ACTIVATION ANALYSIS WITH REACTOR NEUTRONS

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ABSTRACT

The potentialities of neutron as an analytical probe are indicated, pointing out the need for development of other approaches, besides the conventional activation method. Development of instrumental approach to activation and applications, carried out at Analytical Chemistry Division are outlined. The role of, and the need for, the development and application of mathematical methods in enhancing the information content, and in turn the interpretation of the analytical results, is demonstrated.

INTRODUCTION

Neutron activation analysis is a well recognised analytical competence, capable of determining, simultaneously, several elements, at nanogram levels in a variety of matrices. The high flux of neutrons, available in research reactors, has enhanced the attainable sensitivity, while the advent of high resolution detectors and the matching electronics has greatly improved the specificity of measurement and the (analytical) information content of gamma-ray spectra. The minimal handling of samples, prior to excitation (irradiation) and the absence of "reagent blanks" have made the nuclear activation technique as a reference method in trace analysis; the instrumental approach, in particular, has (played) a vital role in providing validation support for analytical programmes.

ANALYTICAL APPROACHES

The advances in nuclear spectrometric measurement and data acquisition and processing capabilities have not been, however, backed by the developments in facilities for excitation ("irradiation") with neutrons from a reactor for analytical applications; almost all the work has been in conventional activation. The potentialities of neutron as an analytical probe are shown in Figure 1. There have been very few reactor facilities with provision for carrying out other approaches using neutrons. While all these other approaches have been looked at in isolated instances, there is a need for systematic work to develop these approaches to the level of well validated analytical competences. It is recognised that these approaches are not as versatile and "simple" as neutron activation, but with the accepted role of nuclear analytical techniques in providing validation support, it is essential to develop these approaches.

FACILITIES FOR IRRADIATION

The facilities available for neutron activation at Trombay are given in the accompanying table.

It is essential to characterise the irradiation positions at reactors, particularly with reference to the extent of interferences from threshold reactions to thermal neutron activation analysis; the most used irradiation positions at Apsara and CIRUS have been characterised[1]. A new facility, in the form of a 'Central Water Hole'[2] has been created at Apsara, which provides a higher flux by a factor of three (with twice the fast component) than that available in the earlier core configurations. This high flux irradiation in combination with the use of Ge(Li) detector with $>$ 25% relative efficiency has enabled the estimation of impurities in the zone refined silicon at parts per billion level, with detection limits at $<$ 1 ppb for several elements.

METHODOLOGY

The analytical methodology adopted has been validated through the analysis of a variety of certified reference materials and more importantly, through continued active participation in several of IAEA's intercomparison exercises. The methodology followed has been described elsewhere[3,4].

One of the major requirements in instrumental activation analysis is to ensure adequate specificity of the radiation measurement; this is particularly so when the products are positron emitters. In order to enhance the specificity of their measurement, above that offered by the 2 detector (180°) coincidence of 511 keV radiation, a triple coincidence system was set up with three detectors, at 120° to each other to measure the triplet state annihilation of the positrons. This system not only provides the enhanced specificity[5] but has the potential for providing chemical state information of the element as well, as shown[6,7] by the different extent of formation of positronium in compounds containing copper in different chemical environments. This observation has been substantiated by doppler broadening and angular correlation measurements, in addition to the effect of magnetic field on angular correlation and peak to valley ratios.

APPLICATIONS

The disciplines in which this technique has contributed significantly include cosmic and earth sciences, environmental and life sciences, natural resource exploration, production and quality control in industrial processes (particularly for high technology materials) and also such social and aesthetic areas like forensic science and archaeology. The forensic and archaeological applications carried out are described elsewhere[8]. The application to environmental sciences has brought out the need for proper interpretation of the data generated. The study of trace elements in hair to investigate the role of hair as an indicator of environmental exposure to inorganic pollutants has clearly brought out this requirement. A view of the role of hair in the scheme of monitoring exposure is depicted in Figure 2. In addition to the development of analytical methodology for the determination of as many as 25 elements in hair, mathematical methods using the pattern recognition (PR) approaches were developed and applied to the analysis of data on hair samples from nationwide student population[9] and general population of metropolitan Bombay area[10]. The details of the PR methods are given elsewhere[11,12].

This approach has clearly established the role of hair as a first level monitor in a multilevel monitoring scheme for environmental exposure to inorganic pollutants.

