MATERIAL ANALYSIS BY ACTIVATION METHOD "IRON ALLOYS"

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To The

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By

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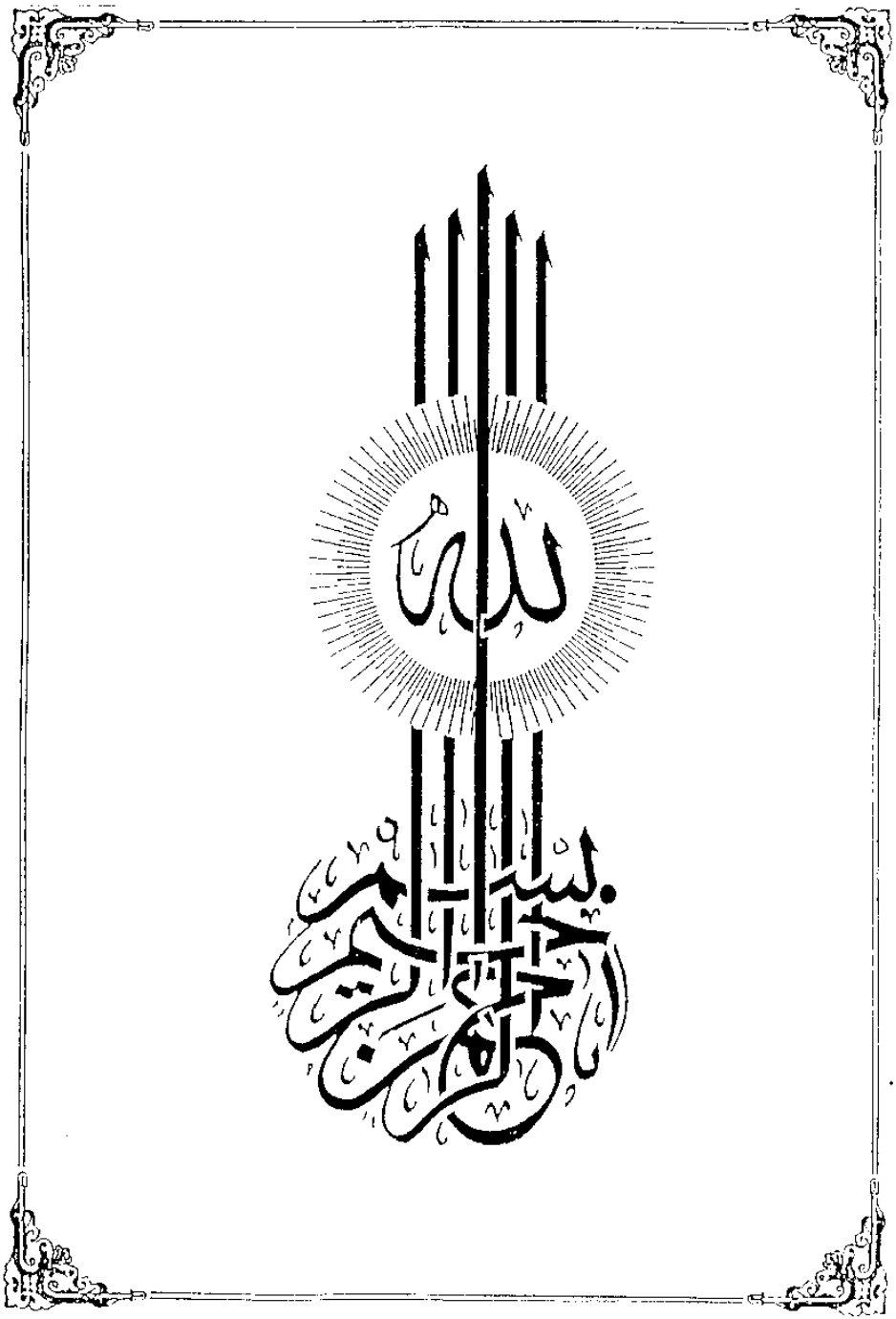
C O N T E N T S



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S U M M A R Y

Activation analysis technique has been applied for the detection and estimation of trace elements existing in three iron alloys. Measurements were carried out by a scintillation counter detector, and a Li-drifted germanium detector. Moreover, "Mossbauer effect" technique was applied for studying the structure of the three alloys.

