# ANALYSIS OF THE CONCENTRATION OF Fe IN IRON ORE SAMPLE BY IRRADIATION WITH Am-Be NEUTRON SOURCE

Htoo Nwe Nwe Aung<sup>1</sup>, Khin Su Su Han<sup>2</sup>, Kyaw Win<sup>3</sup>, Myo Zaw Htut<sup>4</sup>, Myo Nyunt<sup>5</sup>

### Abstract

The iron concentration in iron ore samples have been observed by means of the instrumental neutron activation analysis technique. In NAA method, the samples were activated by isotope Am-Be ( $\alpha$ , n) sealed neutron source. The strength of it is 550 mCi, with an average neutron energy of around 3 MeV, and a half-life of 432 years. After irradiation, the gamma-ray spectra of the samples were collected and analyzed using NaI (Tl) scintillation spectrometer. By NAA method, the iron content of hematite is 65.91%, limonite is 14.24%, and standard iron is 99.12%. NAA method is excellent to detect the mass concentration of iron in the samples.Iron raw materials, especially in small sizes, can be easily detected by NAA to ascertain their authenticity and iron content.

Keywords: Iron (Fe), Gamma radiation, Iron Ore Mine, NaI(Tl) scintillation detector, Activity Concentration,NAA Method.

#### Introduction

Iron ore was first "discovered" by European settlers when the early land surveyor, Burt, was shown an iron ore by an Indian chieftain near where is now Negaunee, Michigan, in 1844. So far, 22 iron ore occurrences have been observed in Myanmar, excluding numerous other very small occurrences of pyrite. None of these has so far acquired major economic importance. Iron ore consist of 13 limonite-hematite ore occurrences, 3 magnetite ore occurrences, 5 lattice iron ore occurrences, and 1 siderite occurrence. Residual deposits of hematite and limonite in nodules of varying sizes are found in the Southern Shan State and less frequently in the form of lattice iron ores and crusts above basic igneous rocks in the vicinity of basalts in central Myanmar.

Iron, denoted Fe, is unquestionably the world's cheapest and most useful metal. In its natural state, iron is chemically bonded with oxygen, water, carbon dioxide or sulfur in a variety of minerals. The average iron content of the Earth's crust is between 5 % and 6%, and the only more abundant and widely distributed elements are silicon, oxygen and aluminum. Iron ore is a commercial term denoting iron-rich minerals with sufficiently high iron content to be commercially available for exploitation. The most important ore-forming minerals are: hematite, Fe<sub>2</sub>O<sub>3</sub> (69.94% iron); bloodstone, red iron ore. It occurs in large quantities throughout the world. Goethite, FeO (OH) or FeO (OH)-nH<sub>2</sub>O (62.9% iron), limonite, Fe<sub>2</sub>O<sub>3</sub>.H<sub>2</sub>O (at most 62% iron); a collective term for more or less impure goethite and a mixture of hydrated iron oxides, e.g., brown iron ore. Siderite, FeCO<sub>3</sub> (48.2% iron) spatic iron ore, Pyrite, FeS<sub>2</sub> (46.55% iron) the most commonly occurring sulphide mineral in the Earth's crust. Used in the production of sulfuric acid and gives iron-bearing pyrite residue.<sup>1</sup>

Iron ores are rocks and minerals from which metallic iron can be economically extracted. The ores are usually rich in iron oxides and vary in colour from dark grey, bright yellow, deep purple, to rusty red. The iron itself is usually found in the form of magnetite (Fe<sub>3</sub>O<sub>4</sub>), hematite (Fe<sub>2</sub>O<sub>3</sub>), goethite, limonite or siderite as shown in Figure (1).

