Detection of Trace Elements in Metals by LIBS

A MASTER'S THESIS

in

Electrical and Electronics Engineering

Atilim University

by

MOHAMED LOTFI EKRIM

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Detection of Trace Elements in Metals by LIBS

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BY

MOHAMED LOTFI EKRIM

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

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IN

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Approval of the Graduate School of Natural and Applied Sciences, Atılım University.

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Prof. Dr. Ali Kara

Director

I certify that this thesis satisfies all the requirements as a thesis for the degree of Master of Science.

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Assoc. Prof. Dr. Kemal Efe Eseller

Head of Department

This is to certify that we have read the thesis " Detection of Trace Elements in Metals by LIBS" submitted by " MOHAMED EKRIM " and that in our opinion, it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science.

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ABSTRACT

Detection of Trace Elements in Metals by LIBS

MOHAMED LOTFI EKRIM

M.S., Electrical and Electronics Engineering Department

Supervisor: Assoc. Prof.Dr. Kemal Efe Eseller

October 2017, 150 pages

Trace Element is a chemical element presents only in minor amounts in a particular sample or environment.
Chromium, cobalt, copper, iron, manganese, magnesium, zinc, and other elements that occur in very small amounts as constituents of living organisms, are necessary for their growth, development, and health.
In this thesis I did an experiment to detect elements by using laser induced breakdown spectroscopy (LIBS).

Keywords: Trace Element, LIBS

ÖZ

LİBS ile Metallerde İz Elementlerinin Saptanması

MOHAMED LOTFI EKRIM

Yüksek Lisans, Bilgisayar Mühendisliği Bölümü

Doç. Dr. Bölüm Başk. Kemal Efe Eseller

Ekim 2017, 150 sayfa

Iz Elementi, belirli bir numune veya ortamda yalnızca çok küçük miktarlarda bulunan kimyasal bir elementtir.

Krom, kobalt, bakır, demir, manganez, magnezyum, çinko ve canlı organizmaların bileşenleri olarak çok az miktarda bulunan diğer elementler, büyümeleri, gelişmeleri ve sağlığı için gereklidir.

Bu tezde lazerle indüklenen parçalanma spektroskopisi LIBS kullanarak elementleri tespit etmek için bir deney yaptım.

Anahtar Kelimeler: İz Elementi, LIBS

DEDICATION

I would like to dedicate this thesis to my mother, my father, my wife, my sisters, and my children.

ACKNOWLEDGMENTS

I express sincere appreciation to my Supervisor, Asst.Prof.Dr. Kemal Efe Eseller for his guidance and insight throughout the research.

Thanks also go to all my teachers and everyone helped me in my Master's Thesis.

MOHAMED LOTFI EKRIM

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LIST OF ABBREVIATIONS

LIBS	Laser-Induced Breakdown Spectroscopy
AES	Atomic emission spectroscopy
LIPS	Laser-induced plasma spectroscopy
LIF	Laser-induced fluorescence
OLCF-LIBS	One line calibration free-LIBS
SC-LIBS	Self-calibration-LIBS
CF-LIBS	Calibration free LIBS
LASER	Light Amplification by Stimulated Emission of Radiation
ND-YAG	Neodymium- yttrium aluminum garnet
HeNe	Helium-neon
CW	Continuous Wave
ICCD	Intensified charge-coupled device
Si	Silicon
Ca	Calcium
Ti	Titanium
Cr	Chromium
Fe	Iron
Co	Cobalt
Ni	Nickel
Cu	Copper
Zn	Zinc
As	Arsenic
Cd	Cadmium
Hg	Mercury
Pb	Lead
Na	Sodium
Al	Aluminum
K	Potassium.

CHAPTER ONE

INTRODUCTION

1.1 Laser spectroscopy

Laser spectroscopy is a main and essential device for realization and breaking down sub atomic, atomic pieces and energies.
Mechanical improvements in lasers, spectrometers and identifiers had mind boggling constructive outcomes on the spectroscopy.
Laser sources like beat lasers, color lasers, and tunable lasers have expanded phantom determination.
A semi-traditional way to deal with outflow and retention wonders gives an approach to realize laser spectroscopic methods.

1.2 Laser-Induced Breakdown Spectroscopy (LIBS)

LIBS is a kind of atomistic emission spectroscopy which uses a highly energetic laser pulse as the excitation source. The laser is focused to form a plasma that atomizes and excites samples. The principle of LIBS is analyzing any matter in any case of its physical state, whether solid, liquid or gas. As known any element emits light of characteristic frequencies when excited to sufficiently high temperatures, LIBS can detect any element, limited only by the power of the laser as well as the sensitivity and wavelength range of the spectrograph and the detector. If we know the constituents of a material to be analyzed, LIBS also used to evaluate the relative abundance of each constituent element, or to monitor the presence of impurities.
LIBS is a technique that operates by focusing the laser onto a small area at the surface of the sample; when the laser is discharged it ablates a very small amount of material in the range of nano to picto grams.

LIBS is an experimental atomic emission spectroscopy (AES) technique that uses a laser to create a plasma source. Since the plasma is created by focused optical radiation, the technique has many benefits, compared to other conventional AES techniques.[9]

LIBS often referred to its alternative name laser induced plasma spectroscopy (LIPS). This term LIPS has alternative meanings that are outside the field of analytical spectroscopy, so the term LIBS is preferred.

Technically LIBS is very similar to a number of other laser-based analytical techniques, sharing a lot of the same hardware. These techniques are the vibrational spectroscopic technique of Raman spectroscopy, and the fluorescence spectroscopic technique of laser induced fluorescence (LIF). Actually devices are being manufactured which combine these techniques in a single instrument, allowing the atomic, molecular and structural characterization of a sample as well as giving a deeper insight into physical properties.

The LIBS technique is useful for a variety of applications including elemental analysis, detecting airborne biological agents, quantitative analysis of aerosols, etc.[10]

1.3 Basics of LIBS

In LIBS, a pulsed laser with high peak power illuminates a sample. The beam is focused into a small analysis spot. In this spot, material from the sample is ablated, forming a cloud of nanoparticles above a small crater in the sample. Due to the peak energy of the laser beam is quite high, absorption and multi photon ionization results in increasing opacity in the gas and aerosol cloud above the sample, even during the short laser pulse. The plasma forms as laser energy is increasingly absorbed in that cloud. The plasma melts the nanoparticles and excites atomic emission. The emission is dispersed onto a detector and we interpret the spectrum that can simultaneously tell us about the presence of multiple elements.

CHAPTER TWO

LITERATURE SURVEY

There are a few of studies of development LIBS and how to use LIBS to detect elements. I have read these studies and researches which is focused on detection of elements by LIBS.

The previous studies are listed as the following.

The researcher **Randall L.2004 (2)** is: focused on testing some metals by using LIBS, They tested the metals (Mg, Al, Si, Ca, Ti, Cr, Fe, Co, Ni, Cu, Zn, As, Cd, Hg, Pb) for laser-induced breakdown spectroscopy (LIBS) determination of trace metal determination in liquids is demonstrated.

The researcher **Kemal E. Eseller 2009 (7)** is: focused on developing LIBS.

He developed a laser induced break down spectroscopy sensor to determine the concentration of impurities in hydrogen gas, developed a LIBS sensor for the slurry measurements and applied LIBS to the diagnosis of biodiesel flames.

The researcher **M. A. Shemis 2014 (1)** is: focused on detecting Carcinogenic Metals in Gall Bladder Stones by LIBS.

They have studied the effect of the laser pulse energy and the delay time dependence on LIBS signal intensity.

The constituting elements present in the test samples were identified by using the finger prints wavelength of elements of interest using NIST database. The detected carcinogenic metals were Cd, Ni, Cr, Pb and Hg.

They got result as shown in fig 2.1

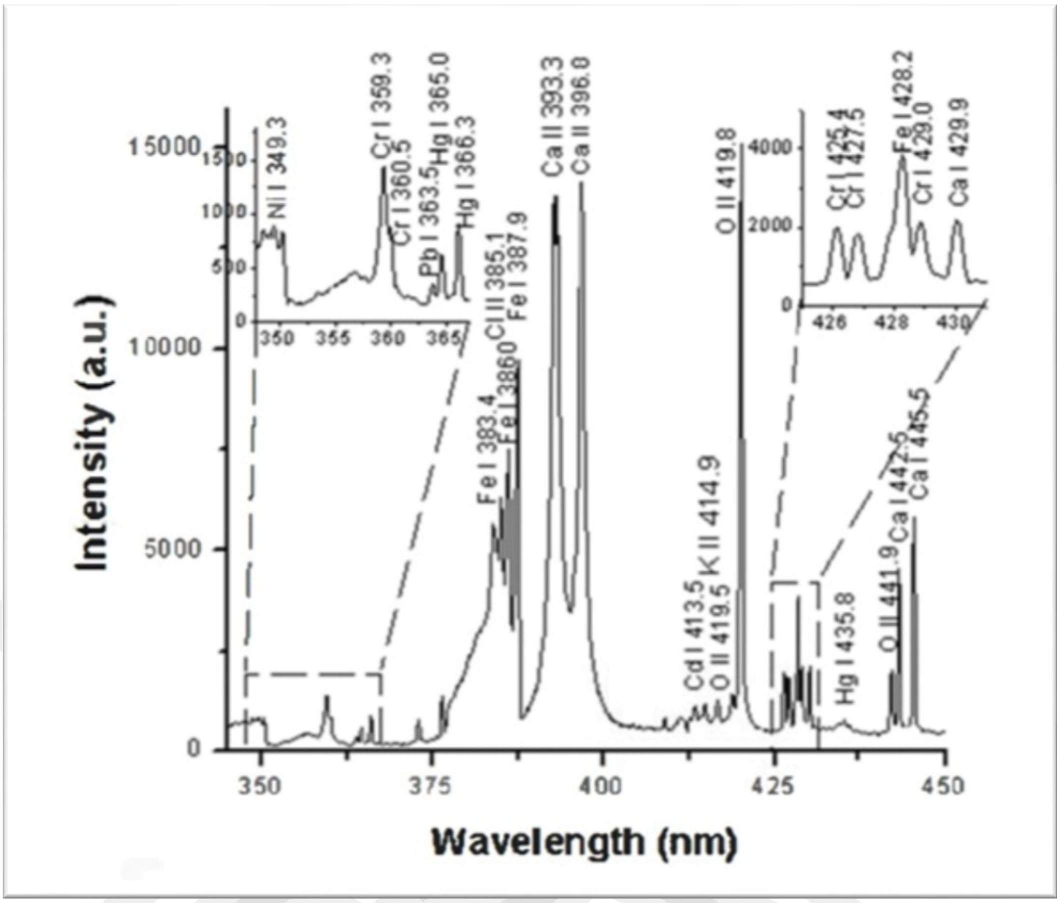


figure 2.1. LIBS spectrum of gallbladder stone in 345-450 nm region recorded at a typical delay time of 200 ns, laser pulse energy of 50 mJ and laser wavelength of 266 nm.