The synergestic combination of the multielement data (from neutron activation) and the pattern recognition analysis of the data not only enhances the information content of the analytical results, but helps achieve the primary objectives of such analyses as for example: studies in environmental exposure involving (i) classification of the population into groups based on the attributes studied on them, (ii) establishment of causative features that are responsible for the grouping/discrimination observed and (iii) extraction of the sources in the environment and their contributions and the pathways to the system.

The procedures developed so far have helped achieve these objectives, albeit incomplete and with further developments, these objectives should be realised in full to provide an integrated view of the environment and the exposure.

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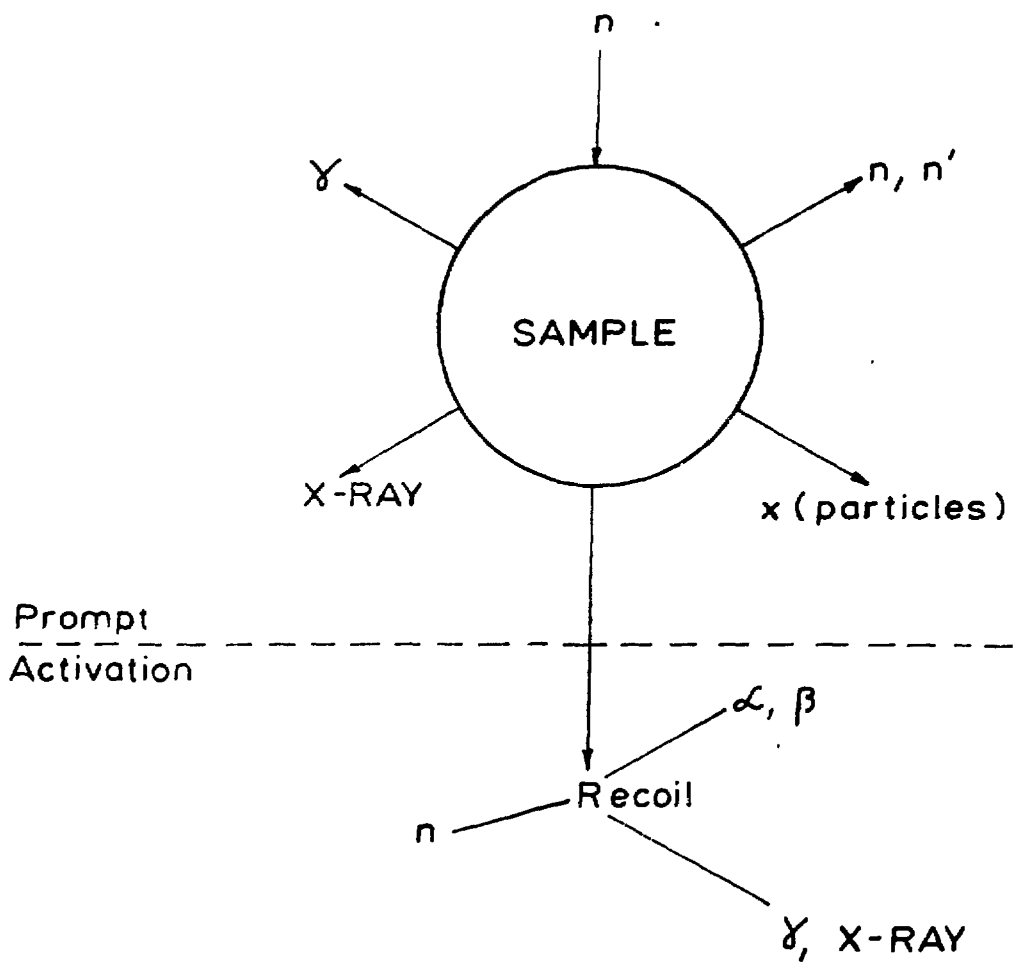
REACTOR NEUTRONS