<sup>&</sup>lt;sup>1</sup> Department of Physics, Defence Services Academy, Pyin Oo Lwin

<sup>&</sup>lt;sup>2</sup> Department of Physics, University of Computer Studies, Mandalay

<sup>&</sup>lt;sup>3</sup> Department of Physics, Defence Services Academy, Pyin Oo Lwin

<sup>&</sup>lt;sup>4</sup> Department of Nuclear Physics, Defence Services Academy, Pyin Oo Lwin

<sup>&</sup>lt;sup>5</sup> Department of Nuclear Physics, Defence Services Academy, Pyin Oo Lwin





# **Experimental Procedure**

### Sample Collection (Sampling Site) and Preparation

The iron ore mine samples were collected from Pinpet iron ore mine. Pinpet Iron Ore Mine is one of the largest excavated iron ore mines in Myanmar. It is situated in Namsee Village tract, Taunggyi Township in the Southern Shan States. It lies at 97°06′30″ longitude and 20°44′30″ latitude. It is 4.5 miles from Taunggyi Hopone motor highway and 7.5 miles from Hopone Township.

Each sample was weighed carefully by digital balance. A 55g of iron ore granules from encapsulated in a polyethylene container was used in this work. This container was chosen due to its low background. Then all samples were put in plastic bags very close to the neutron source for one-week irradiation time.

### Selection of the best Nuclear Reaction for NAA with Isotopic Am-Be (a, n) Neutron source

Iron exists in nature in the four isotope forms of mass 54, 56, 57 and 58. The most abundant isotope of iron is mass 56, and it is the most useful for the activation analysis with 14 MeV and 3 MeV neutrons. The best nuclear reaction for the identification of the isotope of interest should be chosen. Sometimes there is more than one reaction available for the same isotope. The possible induced nuclear reactions in iron <sup>56</sup>Fe together with the reaction characteristics are shown in Table (1).

Reaction	Gamma- Energy (keV)	Half-life (h)	Gamma Emission Probability (%)	Threshold Energy (MeV)	Q- Value (MeV)
<sup>54</sup> Fe (n, 2n) <sup>53</sup> Fe	377.88	0.142	42	13.878	-13.618
$^{54}$ Fe (n, p) $^{54}$ Mn	834.85	7495.2	99.96	-	+0.49
	846.77		98.9		
<sup>56</sup> Fe (n, p) <sup>56</sup> Mn	1810.77	2.576	27.2	2.452	-2.408
	2113.12		14.3		
	14.413		10.56		
<sup>57</sup> Fe (n, p) <sup>57</sup> Mn	122.06	0.024	13.9	1.434	-1.409
	692.03		5.50		
<sup>58</sup> Fe (n γ) <sup>59</sup> Fe	1099.25	1068.07	56.5	_	+6 589
	1291.59	1000.07	43.2		

**Table 1. Neutron Activation Reactions with Fe** 

 $^{56}$ Fe (n,p)  $^{56}$ Mn is the best reaction because it has suitable energy and half-life. Moreover, the absorption reaction cross-section and target isotope abundance of the  $^{56}$ Fe (n,p)  $^{56}$ Mn are relatively high Gamma emission probability (98.9%) from first peak energy (846.77keV) of this reaction is very high. It is also the endothermic reaction (Q-Value is negative) and threshold energy is lower than other reactions. Therefore,  $^{56}$ Fe (n,p)  $^{56}$ Mn reaction is selected from five reactions. The decay scheme is shown in Figure (2). The best choice is this nuclear reaction for iron sample. ( $^{56}$ Fe about 98.9%) This reaction is confirmed by calculating the half-life of this reaction.



Figure 2. Decay Scheme of <sup>56</sup>Mn Radionuclide

# **Gamma Activity Measurement of Irradiated Samples**

Americium also alloys with Beryllium to form Am-Be which produces neutrons via the  ${}^{9}$ Be ( $\alpha$ , n) ${}^{12}$ C reaction was used.  ${}^{241}$ Am has a half-life of about 432 years. Although this isotopic decays by emitting alpha particles of about 5.4 MeV, these particles are following by gamma rays in the 40 to 60 keV region in the majority of the disintegrations. This gamma-ray emission make Americium appear less satisfactory than Plutonium for the preparation of neutron sources. For the Am-Be source in Physics lab, University of Mandalay, total neutron capture from the neutron source N=1.04×10<sup>6</sup> neutrons/cm<sup>2</sup>s. Figure (3) shows <sup>241</sup> Am-Be neutron source for irradiation facility with 550mCi.