The researcher **Nisar Ahmed 2016 (6)** is: highlighted on a comparative study of Cu–Ni Alloy using LIBS.

They used LIBS for the quantitative analysis of Cu–Ni alloy of known composition (75% Cu, 25% Ni) using the one line calibration free-LIBS (OLCF-LIBS), self-calibration-LIBS (SCLIBS) and calibration free LIBS (CF-LIBS), They got result as shown in figs 2.2, 2.3 and 2.4

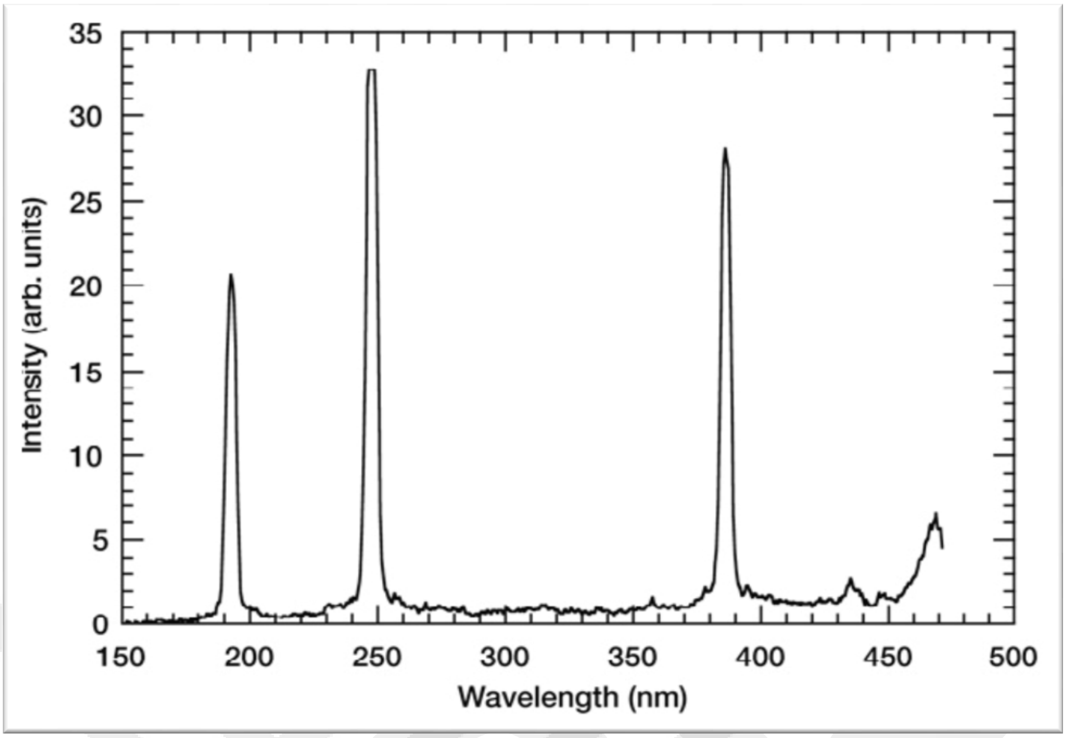


figure 2.2. LIBS spectrum for an un doped disk in 740 torr helium from 150-470 nm. The excitation laser fluence to generate in the spectra was 225 J/cm2. The emission was detected at a time delay of 5µs and integrated over 0.1 µs gate.

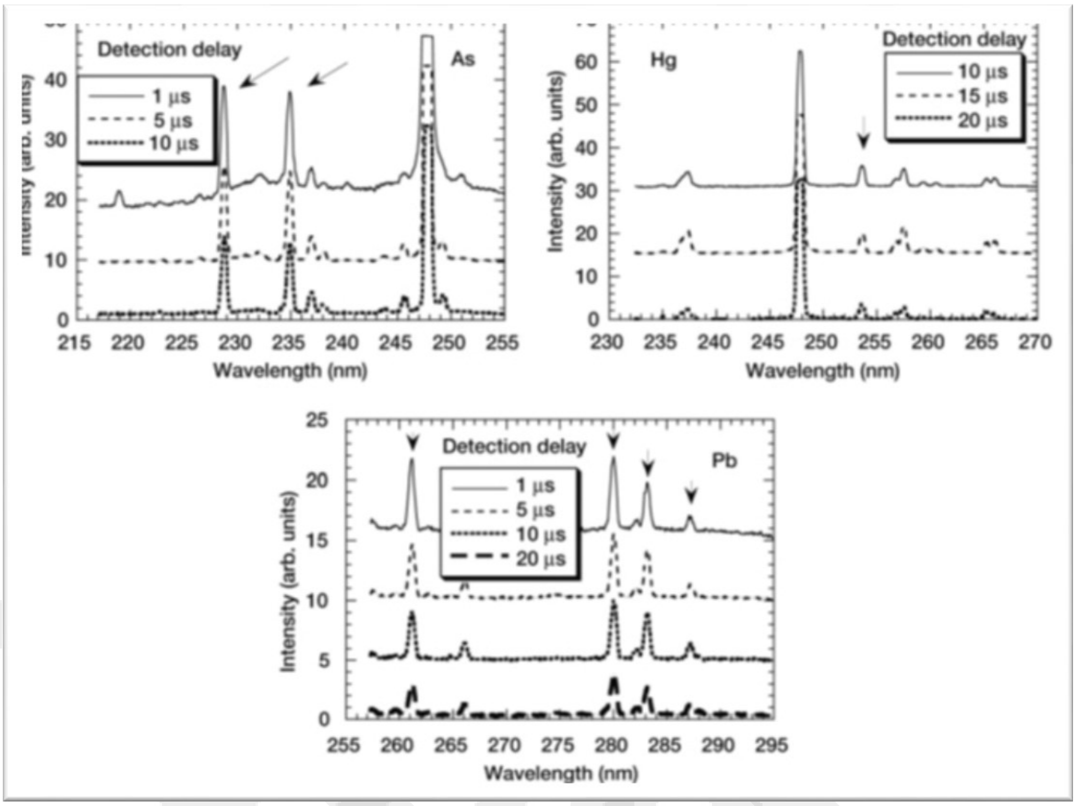


figure 2.3. High resolution LIBS spectra for As, Hg and Pb using a 1 µs gate with various delay times to monitor the emission. Arrows in the spectra indicate emission from the element of interest. The concentration of each metal in the deposited solution was 1000 ppm. For each spectrum, the signal was integrated over 1 µs gate.

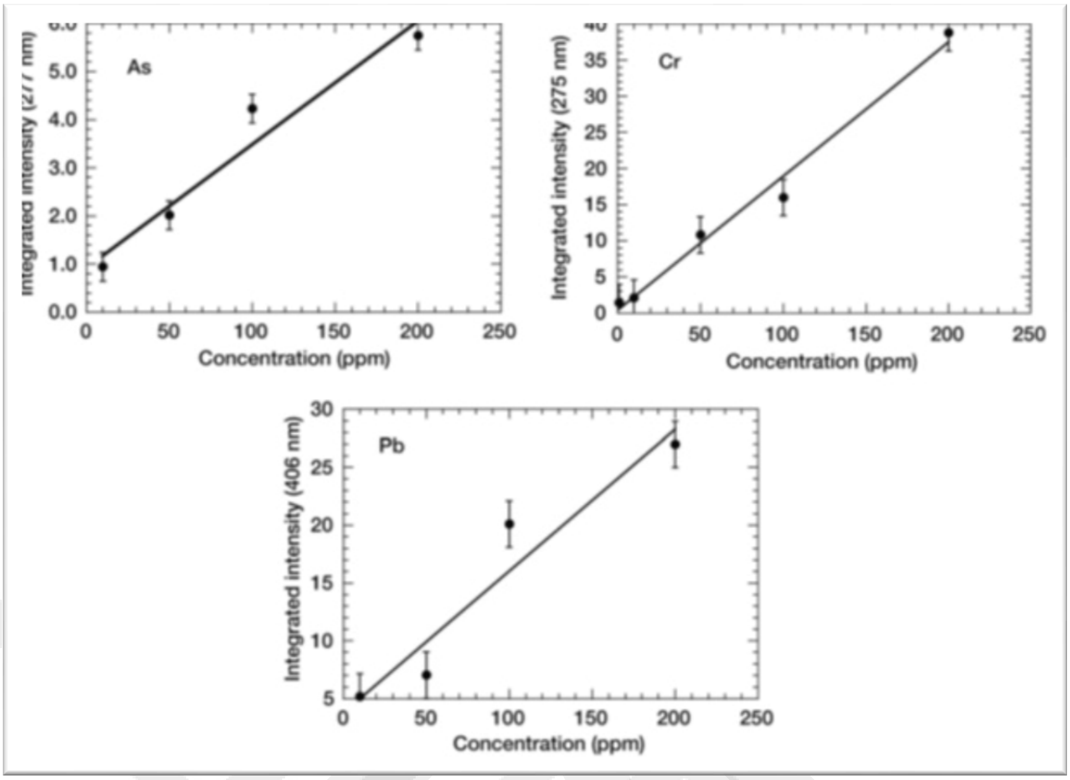


figure 2.4. Analytical working curves based on the integrated elemental emission from LIBS spectral transitions as a function of concentration for the elements As, Cr and Pb.