Various specimens of the three samples were investigated after being irradiated with neutrons at a flux of 10^{13} n/cm^2.sec. in the A.R.E. Reactor, Atomic Energy Establishment. The two spectrometers were calibrated for energy and intensity before carrying out any measurement. The main results achieved in the present work are as follows :-

1- Applying the scintillation counter, the γ-energy groups of Co^{60} (1333, 1173 KeV), Fe^{59} (1295, 1098 Kev) in the three samples were detected in addition to a γ- energy group of Ru^{103} (497 Kev) observed in sample "B", and "C" only. The abundance determination of these elements was measured and results were tabulated.

2- Applying the Li-drifted germanium detector, γ - energy groups of Co^{60}, Fe^{59}, and sb^{124} were detected in addition to other γ - energy groups of As^{76} (559 KeV), and Ru^{103} (497 KeV). Moreover, the γ - energy group of Mn^{56} was easily detected in sample "A" only. This detector is considered to be more sensitive than the scintillation counter since the (FWHM) measured for the same group by both detectors are found to be 18.62 KeV and 70.2 KeV respectively.

3- The abundance estimation of the elements existing in the three samples was measured applying both detectors, the resulting quantities of abundance were almost found to be of the same order for the same element.

4- The structural studies of the three samples proved that the "Mossbauer effect" techique is a helpful one in studying the state of iron, and the magnetic properties of the alloys. Moreover, the considerable variation in the spectra obtained for the three samples indicate a difference in their structure as well as in the method of preperation of these alloys.

General Introduction

The great advances in nuclear physics during the past 30 years has produced new techniques with useful applications in almost every branch of science, medicine and industry. Among these techniques, the production of artificial radio-active isotopes together with the possibility of their detection and abundance estimation has proved to be a powerful and sensitive tool in trace analysis of physical; chemical, and biological systems.

The term "trace" is of a wide range relative to the main constituents of the samples. However, it is considered to have an upper limit of the amount of a trace element in a sample of the order of 100 parts per million by weight. The lower limit is one part per billion which is called "ultra-trace".

Nowadys, iron alloys are playing a significant role in industry. In general, they have common trace components as Mn, Mo, Ni, Cr, Co, S, V, P, and Si, but vary in abundance and may include as well other trace elements. Such differences in composition often seem to affect the corrosion resistance, metalurgical, and mechanical properties of these alloys.

To analyse an iron ore or alloy, various methods could be applied as chemical trace analysis and solid state trace analysis. The latter is considered to have higher superiority and the techniques applied for such type of analysis are as follows :

i- Neutron activation.

ii- X-ray emission spectrography.

iii- Emission spectrography.

iv- Spark source mass-spectrography.

Since the present research work deals with activation analysis so some critical evaluation for this technique as a method for qualitative and quantitative determination of trace elements is of value to acertain its advantages and limitations relative to the other existing methods.

The interest in the field of activation analysis has considerably increased in reacent years from a purely instrumental and methodological approach. Analysis by radioactivation was first suggested by Hevesy and Levie in 1936 when they activated dysporsium traces (0.1 %) in yttrium samples as well as europium traces (0.1 %) in gadolinium samples, with neutrons from a 300 mc radium berylium source.

The discovery of nuclear reactors as sources of sufficiently high neutron flux has considerably increased the sensitivity of this method. Applying Oakridge reactor, Clark and Overman (1947) determined Mn traces (6×10^{-5}%) in Al metal. Later Overman and Swarthout (1948) estimated P, Mn, and Ni traces in carbonyl iron. Boyed (1949) was also able to determine Mn in Al and Na in carbonates.

Many other researchers had performed similar experiments and their investigations had extended to cover other fields in biology and industry. As examples in biology Tobias and Dunn (1949) injected 10 u gm of Au for 30 days in mouse tissues and after neutron irradiation in Hanford reactor, they were able to estimate concentrations from 0.005 to 4.4 u gm gold/gm. in wet tissue located in various organs of the mouse under test. Keynes and Lewis (1950) were able to determine concentrations of 0.3 u gm. of Na and 3 u gm of K in nerves by irradiating them in a reactor. The counts were taken at first, through a brass filter of 4.6 mg/cm^2 to obtain gamma radiation of Na^{24}, and then through a filter of 0.46 gm/cm^2 for detecting K^{42} beta radiation.

In industry, Winteringham (1950) found a limit of detection of 0.01 u gm Br in tobacco leaves after irradiation with neutrons at a flux of 5 x 10^{11} n/cm^2. sec. Atchison and Beamer (1952) analysed a commercial electrolytic magnesium through neutron irradiation of 1.2 gram disks of that sample in Oakeridge Reactor with neutrons at a flux of 5 x 10^{11} n/cm^2.sec. and found the following trace impurities in p.p.m.

