLE SAMPLES

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In the Laboratories of Environmental Diagnosis Division - MOA, methodologies were established for evaluation of physical-chemical, chemical and toxicological parameters to support several research projects in development for environmental diagnosis (FIG.1) and to assist the Nuclear Fuel Cycle Program. All these methodologies adopted are the ones established in the standard methods (ASTM, EPA) or those specified by the contracting. The classic methods and instrumental techniques analysis such as atomic absorption, inductively coupled plasma emission spectrometry (ICP-OES), ion chromatography; gas chromatography (GC), gas chromatography mass spectrometry (GCMS), high performance liquid chromatography (HPLC; HPLC/MS/MS), X ray fluorescence (WD-XRFS); differential pulse anodic stripping voltammetry (DP-ASV) have been used. The members of Division have been participated in several international programs sponsored by: CETAMA (Etablissement Commission des Analyse Méthods, France), IAEA (International Atomic Energy Agency, INTI (Instituto Nacional de Tecnología Industrial, Argentina); SENAC (Brasil) e ABACC (Agência Brasileiro-Argentina de Contabilidade e Controle de Materiais Nucleares; Rede Metrológica Rio grande do Sul; Departamento de Controle Sanitário e Ambiental - SABESP (BRASIL/Estado de São Paulo). The Quality handbook according to ISO GUIDE 25 has been elaborated. The XRF Laboratory have been participated of Quality Control in Analytic laboratories of the AIEA- ARCAL LVXXI Program - Teste de Aptitud). Several methodologies were established to support research projects in development:

1. The specificity and sensitivity in the pesticides analyses were enhanced by a new methodology using solid-phase extraction followed by high performance liquid chromatography coupled with tandem mass spectrometry (SPE-LC-MS/MS). The impact of pesticides use in agriculture was verified (FIG.2).

2. Preparation of organic matrices for determination of trace elements by differential pulse anodic stripping voltametric: In this studied, the content of zinc, cadmium, lead and copper was determined in adults diet samples and bovine liver, applying the DP-ASV technique. The established methodology was validated with the use of a certified sample as reference (NIST 1577b). In the digestion of the matrices in acid medium, conventional methodologies were used, conductive heating in open recipients and equipment with microwaves source in open and closed vessels.

3. Evaluation of arsenic, selenium, antimony and mercury in water and sediments samples by Atomic Absorption Spectrometry (AAS, HG AAS; CVAAS) and inductively coupled plasma optical emission spectrometry (ICP-OES). Arsenic and selenium are associated to some human metabolic processes, however, they become toxic when present at high levels. The most recent legislation of Brazilian Health Ministry established a limit of 10  $\mu\text{g L}^{-1}$  for these elements in human consumption destination water.

4. Evaluation for quality control of metal and rare earth impurities of Nuclear Fuel Cycle Program ( $\text{U}_3\text{Si}_2$ ,  $\text{UF}_4$ ,  $\text{U}_3\text{Si}_2\text{-Al}$ ) using extraction chromatograph and ICP-OES.

5. Development of method for Si and U determination in  $\text{U}_3\text{Si}_2$  system by X ray fluorescence and gravimetric/volumetric techniques. This method is applied to the assembled fuel elements.

6. Evaluation of water quality for the IEA-R1.



FIGURE 1 - Analytical and environmental laboratory.



FIGURE 2 - Environmental Diagnosis São Lourenço River - SP.