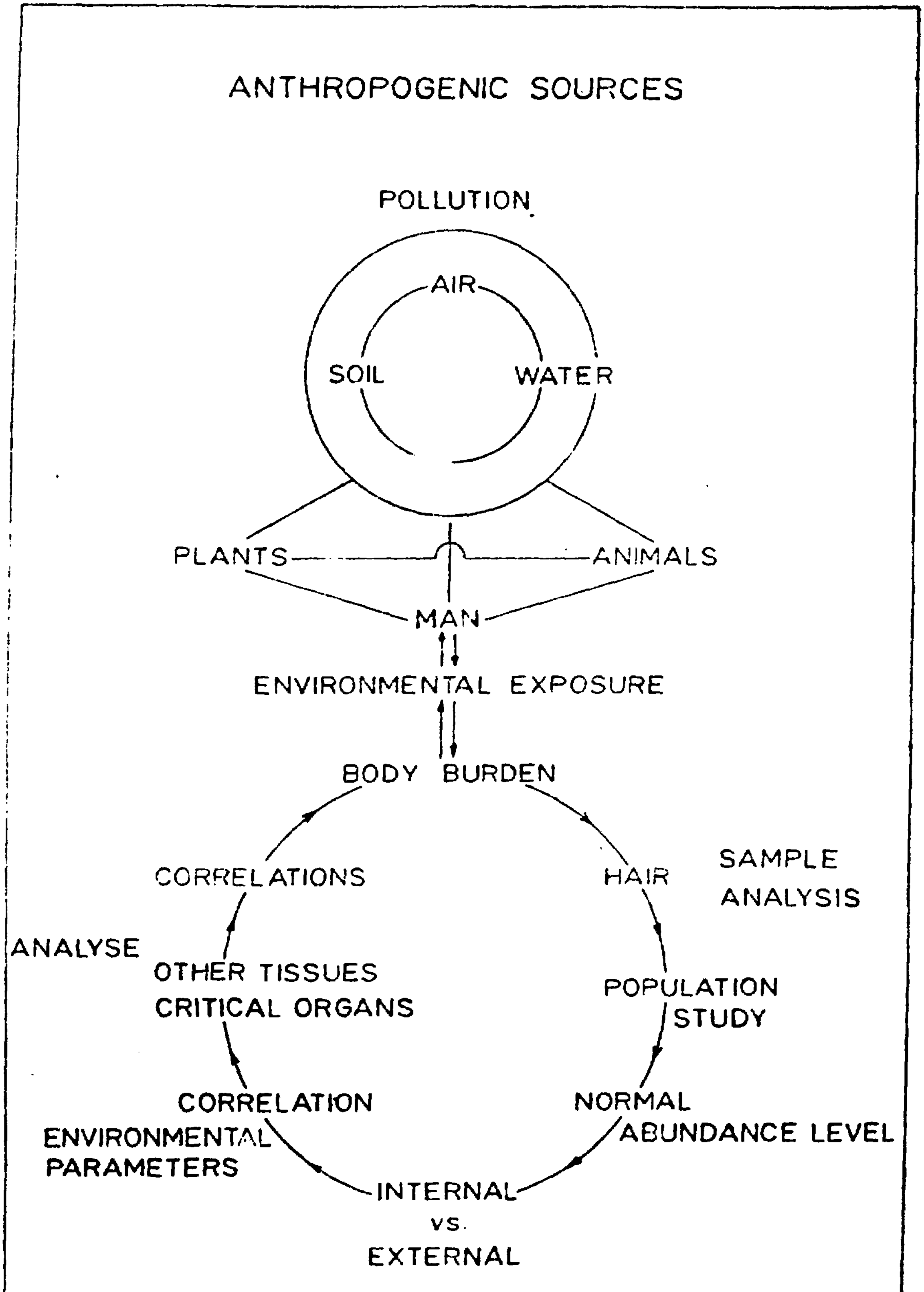
Reactor	Core position: Flux $n.cm^{-2}sec^{-1}$	Core position: Cd Ratio $^{59}Co(n,\ )^{60}Co$	Pneumatic carrier: Flux $n.cm^{-2}sec^{-1}$	Pneumatic carrier: Capsule volume c.c.	Pneumatic carrier: Transit time sec.	Assoc. Labs.
Apsara	1×10^{12}	17	4×10^{10}	3	1	-
CIRUS	1×10^{13}	30	9×10^{12}	2	8	Lab. with F.H. facility
DHRUVA	1×10^{14} (estimated)		1×10^{14}	10	10	2 Labs. & counting room



NEUTRON AS AN ANALYTICAL PROBE - APPROACHES

FIG. 1

Hair - First Level Monitor in a Multi-Level Monitoring Scheme of Environmental Exposure



 AChD BARC

FIG. 2