As : 0.12 ; Ca : 1.3 ; Cr : 0.1 ; Cu : 8.0;
K : 1.4 ; P : 2.2 ; Sr : 2.10.

Milner and Smales (1954) were able to determine tantalum in a mixture of Nb and Ti obtained from stainless steel by irradiating about 20 mg of sample for 2 hours together with tantalum oxide as standard, in the Harwell reactor. Using a NaI (Ti) scintillation spectrometer the gamma activity of 111 days Ta^{182} (1.11 MeV γ) was determined and the concentrations of Ta from 0.05 to 0.10 % was estimated. James and Richards (1955) were also able to determine arsenic in silicon and estimated the sensitivity to be about 0.0003 p.p.m. for 1.0 gm sample.

Smales and Pate (1952) irradiated 10 ml of sea water with neutrons at a flux of 10^{12} n/cm^2.sec. for 70 hours, several samples showed the existance of As with concentrations ranging from 1.6 to 5.0 p.p.m.

It should be pointed out that neutrons could be also obtained through some nuclear reactions applying accelerators. Atchison and Beamer (1956), for example, used 2 MeV Van de Graff to achieve neutrons at a flux of 2.5×10^6 n/cm^2.sec. applying the reaction Be^9 (d, n) B^{10}. Utilising these neutrons for activation analysis, they were able to determine Cl, Br, and I of the order of micrograms quantities in samples of aqueous solution.

With regard to the application of activation analysis to iron alloys, one has to point out that the first analysis was made by Goldberg (1950), who was able to detect gold and rhenium in two samples of iron meteorities as 2.1 p.p.m. and 1.2 p.p.m. for gold, as well as 1.6 p.p.m. and 0.3 p.p.m. for rhenium. Later Meiser and Schneider (1961) irradiated samples of steel and they were able to detect Mn, Cu, Cr, and Mo without chemical separation. Ross (1963) irradiated high purity samples of iron with neutrons at a flux of 6×10^{13} n/cm^2.sec. Three trace elements were determined

as impurities which were Mg, Mn, and Rh. He tabulated his results as follows :

Daughter isotope	Mg^{27}	Mn^{56}	Rh^{104}
half life time	9.55(min)	2.58(hr)	42 sec
Detected abundance (ug)	7	0.006	0.02

Wasson (1966) made a determination of Ga and Ge in samples of iron meteorities. He used in irradiating the samples neutrons at a flux of 10^{13} $n/cm^2.sec$. He was able to detect Ge and Ga to be 0.02 p.p.m. and 0.001 p.p.m. respectivily. Artyukhin, Gil'bert, and Pronin (1967) irradiated high purity samples of iron with neutrons at a flux of 1.8×10^{14} $n/cm^2.sec$. They were able to determine Co, Cu, In, Zn, As, Te, and Sn in the order of $10^{-6} - 10^{-5}$ gm.

The above mentioned experiments which were done on various samples of iron, can show that the trace elements which could be detected do not exceed Co, Cu, In, Zn, As, Te, Mn, Mg, Rh, Ge, and Ga. Moreover, their trace elements can vary from one sample to another, and so, it is recommended to carry out further investigations on other iron samples.

For a trace element determination, the minimum detectable amount of that element, which indicate the sensitivity of the applied method of analysis for that element, must be estimated. Sensitivity may be expressed in either absolute or relative units. The absolute estimation refers to the smallest detectable weight of the trace element in micrograms (10^{-6} gm), nanograms (10^{-9}gm), or picrograms (10^{-12} gm). The relative process is the smallest detectable concentration in percentage, which may be p.p.m., micrograms per gram, or micrograms per millitre .

Activation analysis has been reviewed by many authors reporting excellent summaries on the sensitivities of this method of analysis for various elements. For example, Brooksbank and Leddicotte (1955) irradiated various samples with neutrons at a flux of 5 x 10^{11} n/cm^2.sec., and found that the sensitivities of Na^{24}, Br^{82}, Ga^{72}, and Au^{198} were ranging from 0.002 u gm to 0.001 u gm, while the range for K^{42} was from 0.02 u gm to 0.1 u gm.

Later, Steele and Meinke (1961) irradiated Cr, Cu, Fl, Fe, N, O, and Si with neutrons at a flux of 10^8n/cm^2.sec., and they found that the lower limit of detection was ranging