CHAPTER THREE

THEORETICAL BACKGROUND

3.1 Atomic emission spectroscopy

Atomic emission spectroscopy is used to investigate and distinguish the qualities of radiations discharged from molecules.

Each component has atomic or ionic unearthly line at suitable wavelength and power in view of electron moves between atomic circles.

Assimilation is characterized as a move of an electron from a lower circle to an upper circle because of remaining affected by a source like laser light. This implies ingestion. After that, in the inverse circumstance, an electron tumbling from an upper circle to a lower circle causes an emission of a photon. After the assurance of the ghastly lines with their wavelengths and powers by making deductions about which move has a place with which phantom line, the closeness of related components in a material can be uncovered.

My goal is to test and to detect the elements in any alloy by using LIBS.

3.2 Laser-Induced Breakdown Spectroscopy (LIBS)

LIBS is a kind of atomic emission spectroscopy which utilizes an exceedingly vivacious laser beat as the excitation source (3, 4). The laser is engaged to shape a plasma, that atomizes and energizes tests. On a basic level, LIBS can break down any matter paying little respect to its physical state, be it strong, fluid or gas. Since all components emanate light of trademark frequencies when eager to adequately high temperatures, LIBS can recognize all components, restricted just by the force of the laser and the affectability and wavelength scope of the spectrograph and indicator. On the off chance which the constituents of a material to be dissected are known, LIBS might be utilized to assess the relative plenitude of every constituent component, or to screen the nearness of pollutions. By discovery points of confinement are an element of:

i) The plasma excitation temperature.

ii) The light accumulation window.

iii) The line quality of the saw move.

LIBS makes utilization of optical emission spectrometry and is to this degree, fundamentally the same as circular segment starts discharge spectroscopy.

LIBS works by focusing the laser onto a little zone at the surface of the sample; when the laser is released it removes a little measure of material, in the scope of nano to pico grams, which create a plasma tuft with temperatures in overplus of 100,000 K.

Amid information accumulation, commonly after neighborhood thermodynamic harmony is set up, plasma temperatures run from 5,000–20,000 K.

At the high temperatures amid the early plasma, the removed material separates into energized ionic and atomic species.

Amid this time, the plasma emanates a continuum of radiation that does not contain any valuable data about the species introduce, however inside a little time allotment the plasma grows at supersonic speeds and cools.

The trademark atomic outflow lines of the components can be watched. The deferral between the outflow of continuum radiation and trademark radiation is in the request of 10 μs, this is the reason it is important to transiently door the finder.

In fact LIBS is fundamentally the same as several other laser-based investigative systems, sharing a great part of similar equipment.

These strategies are the vibrational spectroscopic method of Raman spectroscopy, and the fluorescence spectroscopic procedure of laser instigated fluorescence (LIF).

Gadgets are currently being generated which consolidate these strategies in a solitary instrument, permitting the atomic, atomic and auxiliary characterization of a sample and giving a more profound understanding into physical properties too.

3.3 Spectroscopy

Spectrometer is used to measure the properties of light for a variety of applications including environmental or chemical analysis, fluorescence, or Raman. . Spectrometer is optical instrument which can detect spectral lines and measure their wavelength or intensity.

Spectrometers are ideal to determine compositional makeup for detecting weak light signals. Spectrometers also used to test the efficiency of an optical filter in order to determine whether a filter has properly blocked or transmitted specific wavelengths.

Edmund Optics offers a wide variety of Spectrometers, including Spectrometers which have been optimized for Raman or fluorescence spectroscopy. Raman Spectrometer is ideal for Raman applications in the visible to the near infrared. The Fluorescence Spectrometer is ideal for use in low light fluorescence applications where the ability to detect weak signals is crucial for proper measurement.

In addition, fluorescence Spectrometers feature broadband spectral response ranges for detecting wide wavelength bands along with a wide slit width for increased throughput

3.4 Lasers

LASER is constructed from Light Amplification by Stimulated Emission of Radiation. Laser has become very common and popular in everyday life that it is now referred to as laser.[11]

A laser is created when the electrons in atoms in special glasses, crystals, or gases absorb energy from an electrical current or another laser and become excited. The excited electrons move from a lower energy orbit to a higher energy orbit around the nucleus of the atom. When they turn back to their normal or ground state, the electrons emit photons which are particles of light.

Fig 3.1 laser

All these photons are at the same wavelength and are coherent, which mean the crests and troughs of the light waves are all in lockstep.

In contrast, ordinary visible light includes multiple wavelengths and is not coherent. Laser light is different from normal light in other ways as well. Its light contains only one wavelength or one specific color.

The private wavelength of light is determined by the amount of energy released when the excited electron drops to a lower orbit. Laser light is directional. Whereas a laser generates a very narrow beam, a flashlight generates light that is diffuse. Because laser light is coherent, it stays focused for large distances, even to the moon and back.

3.4.1 Types of Lasers

There are a lot of types of lasers available for research, industrial, medical, and commercial uses. Lasers are overwhelmingly described by the kind of lasing medium they use - solid state, gas, excimer, dye, or semiconductor.

1- Solid state lasers, they have lasing material distributed in a solid matrix, like the ruby or ND-YAG (neodymium-yttrium aluminum garnet) lasers. The neodymium-YAG laser emits infrared light at 1.064 micrometers.
The solid state laser, as well called the doped insulator laser to avoid connotation of semiconductor. [13]

2- Gas lasers such as helium and helium-neon, HeNe, are the most common gas lasers have a main output of a visible red light. CO_2 lasers emit energy in the far infrared, at 10.6 micrometers, they are used for cutting hard materials.

3- Excimer lasers, the name excimer is derived from the terms excited and dimers. They use reactive gases such as chlorine and fluorine mixed with inactive gases such as argon, xenon, or krypton. When electrically stimulated, an artificial molecule or dimer is created and when lased, produces light in the ultraviolet range.

4- Dye lasers, they use complex organic dyes such as Rhoda mine 6G in liquid solution or suspension as lasing media. Dye lasers are tunable over a broad range of wavelengths.

5- Semiconductor lasers, also called diode lasers. They are not solid state lasers. These electronic devices are mostly very small and use low power. They may be built into larger arrays, for example, the writing source in some laser printers or compact disk players.

The simplest structure of a semiconductor laser consists of a thin active layer sandwiched between *p*-type and *n*-type cladding layers of another semiconductor with a higher band gap.[12]

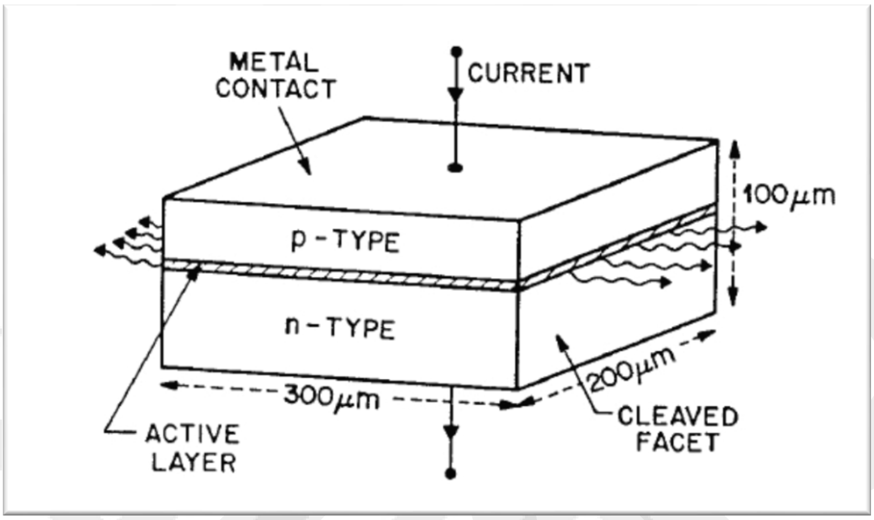


Figure 3.2. A broad-area semiconductor.[12]

6- Continuous Wave (CW) lasers which operate with a stable average beam power. In most higher power systems, one is able to modify the power. In low power gas lasers, such as HeNe, the power level is fixed by design and performance usually degrades with long term use.

7- Single Pulsed or normal mode lasers mostly have pulse durations of a few hundred microseconds to a few milliseconds. This operation mode is sometimes referred to as long pulse or normal mode.

8- Single Pulsed Q-Switched lasers are the result of an intracavity delay (Q-switch cell) that allows the laser media for storing a maximum of potential energy. Then, under optimum gain conditions, emission occurs in single pulses; typically of 10^{-8} second time domain. These pulses have high peak powers often in the range from 10^6 to 10^9 Watts peak.

9- Repetitively Pulsed, also called scanning lasers generally involve the operation of pulsed laser performance operating at a fixed or variable pulse rates which may range from a few pulses per second to as high as 20,000 pulses per second. The direction of a CW laser could be scanned rapidly using optical scanning systems to create the equivalent of a repetitively pulsed output at a given location.

