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IMPORTANCE OF TRACE ANALYSIS

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Abstract

Determination of trace elements is important and required for many applications. In nuclear technology, for example, the chemical characterisation of different materials (nuclear fuels, reactor materials, nuclear waste streams etc.) with respect to trace elements is a necessary quality assurance step. Critical trace constituents like boron which has high thermal neutron absorption cross section, need to be determined with the highest possible precision and accuracy for a rigorous quality assurance. Even though there are analytical techniques available for this purpose, but there is enough scope for further improvement in the existing methodology either in terms of extending their applicability or eliminating known limitations. In addition, many of the sophisticated instrumental analytical techniques require matrix matched reference materials for validation and quantification of analytical results. Many times these matrix-matched reference materials are not either available or accessible. The only recourse is to validate the analytical results by as many techniques based on independent theoretical principles as possible.

Keywords: *Trace elements, gravimetric analysis, hydel resources, thermal neutron absorption.*

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Result and Discussion: The analytical requirements imposed by the minute quantities and typically complex matrices involved have led to the development of methodologies and instrumentation so specialized as to warrant consideration as a distinct field of analytical chemistry - trace analysis. In the beginning of the 19th century "trace" is defined as any element whose concentration in a certain medium is too low to be quantitatively determined. In gravimetric and volumetric analysis, it is still usual to designate all concentrations lower than the sensitivity limit of the analytical method as "traces" or "content too low to be determined". Due to advances in science and technology, the minimum detectable concentrations in a given medium keep decreasing. Consequently, the concentration levels formerly designated as "traces" now fall within the range of concentrations which can be determined by instrumental methods like spectroscopic methods, electrochemical methods and nuclear analytical methods. In general "trace analysis" refers to the determination of element of interest in any matrix which is containing less than 0.01 wt%. Depending on the content of element of interest in matrix, it is considered as major, minor, trace and ultra trace element. They are characterized as a following way

Major: >1 wt%

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Minor: <1 wt% and >0.01wt%

Trace: <0.01wt% and >0.0001wt%

Ultra trace: <0.0001 wt%.

Importance of trace analysis

It is a well established fact that the physical properties of metals and alloys can be profoundly affected by extremely minute concentrations of certain elements. The list of metallurgical properties that are significantly altered (for good or otherwise) by residual level of elements is quite extensive. In most of the cases, these effects are deleterious. Residual elements in the major matrix may be due to the result of unwanted contamination from extraneous environment from materials that contact the product during processing or storage. There are also cases where, trace elements are known to cause beneficial effects and are intentionally added to alloys either for their own influence or for the synergistic influence they exert on the effects of other alloying additions. Nowadays trace analysis is one of the factors to improve the economy of a country. Trace analysis is required to characterize very pure materials that are essential to prepare good quality electronic goods. Also trace analysis is essential to assess the quality of food products and to monitor the environmental contaminants. Trace analysis also plays key role in chemical quality control of nuclear and reactor materials in nuclear technology.

Importance of nuclear technology in India

With the growth of industrial development and domestic requirements, there is need for a substantial increase in the energy production in India. In the coming decade, the installed capacity is required to be doubled from the present capacity of about 1,00,000 MWe. The conventional methods of power production are coal and hydel based. The environmental concerns and the substandard quality of Indian coal will not permit larger installed capacity of thermal plants. The hydel resources are limited to 35,000 MW capacity and involve environment and human resettlement problems. Presently non conventional energy resources like wind and solar energy are highly cost intensive as the cost per kW of power is too high to be economically viable. Nuclear power is poised for a renaissance and is an inevitable option for a large developing country like India to meet the ever increasing demand of electricity, at an affordable cost to the common man and without degrading the environment in terms of global warming. Nuclear energy is an important clean and environmental friendly source of energy.

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It has potential to meet the demands of energy for the coming few hundred years. India has embarked on a three stage nuclear power programme, which is based on utilization of natural uranium as nuclear fuel in the first stage i.e., pressurized heavy water reactors, utilization of plutonium which is generated in first stage in second stage i.e. fast breeder reactors, for breeding of 233U from 232Th. Finally utilization 233U in third stage as a fissile material along with thorium, uranium and plutonium in advanced heavy water reactors.

Importance of trace analysis in nuclear technology

In nuclear fuel cycle activities, in the front-end cycle, chemical characterization of nuclear fuels and other reactor and structural materials for trace elements is indispensable. The performance of nuclear fuels critically depends on the presence of elements with highthermal neutron absorption cross sections as their presence is detrimental to the continued operation. Chemical quality control provides a means to ensure that the quality of the fabricated fuel conforms to the chemical specifications for the fuel laid down by the fuel designer. These specifications are worked out for the major, minor and trace constituents which affect the fuel properties and hence its performance under conditions prevailing in an operating reactor.

