Trace element analysis in chert samples of paleontological interest using neutron activation

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Introduction

Neutron Activation Analysis (NAA) is an excellent non-destructive technique for multi element analysis for samples with uncertain composition. When the concentration of the element present in the sample is at the ppm level, it can be considered as a trace element and NAA is highly sensitive for the determination of such trace elements with a low background count compared to other analytical techniques like X-ray Fluorescence, Atomic absorption spectroscopy etc. Another advantage is that this technique “sees” material as whole and hence surface changes will not affect the results [1].

NAA is an effective tool for the characterization of sedimentary rock samples where the concentration of trace elements is of paleontological interest. Such studies are underway with 16G Am-Be neutron source (yield 4.0×10^7 neutrons/sec) facility at the Manipal Centre for Natural Sciences (MCNS). The present work describes preliminary results for the trace element characterization of chert samples collected from various locations in Karnataka.

Experimental Procedure

The set up includes a 2"x2" co-axial HPGe detector with a resolution of 0.24% at 1332.5 keV coupled along with a WINSPEC multi-channel analyser. This detector is calibrated for energy using ^137Cs and ^60Co standard sources. A polynomial fit has been carried out to establish a relationship between the channel number (x) and energy (y[keV]= -7.46×10^{-4} + 0.484×x -3.5×10^{-7}×x^2). Similarly, the detection efficiency is measured using ^137Cs, ^60Co and ^152Eu standard sources whose activities are known. The efficiency thus obtained is fitted to a polynomial with natural logarithmic variables and the resulting graph is shown in Fig.1.

Fig. 1: Efficiency calibration curve as a function of gamma energy.

The MCNP code [2] has been used to simulate the results to quantify the composition of elements in the sample.

Results and Discussion

Rock samples collected by the Earth Sciences group at MCNS were taken as test specimens for a pilot study. The samples were cleaned to remove the surface contaminations and weighed before they were irradiated for a day. Post irradiations, the samples were counted for 20 minutes each with the HPGe detector and the counts per second under the characteristic peak of each isotope were used for calculating the trace element compositions in the given sample. As a test case, one sample was chosen which is expected to be fairly representative of samples collected at the so-called K/T boundary (geological time about 65 My ago).
A preliminary gamma ray pulse height spectrum (Energy (keV) vs counts) obtained from one sample shows a predominant peak at 847 keV, which is identifiable as a gamma line emitted by $^{55}\text{Mn}$. This indicates that the irradiated sample contains $^{55}\text{Mn}$ yielding $^{56}\text{Mn}$ through thermal neutron activation. However, $^{56}\text{Fe} \rightarrow \text{(n,p)}$ reaction could have resulted in $^{56}\text{Mn}$ through fast neutron component present in the spectrum. It would be interesting to determine the concentration of both parent nuclides in the sample and determine whether $^{56}\text{Mn}$ comes from the fast or thermal component. Since $^{56}\text{Fe}$ has a fast cross-section of about 6 µb (at 4 MeV, energy dependent) and $^{55}\text{Mn}$ has a thermal cross-section of about 13.3 barns, the latter would appear to be more likely. We assume this here in the absence of a thermalized column of neutrons which would assist in increasing the thermal to fast ratio and reveal peaks hidden within the low energy background. These would include other Group 8 Fe-peak elements, the rare “Platinum Group”, and Iridium the last of which is famously known to be enhanced in samples representing the K/T boundary.

As a first step, the induced activity on the sample due to neutron irradiation is calculated using the following formula

$$A = \frac{\text{cps}}{\varepsilon I_\gamma} \exp(\lambda t_c) \quad (1)$$

Where $\varepsilon$ is the efficiency of the detector, $I_\gamma$ is the photon yield, $\lambda$ is the decay constant and $t_c$ is the cooling time. The measured induced activity of the sample is 7.8 Bq.

To estimate the quantity of manganese in the sample, the induced activity per milligram of $^{55}\text{Mn}$ is estimated to be 0.604 Bq using the MCNP code. From these observations, the quantity of $^{55}\text{Mn}$ present in this sample is determined to be 12.9 mg. Further investigations are underway to interpret this result.

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References