10-Mode Locked lasers, they operate as a result of the resonant modes of the optical cavity which can affect the characteristics of the output beam. When the phases of different frequency modes are synchronized, for example, locked together, the different modes will interfere with one another to generate a beat effect. The result is a laser output which is observed as regularly spaced pulsations. Lasers operating in this mode-locked fashion, usually produce a train of regularly spaced pulses, each having a duration of femto to pico sec. A mode locked laser can deliver extremely high peak powers than the same laser operating in the Q-switched mode. These pulses will have enormous peak powers often in the range from tera Watts peak.

Laser is characterized by the duration of laser emission continuous wave or pulsed laser. A Q-Switched laser is a pulsed laser that contains a shutter like device which does not allow emission of laser light until opened. Energy is built up in a Q-Switched laser and released by opening the device to create a single and intense laser pulse.

3.5 Detectors

A detector is used to measure the illumination of a light source for a number of optical or spectrometry applications.

The detector consists of arrays of photodetectors or photodiodes which transmit electrical current when excited by collision photodetectors from photons.

The detector enables the measurement of important light characteristics that are not conveniently acquired in other ways.

A selection of photodiodes is available with linked amplifier capabilities for easier, more precise measurement capacities.

3.5.1 Photodetectors

photodetectors also called Photosensors, they are sensors of light or other electromagnetic energy [5]. A photo detector has a p–n junction which converts light photons into current. This junction is covered by an illumination window, usually having an anti-reflective coating. The absorbed photons make electron-hole pairs in the depletion region. Photodiodes and photo transistors are types of photo detectors. Similarly solar cells absorb light and turn it into energy.

Fig 3.3 Photodetector.

3.5.2 Types of Photodetector

Photodetectors are classified by their mechanism for detection:

1- Photoemission: in this type, the Photons cause electrons to transition from the conduction band of a material to free electrons in a gas or a vacuum.

2- Photoelectric: in this type, the Photons cause electrons to transition from the valence band to the conduction band of a semiconductor.

3- Photovoltaic: in this type, the Photons cause a voltage to develop across a depletion region of a photovoltaic cell.

4- Thermal: in this type, the Photons cause electrons to transition to mid gap states then decay back to lower bands, inducing phonon generation and thus heat.

5- Polarization: in this type, the Photons induce changes in polarization states of suitable materials, which may lead to change in index of refraction or other polarization effects.

6- Photochemical: in this type, the Photons induce a chemical change in a material.

7- Weak interaction effects: in this type, the photons induce secondary effects such as in photon drag detectors or gas pressure changes in Golay cells.

Photo detectors may be used in different configurations.

Single sensors may detect overall light levels.

A 1-D array of photo detectors, as in a spectrophotometer or a Line scanner, may be used to measure the distribution of light along a line.

A 2-D array of photo detectors may be used as an image sensor to form images from the pattern of light before it.

CHAPTER FOUR

EXPERIMENTAL SECTION

4.1. INTRODUCTION

Laser Induced Breakdown Spectroscopy is a direct elemental analysis method which provides rapid results. A tightly focused pulsed laser ablates material at the laser's focal point, and forms an analytical plasma which is the source for elemental analysis.

LIBS is a laser diagnostics, where a laser beam focused onto a material produces transient high density plasma as the laser intensity exceeds the breakdown threshold of the material.

First used of LIBS was for the determination of elemental composition of materials in the form of gases, liquids and solids during 1960's.

Research on LIBS has been continued to grow and reached a peak around 1980 and field portable gadgets capable of in situ and real time analysis of samples have been developed in recent years with the availability of reliable, smaller and less costly laser systems along with sensitive optical detectors, like the intensified charge coupled device (ICCD).[8]

In all LIBS experiments the delay time, time interval between the arrival of the laser pulse on the sample and activation of the detector, is to be optimized so as to minimize the background noise because of continuum emission and maximize the emission intensity of the spectral line of interest. In general the background noise is negligible after the delay time of about 0.1µs or less while the intensity of most of the neutral atom spectral lines is maximum in 0.1 to 2.0 µs range have observed that the RSTD is almost constant in 0.1 to 2 µs range while it increases from 5.5% to 14% when the delay time is changed from 2 µs to 4 µs. The increase in RSTD at higher delay times is as result of decrease in signal to noise ratio resulting from the decrease in emission intensity with increase in delay time.[14],[15] Different types of LIBS experimental set up have been used which differ fundamentally in the form of collection optics for the radiation emitted by the plasma plume. In one of the arrangements the emission from plasma is collected in the direction perpendicular to the direction of the incident laser. Additionally the difficulties of alignment and reduced sensitivity the collected emission exhibits spatial dependence leading to loss of spectral information about emission from the whole plasma plume, as shown in fig (4.1).

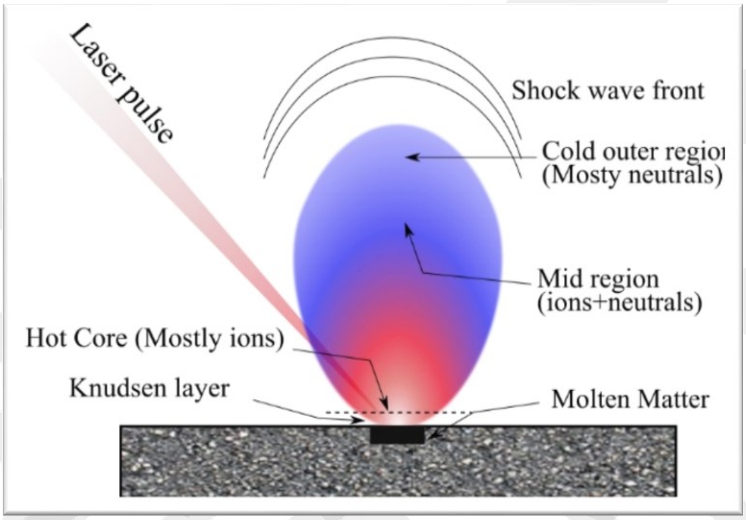


fig (4.1). plasma plume

These shortcomings are removed in another arrangement where focusing lens itself acts as the collecting lens for the plasma emission.

Plasma temperature can be determined from the measurement of ratios of the intensities. There are two cases for it; first case is ion to neutral lines. The second case is neutral to neutral lines, usually for the same element.

Plasma temperature and electron number density can be estimated from the continuum emission and peak width of atomic and ionic emission lines.

4.2 Experimental section

4.2.1 Sample preparation

The principle of my work is tracing elements in metals, so we bring an alloy and started work to analyse and detect the elements in this alloy. This experiment was done at Hacettepe University's laboratory.

4.2.2 LIBS setup

In this experiment we used ND YAG laser 1046 nm , spectrometer with 5 channels, broad band mirror 1046 nm , cooling laser head power supply, delay generator, lenses 10 cm, and computer.

First we bring the alloy and began our experiment by using LIBS. Data analysis software was used to analyse the signals and collect the data. Then we used excel to do calculations and compared the results with NIST Atomic Spectra Database to make sure what are the elements.