Nuclear reactor design incorporates detailed specifications of different systems, which must be satisfied for smooth and efficient functioning of the reactor. Fuel being the heart of the reactor, its chemical characterisation is an important component of this design. Both the fuel materials and finished fuel products are to be characterised for this purpose. Each fuel batch has to be subjected to comprehensive chemical quality control for trace constituents, stoichiometry and isotopic composition. Presence of trace elements in nuclear materials affects the nuclear reactor operation significantly. This is mainly because of adverse changes in neutron economy, fuel integrity, thermal and mechanical properties and failure of clad occurring due to presence of some of the trace constituents. In order to attain reliable, safe and efficient nuclear reactor operation there is need to monitor and control the trace constituents in nuclear materials prior to their use. Hence control of trace constituents in the fuel is necessary to obtain the designed burn-up. Certain upper concentration limits have been specified for a number of trace elements in nuclear fuel materials. These are called specification limits. It is interesting to note that during the time of first nuclear reactor construction in USA in 1940's, designers, with whatever knowledge and expertise available with them at that time and in consultation with nuclear physicists, technologists and chemists laid down these specifications. It is remarkable that these specifications are accepted even today. In particular, boron, which is having very high thermal neutron absorption cross section, has very stringent specification for all nuclear and reactor materials. Natural boron contains about 20% of 10B. The thermal neutron absorption cross-section (σ) is 3846 barns for the reaction 10B (n,α) 7Li, due to which lot of gaseous (helium) product is formed. This reaction affects the (i) neutron economy and (ii) may lead to structural changes/damages to reactor materials. Hence, all the nuclear materials have stringent specifications for boron (it is less than 1 μg.g-1 for fuel material in thermal reactors). Therefore, an accurate knowledge of boron content in nuclear fuel materials and other reactor materials is essential. While boron presence is detrimental in some cases as mentioned above, it is also used for controlling the excess reactivity for safe operation of the reactor. Boron is one of the elements to possess nuclear properties, which warrant its consideration as neutron absorber material. Boron and its compounds are extensively used in nuclear industry for application as control rod, human shielding against neutrons and as sensor elements. Neutron absorption of boron is sufficiently high in the low neutron energy range to make it an excellent candidate for use in thermal reactors. At higher energies, the cross section of most of other elements become very small, often abruptly as in the case of cadmium, whereas that of 10B, decreases monotonically with energy. Absolute cross sections for neutron absorption along the entire energy spectrum are of sufficient magnitude to make it very effective in the intermediate and also in the high energy range. Boron has another advantage over other potential neutron absorption materials. The reaction products of neutron absorption namely helium and lithium are formed as stable, non radioactive isotopes. As they do not emit nuclear radiation, decay heat problems during reactor shutdown and transfer of depleted control rods are minimal. Considering these attractive properties of boron, boron doped zirconium niobium alloys are used for controlling the excess neutron reactivity in advanced nuclear reactors. For effective utilization of these materials and for absolute neutron reactivity calculation inside nuclear reactor core, boron content has to be determined in these materials. In addition to the total boron content, its distribution over entire length of material rod is also essential for nuclear physics calculations. An accurate knowledge of boron content with its associated uncertainty is absolutely required for the reactivity calculations. In the back-end of fuel cycle, the radioactive waste generated contains host of radionuclides produced in nuclear fission may have environmental concerns. Cesium and

strontium are two important fission products present in this radioactive waste that are responsible for MANREM problems. These are long lived and heat generating radionuclides.

It is estimated that the cumulative spent fuel, arising from existing nuclear reactors all over the world, could be around 3.5 x 105 tons by the year 2010. In this, the yearly yield for 137Cs alone is estimated to be around 27 MCi [1]. Due to long half life (t1/2 = 31.2 year) and reasonable thermal output (0.42 W/g), 137Cs has a potential application as the source in gamma irradiators, which are used in environment pollution control, food preservation and sterilization of medical accessories. Due to the toxicity of cesium and its ability to displace potassium from muscles and red cells, removal of cesium from medium and low level nuclear wastes is also desirable. Removal of 137Cs from nuclear waste facilitates the safe and less expensive methodologies for disposal of high-level waste in deep geological repositories asvitrified waste. Hence lot of research is going on to develop a suitable reagent or technique to remove effectively the Cs from nuclear waste. To assess the separation efficiency of a technique, Cs has to be determined.

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