The sample material is irradiated by emitted neutrons from a neutron source. In this process, isotopes are converted into radioactive isotopes. These isotopes decay, with half-life varying from seconds to years, which emit  $\gamma$ -radiation of specific energy. This  $\gamma$ -radiation is measured with NaI(Tl) scintillation detector.



Figure 3. Am-Be Neutron Source for Irradiation Facility with 550 mCi

# **Experimental Set-up for Gamma Emission Measurement**

After irradiation, the gamma ray spectra of the radioactive iron ore samples were measured and analyzed by NaI(Tl) scintillation detector and Gamma Vision software. The amplifier gain was adjusted until peaks were obtained at the desired channel numbers. The gamma-ray spectra of four standard sources <sup>241</sup>Am (60 keV), <sup>137</sup>Cs (662 keV), <sup>54</sup>Mn (835 keV) and <sup>60</sup>Co (1173 keV, 1332 keV) were measured for 600 secby using NaI(Tl) scintillation detector for the energy calibration. By plotting the counting efficiencies at various energies for source-detector suitable distance, efficiency calibration curve was obtained. The experimental set-up is shown in Figure (4).



Figure 4. Experimental Set-up for Gamma Radiation Measurement

First, the background was measured for about one hour. Then the plastic bag filled with ore samples was put on the NaI(Tl) scintillation detector. The ore samples were measured for about one hour. At the end of the counting period, the spectrum that was recorded may be stored in MCA. The spectra stored in MCA were analyzed by the application of Gamma-Vision 32 software. Using the energy information, an unknown and known radioisotope can be identified radionuclides with activity concentrations and a picture of the spectrum and then determination of the gross area and net area of full energy peak are shown.

The activity (A) of the isotopes also depends on irradiation time. The intensity of the measured gamma line is proportional to the activity.

The equation of the activity after irradiation of the sample is

$$A = \phi \sigma f m \frac{N_A}{M} (1 - e^{-\lambda t_i}) e^{-\lambda t_d}$$
(1)

where, A = activity (cps)

- $\phi$  = neutron flux (n/cm<sup>2</sup>s)
- $\Sigma$  = neutron capture cross section (cm<sup>-2</sup>)
- m = the mass of irradiated element (g)
- f = isotope abundance (%)
- $N_A$ = Avogadro number (6.02× 10<sup>23</sup> atoms/g.mole)
- M = atomic mass of target element
- $t_i$  = irradiation time (s)
- $t_d$  = decay time (s)

Therefore, the same parameters for the samples are cancelled out each other, the comparative NAA method is obtained as followed;

$$\frac{\mathbf{A}_{sam}}{\mathbf{A}_{std}} = \frac{\mathbf{m}_{sam} (\mathbf{e}^{-\lambda t_d})_{sam}}{\mathbf{m}_{std} (\mathbf{e}^{-\lambda t_d})_{std}}$$
(2)

where,  $A_{sam} = activity$  of the sample

 $A_{std}$  = activity of the standard

 $m_{sam} = concentration of sample$ 

 $m_{std}$  = concentration of standard

 $t_d = decay time = cooling time + counting time$ 

 $\lambda = decay \ constant = 0.693/T_{1/2}$ 

# **Results And Discussion**

The experimental work took place at the Nuclear Research Laboratory, Department of Physics, Mandalay University. In the Neutron Activation Analysis (NAA) experiment, one pure iron standard and seven iron samples underwent irradiation using an Am-Be ( $\alpha$ , n) isotopic neutron source. Subsequently, the induced gamma rays were detected by a NaI(Tl) (3"x3") scintillation detector and analyzed using MCA, Gamma Vision 32 Software, and Excel Software. The neutron flux from the Am-Be ( $\alpha$ , n) isotopic neutron source (~3MeV), calculated using copper foil, was determined to be  $1.658 \times 10^6$  n/cm<sup>2</sup>s.