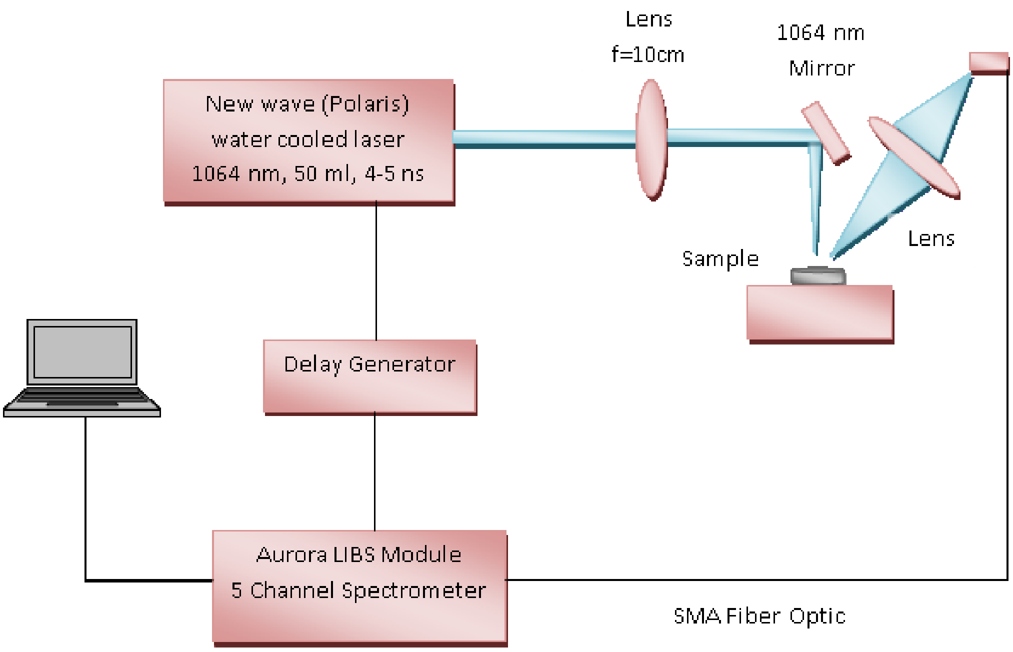


fig (4.2). LIBS experimental setup

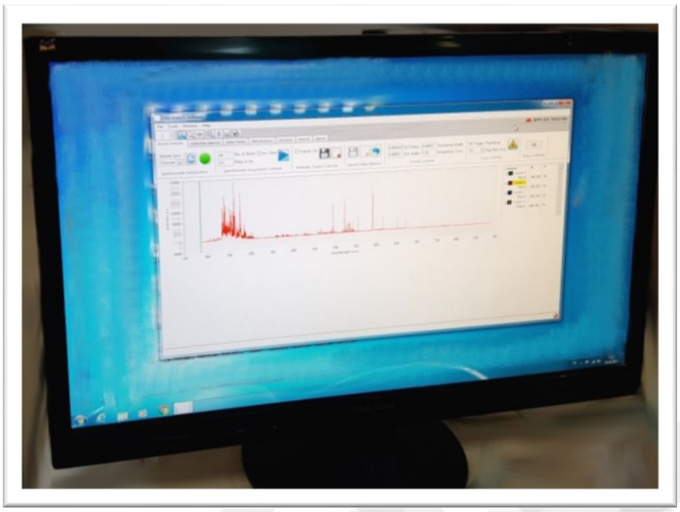
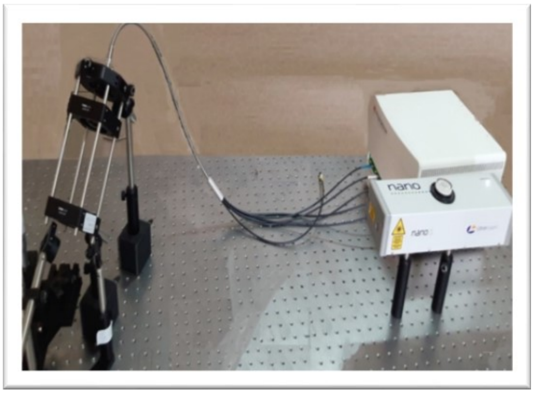
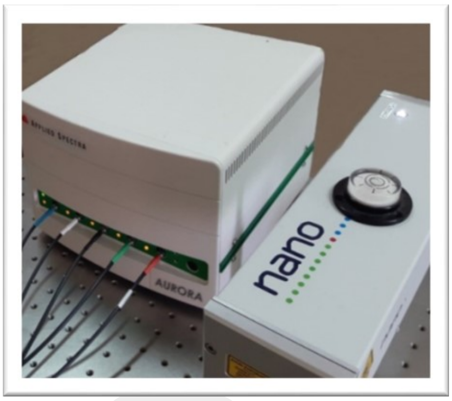


fig (4.3). Data analysis software

fig (4.4). the sample (alloy)





a b

fig (4.5.a,b).spectrometer and ND YAG laser

fig (4.6). cooling laser head power supply

fig (4.7). ND YAG laser.

We took the values from data analysis software and used excel to analyse the data, then we used ''X Y scatter'' to get LIBS spectra as shown in the following fig.

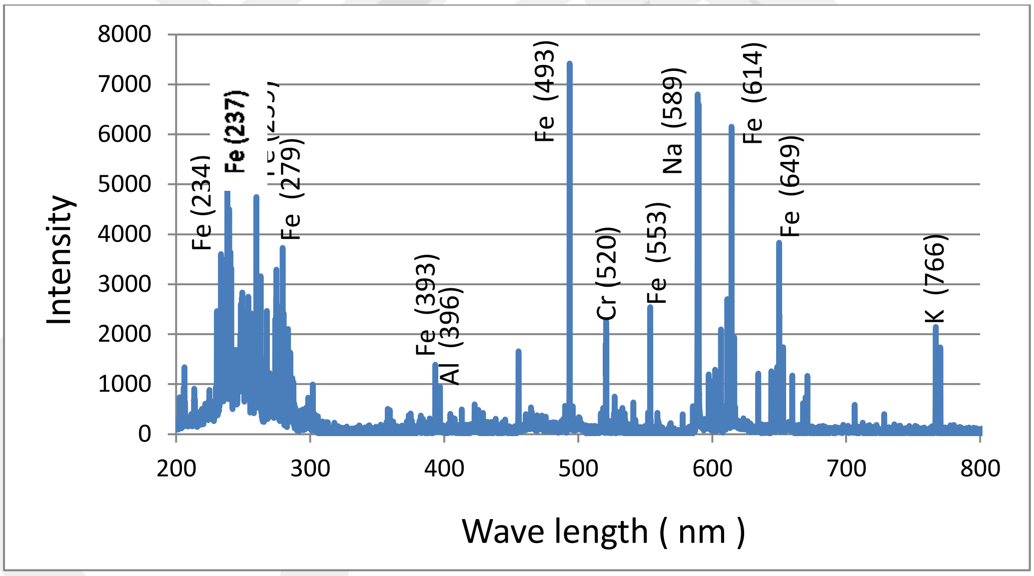


fig (4.8). LIBS spectra for the sample (alloy)

In this experiment, we used three delay times 0.1, 0.3 and 0.5 µs, also 20, 30 and 40 mJ of power. The alloy was moved in to three locations to get the best data of the alloy, the frequency was 10 hz and 50 shots every time.

We began with 0,1 µs time delay, and power of 20 mJ, with frequency 10 hz, and 50 shots, then we have moved the alloy in three locations, and we got the following results as shown in the following figures.

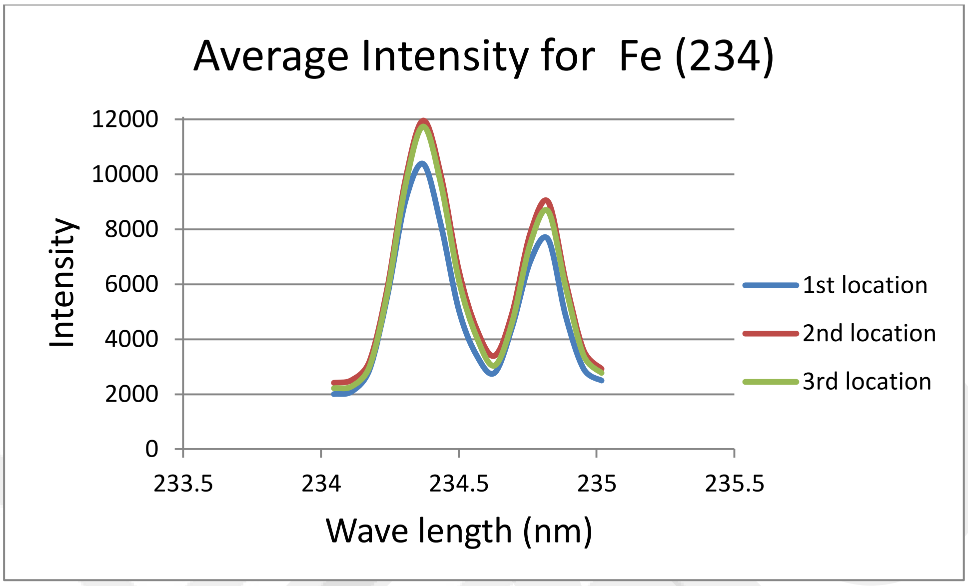


fig (4.9). Average Intensity for Fe (234)

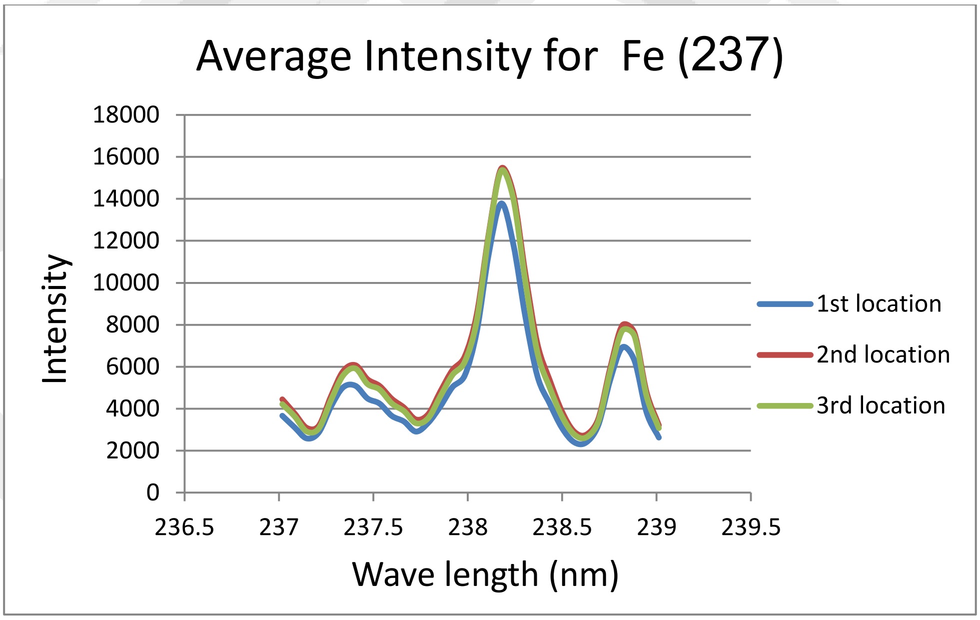


fig (4.10). Average Intensity for Fe (237)

To get the average intensities we used the function ''average'' of the values for each spectrum for 50 shots for first location, next we took the summation of them by using the function ''sum''. After that we used ''X Y scatter'', then we repeated that for the second and the third locations after that we added them in the same curve.

