

## 8 Summary

Trace elements are important because of their association with environmental issues and the health of plants, animals, humans and also play significant role in science and technology. Good quality measurements are essential to control and maintain products and processes quality, both in manufacturing, trade and in research. Therefore, nuclear and analytical techniques have been acclaimed as the most versatile analytical tools for quantitative multielement analysis. In case of nuclear detection technique radioactive elements can easily be detected in trace or ultra trace level employing their suitable gamma spectroscopic properties.

Within the scope of the thesis, methods for the extraction of some elements from fourth period of the periodic table in their no-carrier-added (nca) state have been developed employing radioanalytical methodologies. In case of trace elements analysis by inductively coupled plasma optical emission spectrometry (ICP-OES), in most of the cases samples are in solution form and, therefore, sample preparation methods are designed to transform solid samples into solution. Closed-vessel microwave digestion is a popular wet acid digestion procedure to achieve this step. Microwave digestion procedure has been widely used in analytical chemistry for any types of samples, such as geological, biological and environmental samples. This technique can supply not only dramatically decreased digestion time, but also reduction in contamination, less reagent and sample usage and suitable for samples that are very difficult to digest and contained volatile element. It is safer than traditional heating digestion technique because of prevention of contact and production of fumes. The scope also includes studies on the behaviour of trace elements in multielement environment.

An improved non-destructive method of potassium determination experiment requires neither chemical treatment of the sample nor irradiation of the sample in a nuclear reactor. It even does not require any excitation source for determination of potassium. The proposed method was further validated by measuring K in the SRM samples by ICP-OES and WDXRF. The method therefore truly follows green chemistry principle, minimizing the chemicals and other hazards. In this experimental condition we have detected potassium sample as low as 10 mg.

The total amount of K in Spinach (*Spinacia oleracea*), Red amaranth (*Amaranthus cruentus*), Green amaranth (*Amaranthus tritris*) and Jute leaves (*Corchorus olitorius*) leaves have been measured with the help of the Saha Institute of Nuclear Physics Compton Suppressor Spectroscopy (SINP-CSS). It has been found that raw spinach, Red amaranth, Green amaranth and jute leaf contain 3.94, 3.04, 3.46 and 2.46 % of K. After boiling in deionized water for 20 minutes, the dried samples were studied and the percentage of K was determined. The dried samples contain 1.05, 1.28, 0.81 and 0.42 % of K, respectively. After boiling, amount of potassium content in all leaf vegetables decreased significantly.

The liquid anion exchanger trioctylamine (TOA) dissolved in cyclohexane was employed for the separation of  $^{61}\text{Cu}$  from bulk cobalt in HCl solution. It was found that at 0.1 M TOA and 2.5 M HCl, 71 %  $^{61}\text{Cu}$  was extracted into the organic phase with 4% contamination of bulk cobalt. Another series of extraction was carried out with varying concentration of TOA keeping HCl concentration fixed at 2.5 M. In this condition extraction of copper into organic phase increased with increasing concentration of TOA. When liquid-liquid extraction was carried out by varying concentration of cationic ion-exchanger, HDEHP and keeping fixed ammonium hydroxide concentration, extraction of copper and cobalt shows same behaviour in both phases.

Solubility plays an important role for all chemical studies. We have studied the solubility of  $\text{GeO}_2$  in various strengths of  $\text{HNO}_3$  and HCl with the help of inductively coupled plasma optical emission spectrometry (ICP-OES). The Handbook of Chemistry and Physics says  $\text{GeO}_2$  is insoluble in concentrated HCl and no information is given on its solubility in other acids. We observed that the solubility of  $\text{GeO}_2$  in concentrated HCl and  $\text{HNO}_3$  are as high as 17.5 mg and 70.3 mg per litre of the respective acids and solubility also changes with changing  $\text{HNO}_3$  acid strengths.

We have also reported the production and radiochemical separation of nca  $^{73}\text{As}$  and  $^{75}\text{Se}$  from  $^7\text{Li}$  irradiated germanium oxide target.  $\text{GeO}_2$  is insoluble in concentrated HCl. Therefore,  $\text{GeO}_2$  was dissolved in microwave digestion system. The resulting solution was used for radiochemical as well as ICP analysis. The radiochemical separation was carried out using TOA dissolved in cyclohexane as a liquid anion exchanger. The best separation of  $^{73}\text{As}$  from  $^{75}\text{Se}$  was achieved at 0.1 M TOA and 10 M HCl. In this condition 99.6% nca selenium radionuclide was extracted into the organic phase and 99.3%  $^{73}\text{As}$  remained in the aqueous phase. Another series

of extraction was carried out by varying concentration of TOA keeping HCl concentration fixed at 10 M. The back-extraction was done by 1 M NaOH and 0.1 M DTPA in 1M NaOH to get back Ge and Se, respectively, in the aqueous phase. The aqueous fraction containing  $^{73}\text{As}$  was checked by ICP-OES to examine absence/presence of Ge in this fraction and Ge was found less than 0.1 ppm. It may be noted that in the initial solution Ge concentration was 32.49 ppm.

To see the uptake behaviour and interdependence study of arsenic and selenium in Mung bean, various strength of As(III) and Se(IV) solutions were added to the medium. It was observed that seedlings were not germinated at all by addition of 15 ppm As solution (volume 15 mL) to the sand bed. Even addition of 1.5 ppm As solution resulted in slight wrinkling of the leaves. Whereas normal growth of seedlings were observed upto 9 ppm Se, and upto 15 ppm concentration of Se, only slight hindrance in growth was observed. Therefore, we took solutions of As from 15 ppb to 1.5 ppm; while 1 ppm to 15 ppm Se solutions were taken. We have shown that the concentration of As and Se in root and shoot part in a common plant, Mung (*V. radiate*) shows migration and bio-magnification behaviour of As and Se to human body. The resulted data indicate that the addition of 150 ppb to 1.5 ppm solution shows uptake of As in Mung beans and the concentration of As was magnified up to the level of 0.15 to 2.6 ppm and relative uptake of Se in shoot was less than that of root. Maximum Se concentration in root was ~ 250 ppm. Further, Se accumulation was reduced in presence of minute amount of As while As uptake was higher in presence of Se.

We believe that our experimental techniques would provide sufficient guideline on cost effective, rapid, clean, less hazardous and reliable radiochemical separation for the production and separation of elements of the periodic table in their no-carrier-added (nca) state and quantitative multielement analysis at trace and ultratrace scale and also provide easy sample preparation by microwave digestion system for any types of samples. The thesis also motivates to see the behaviour of trace elements in multielement environment and evaluates the level of environmental pollution.