
Trace element analysis of selenium

Mahmoud Shahat Ahmed Soliman

The present thesis divided into four chapters as follows: Chapter (1): This chapter contains an introduction on different oxidation states of organic and inorganic species for selenium in environmental and biological samples, the process for separation of selenium from these samples and the importance of selenium as a component for these samples. Also, it gives notes about the techniques which are used in the elemental analysis for selenium species and the detection limits for selenium in these techniques, selenium species in human body and the importance of these species in protecting the body from the different types of cancer and the sources of selenium in environmental samples (soil and water) and distribution levels of selenium in these samples. This chapter contains short notes on Inductively Coupled Plasma Mass Spectrometer (ICP-MS) and different types of this technique which are used in measuring selenium species and the process of interference between this technique and other techniques to improve selenium measurement. Also, the connection between hydride generation as introduction system with atomic absorption spectrometry to generate hydride for the volatile elements for groups IV, V and VI to measure it in high temperatures. Chapter (2): This chapter is divided into two parts: The first part deals with the sample collection process for environmental samples (underground water, soil) and the wet digestion (microwave digestion) process of soil samples. Also, it contains the theory of work of the closed microwave digestion system. The second part contains detailed information concerning the theoretical considerations of the used analytical techniques. These techniques include Hydride generation - Atomic Absorption Spectrometer (HG-AAS), Inductively Coupled Plasma Mass Spectrometer (ICP-MS) and Neutron Activation Analysis (NAA). Chapter (3): This chapter includes the methods of sampling, sample preparation, and sample digestion. The measures of quality assurance are discussed in this chapter. It describes in details the closed microwave digestion technique and the analytical methods used in this study which are present in Central laboratory for elemental and isotopic analysis (CLEIA) and the Egypt second research reactor (ETR-2). The described techniques are atomic absorption spectrometer (AAS 6 vario, Analytical Jena GmbH, Germany), JMS-PLASMAX2 mass spectrometer (ICP-MS) and the Egypt second research reactor (NAA). Chapter (4): This chapter contains the results of the analysis of selenium in soil, underground water and toenail samples and the main component and trace elements for these samples, also contains the general presentation of the results of the intercomparison as tables of results for each analyte, as well as graphical representations of results and as a summary table. The terms used in the tables are

defined in this chapter. This chapter also discussed in details the techniques able to detect selenium and the other techniques which are not able to detect it but used for the purpose of comparison:

- o The EDX and XRF is used only for solid samples (soil and toenails). Both these techniques were used to detect the trace elements present in toenail samples. Both of these techniques were not so useful for the detection of selenium, because selenium detection limit is higher than below mentioned all trace elements.
- o ICP-MS was used for the detection of trace metals in soil, under ground water and toenail samples, the concentration of trace elements is within the predictable concentration except selenium. The concentration of selenium is lower than the predictable concentration which appears by measuring known standard of selenium because hexavalent selenium is not stable at higher temperature and it doesn't generate a measurable signal in any situation.
- o Hydride generation atomic absorption spectrometer (HG-AAS) is the perfect technique to measure Se. NaBH_4 and hydrochloric acid was used as a reducing agent to reduce Se (VI) to Se(IV) and this applied to all liquid samples.

In this chapter selenium concentration between the underground water and soils from the same sites don't give any direction of the same way or attitudes. This means that the nature and composition of soil is different from that of underground water samples for these elements. This also appears by comparing the trace element (Cr, Fe, Mn, Pb, Se and Zn) in the samples under investigation by ICP-MS technique. Also we can see that the area from which we have collected our sample is found to be in the normal range of distribution of selenium in underground water and soil samples. In case of healthy people selenium present in the normal range and also comparing the results obtained by using HG-AAS and INNA results showed great agreement.