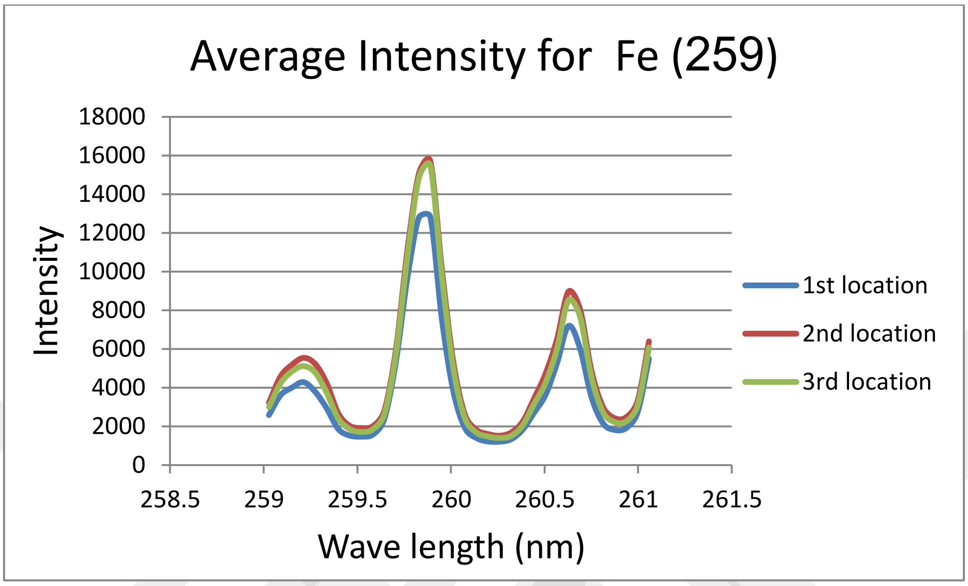


fig (4.11). Average Intensity for Fe (259)

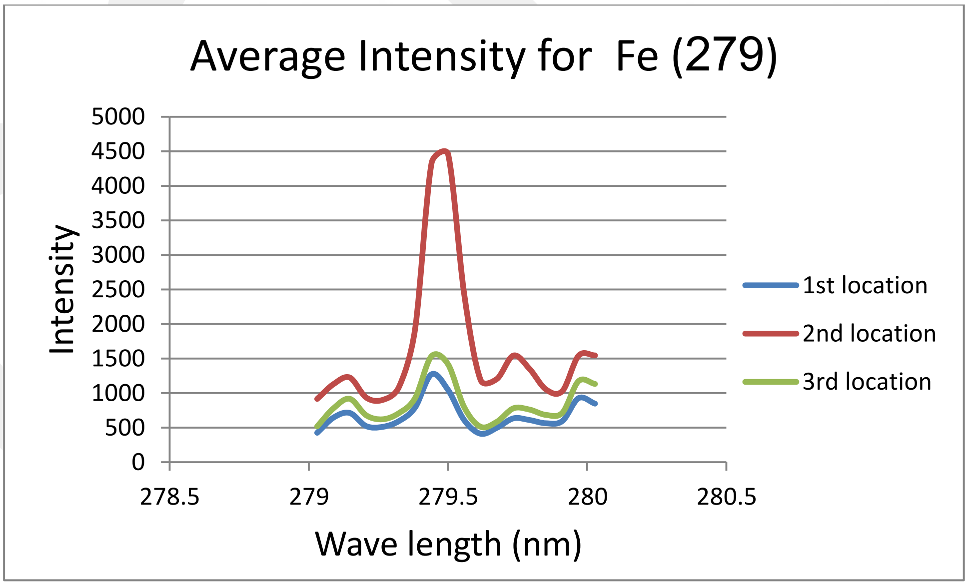


fig (4.12). Average Intensity for Fe (279)

The element as shown in fig (4.12) is concentrated in second location.

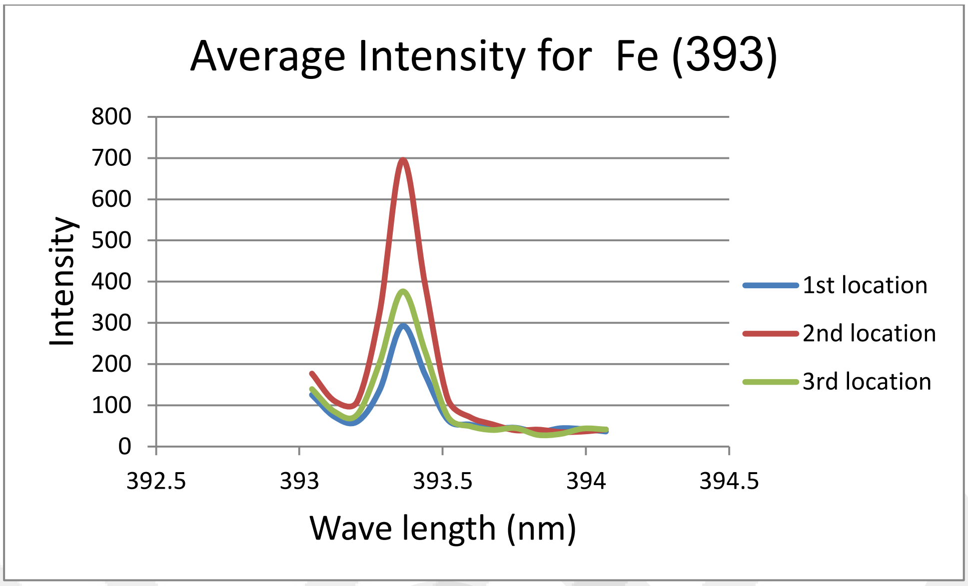


fig (4.13). Average Intensity for Fe (393)

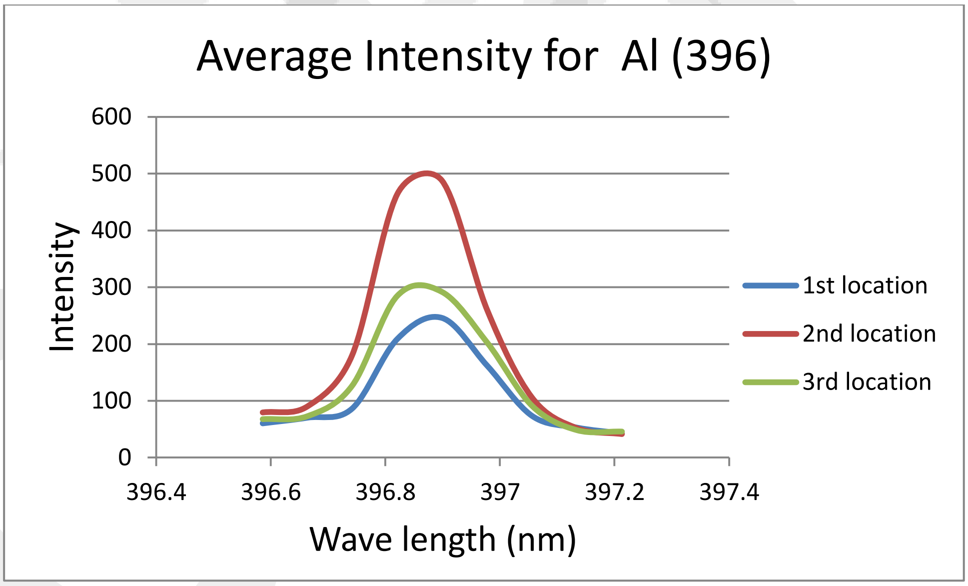


fig (4.14). Average Intensity for Al (396)

The element as shown in fig (4.14) is also concentrated in second location.

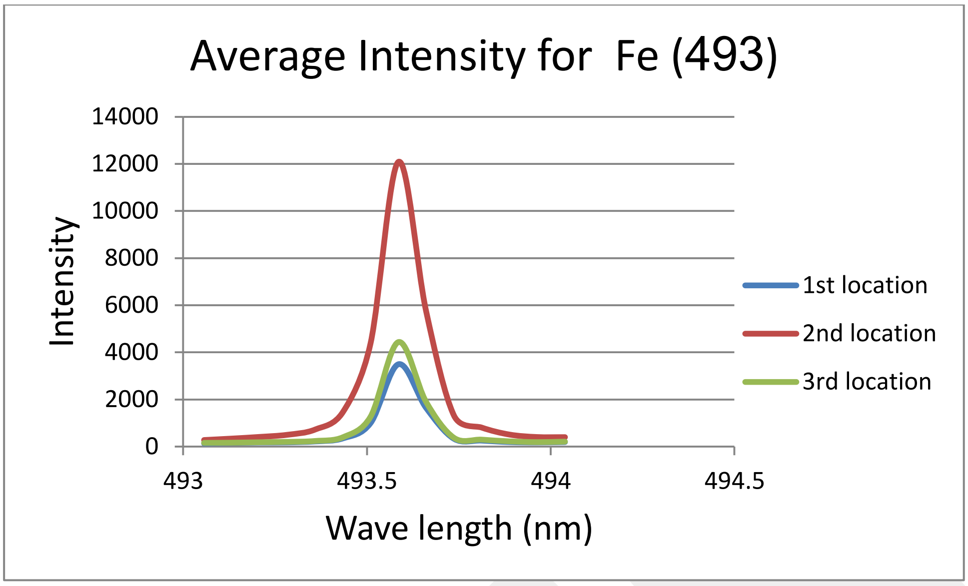


fig (4.15). Average Intensity for Fe (493)

The element as shown in fig (4.15) is concentrated in second location too.

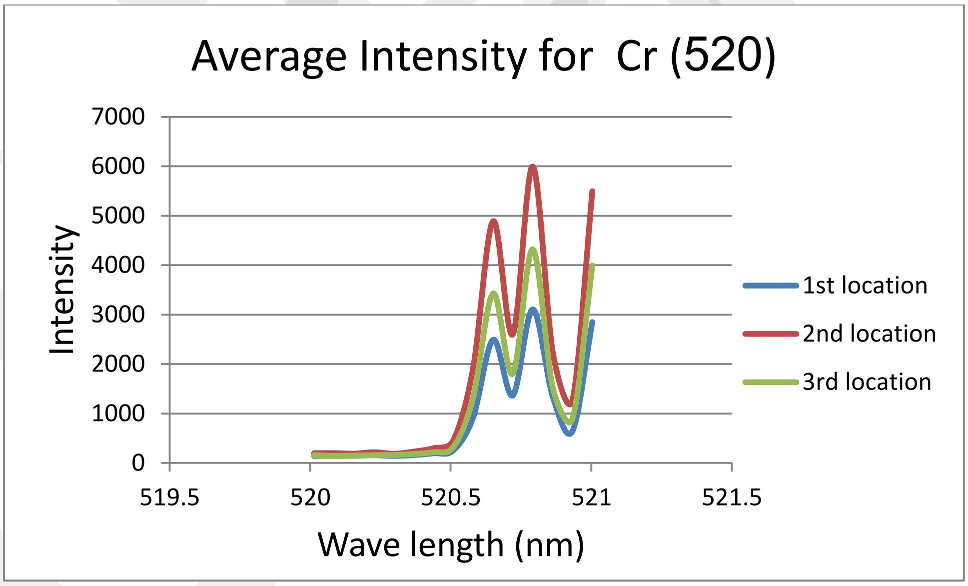


fig (4.16). Average Intensity for Cr (520)