Hematite and limonite served as standards in this research, with other samples compared against them. Parameters such as ROI, FWHM, Peak Channel, Gross Area, and Net Area were derived from measured peak data. The iron concentration of the samples was calculated using the relative NAA method. Irradiation time, counting time, and the results are presented in Table (2) and Table (3). Measurement results for all samples through the NAA method are detailed in Table (4). A comparison of iron contents via NAA methods is outlined in Table (4) and Figure (5). Figure (6) displays a comparison with background and an iron standard sample spectrum, while Figure (7) showcases a comparison of gamma spectra with background and hematite in the Pinpet iron ore sample. Finally, Figure (8) illustrates a comparison of gamma spectra with background and limonite in the Pinpet iron ore sample spectrum.

Sample	Mass (g)	Irradiation Time (h)	Counting Time (s)	Cooling Time (s)	Decay Time (s)
Standard	55	168	3600	60	3660
Hematite	55	168	3600	60	3660
Limonite	55	168	3600	60	3660

**Table 2. Irradiation and Counting Conditions of Research Samples** 

Sample	ROI	FWHM	Gross Area	Net Area	Error (%)
Standard	770-882	47.57	21738	14179	± 383
	773-885	48.18	22440	14716	$\pm 403$
	776-885	47.48	21674	14144	± 381
Hematite	769-873	45.11	16452	9090	± 336
	773-881	45.39	16426	9910	± 347
	776-884	45.30	16408	9622	± 353
Limonite	773-865	23.33	7171	1973	± 276
	778-870	23.40	7201	2202	± 271
	780-872	31.02	7208	2010	$\pm 276$

Table 3. Peak Data and Counting Results of Research Samples

Table 4. The Measurement Results of Fe Concentration in Research Samples by NAA Method

Name of Sample	Mass (g)	NAA Result of Fe concentration (%)
Standard	55	99.12
Hematite	55	65.91
Limonite	55	14.24



**Comparison of Iron Concentration in Research Samples** 

Figure 5. Result of Fe Concentration by NAA Method in Research Samples



Figure 6. Comparison of Gamma Spectra with Background and Standard Sample Spectrum



Figure 7. Comparison of Gamma Spectra with Background and Hematite in Pinpet Iron Ore Sample Spectrum



Figure 8. Comparison of Gamma Spectra with Background and Limonite in Pinpet Iron Ore Sample Spectrum

### Conclusion

By NAA method, the iron content of hematite is 65.91%, limonite is 14.24%, and standard iron is 99.12%. Thus, the iron weight percent was calculated three times to get a better statistic. By using the analyzed data, the pure iron percent of each sample was evaluated by relative NAA method. NAA method is excellent to detect the mass concentration of iron in the samples. The iron raw materials, especially for small sizes can easily be detected by NAA whether they are real or not and the iron contents. Because of non-destructive testing (NDT) and providing results rapidly, NAA has great advantages over other elemental analysis methods. The study delves into the analysis of iron ore concentration using neutron activation facilitated by an Am-Be neutron source. By leveraging the unique properties of neutron interactions, this method offers a powerful means of quantifying iron content in a non-destructive manner. The results not only aid in understanding the composition of the iron ore sample but also highlight the broader significance of neutron activation analysis in scientific and industrial applications.

### Acknowledgements

I would like to extend their appreciation to Dr. Kaythi Tin, Rector of the University of Taunggyi, for granting permission to conduct this research. They also express great gratitude to Lt. Col. Naing Win, Associate Professor in the Department of Physics at the Defence Services Academy in Pyin Oo Lwin, for his invaluable support, insightful guidance, and encouragement throughout the compilation of this paper

#### References

A.M. Hassan and J. Madani, "Neutron Activation Analysis of Saudi Hematite and Phosphate Samples Using the <sup>241</sup>Am-Be Neutron Irradiation Facility,"Al-Azhar University, Egypt (1994).

"Gamma Spectroscopy,"<u>http://en.wikipedia.org/wiki/Gamma</u> Spectroscopy.htlm (2007).

- "Mineral Information Institute of Iron Ore, http://www.mii.org/minerals/photoiron.htlm, 4 December (2008)
- Z. Molnar, "Neutron Activation Analysis," http://www.reak.bme.hu/Education /Wigner-course /Wigner-Manuals/Budapest/NEUTRON-ACTIVATION-ANALYSIS.htm(2002).