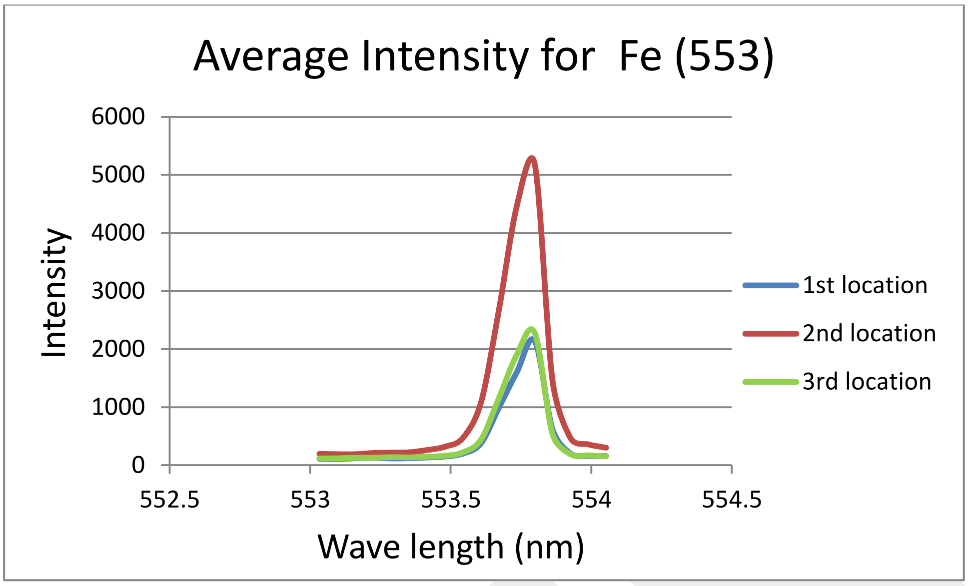


fig (4.17). Average Intensity for Fe (553)

The element as shown in fig (4.17) is also concentrated in second location, first and third locations are almost identical.

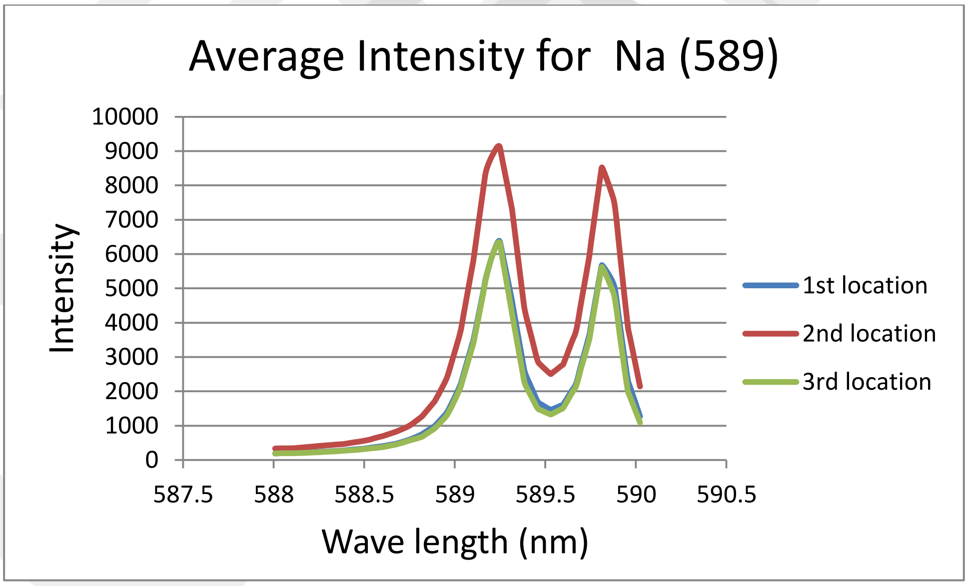


fig (4.18). Average Intensity for Na (589)

First and third locations are almost identical for this element.

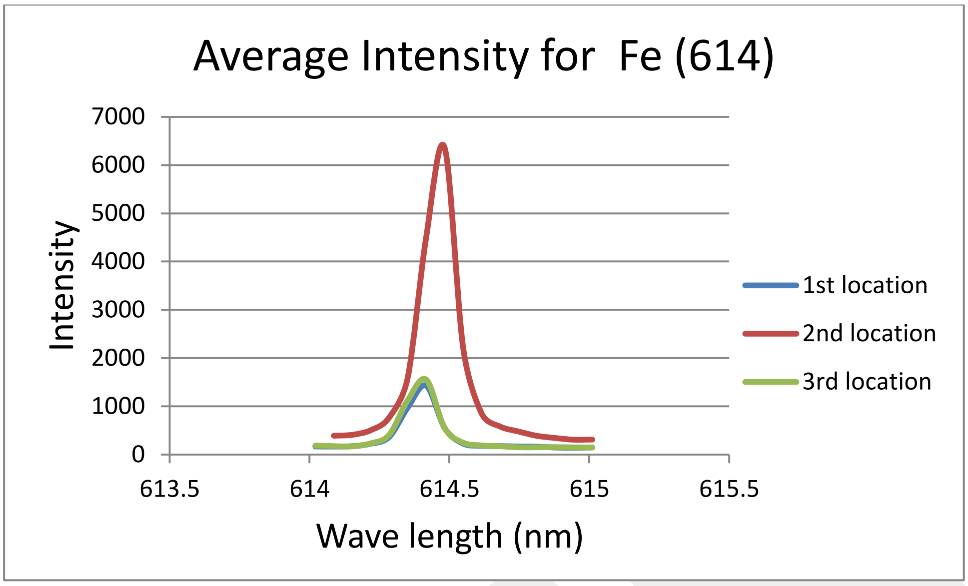


fig (4.19). Average Intensity for Fe (614)

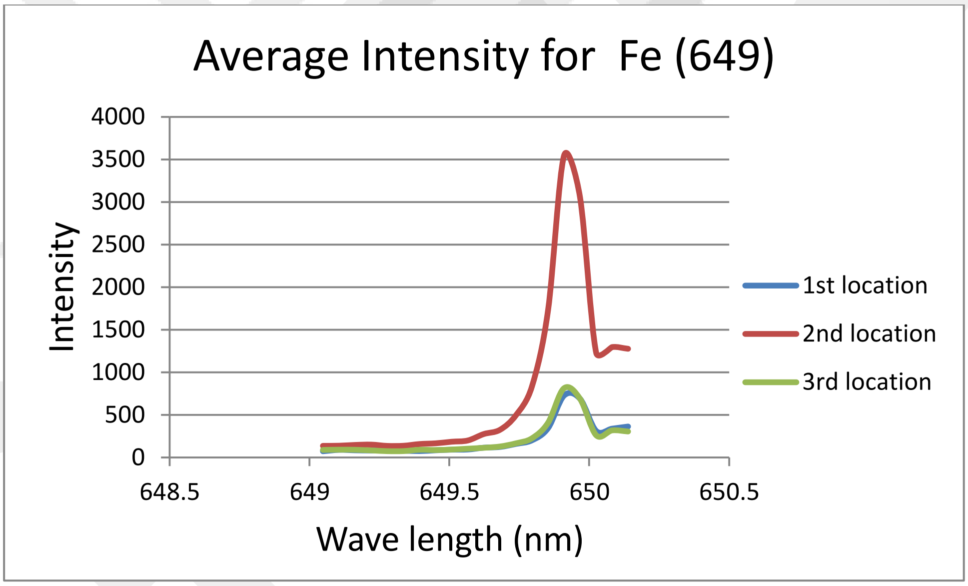


fig (4.20). Average Intensity for Fe (649)

The elements as shown in figs (4.19), (4.20) are also concentrated in second location, first and third locations are almost identical too.

We noticed that there is a shift for the spectrums because the spectrometer is not calibrated well.

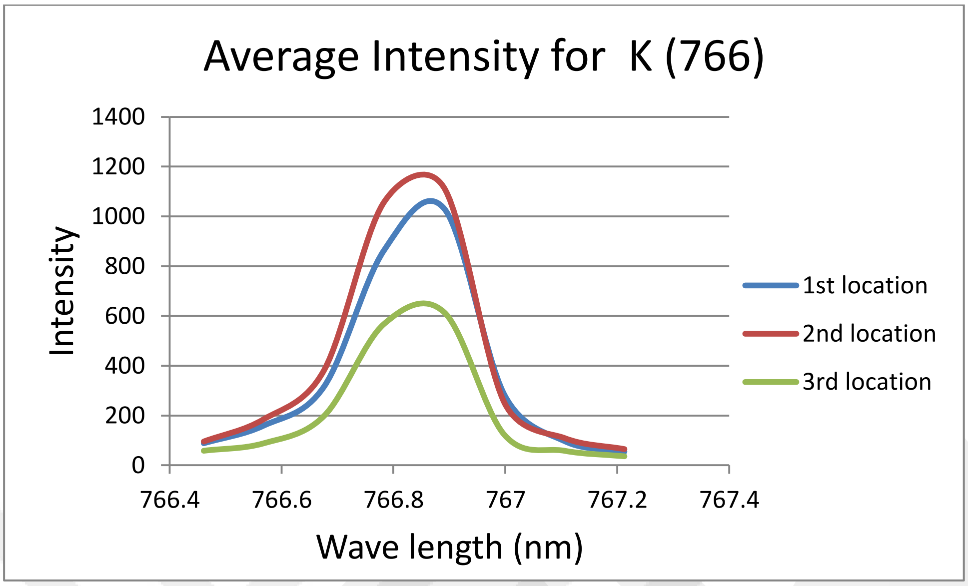


fig (4.21). Average Intensity for K (766)

For area under the curve we took the summation of averages for first location and the same for second and third locations, then we used ‘‘line chart’’. After that we used the function ‘‘STEDV’’ for the summation of averages for three locations to get the standard error.

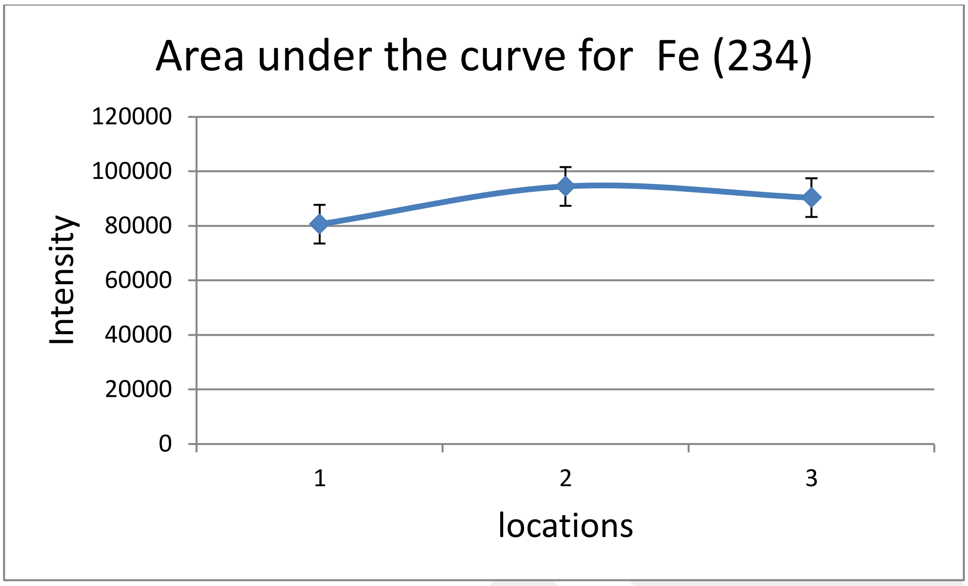


fig (4.22). Area under the curve for Fe (234)

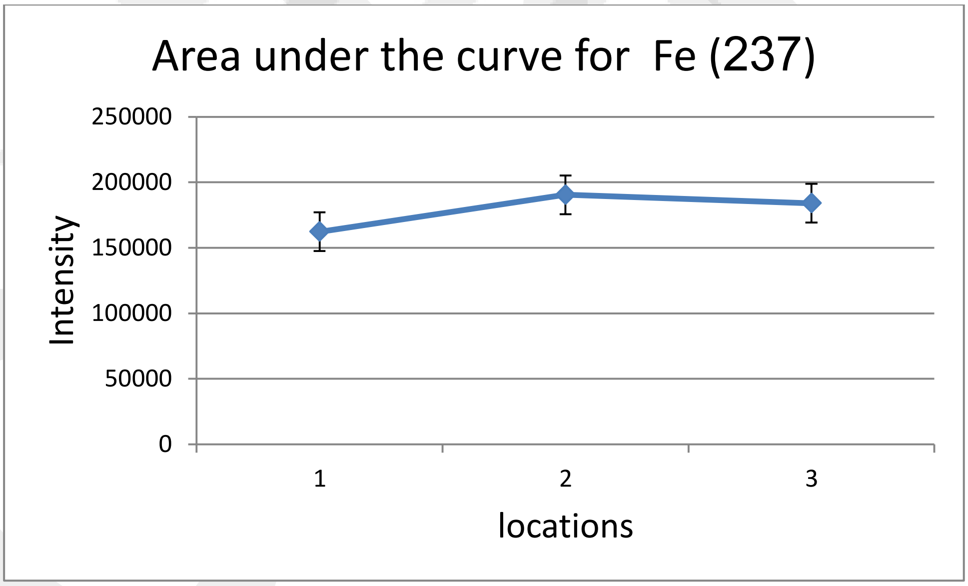


fig (4.23). Area under the curve for Fe (237)

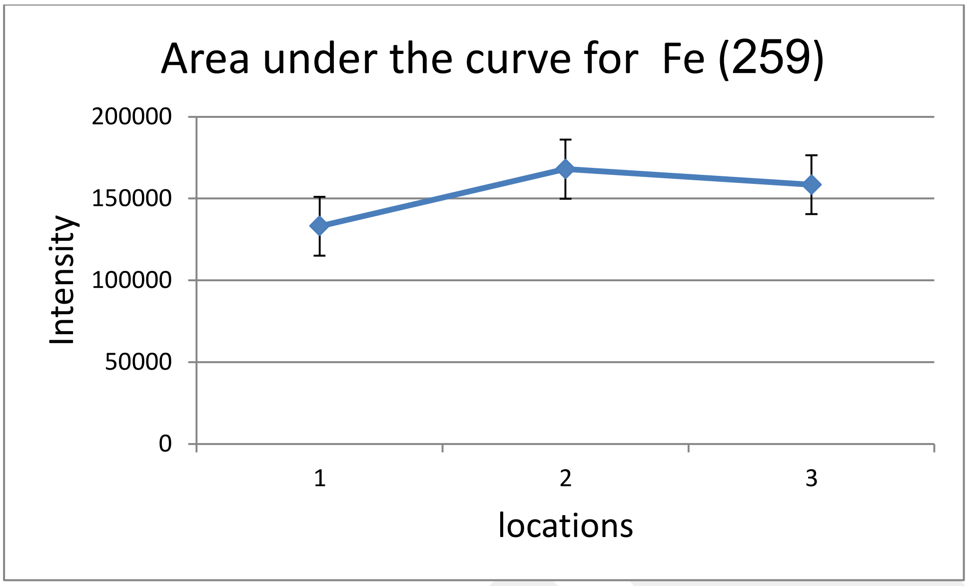


fig (4.24). Area under the curve for Fe (259)

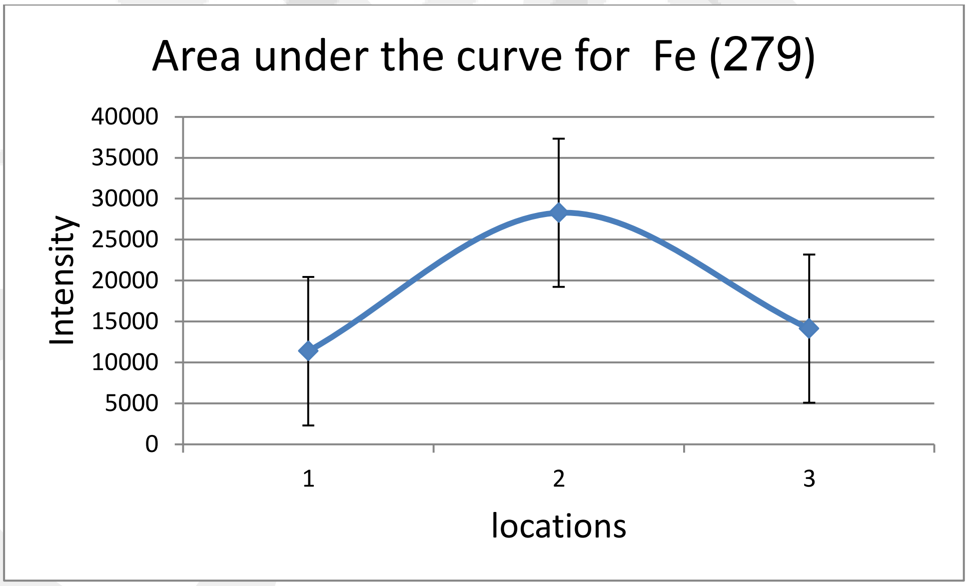


fig (4.25). Area under the curve for Fe (279)

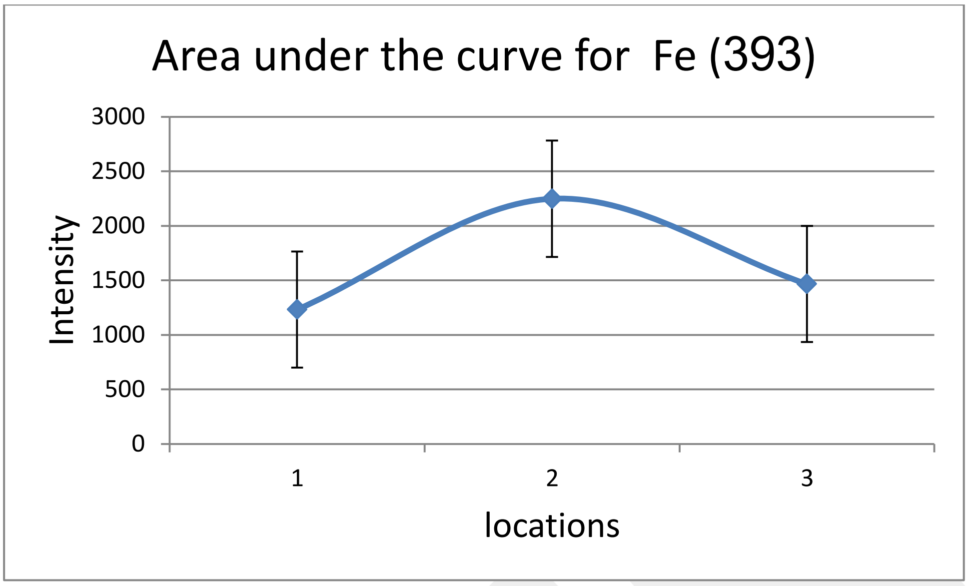


fig (4.26). Area under the curve for Fe (393)

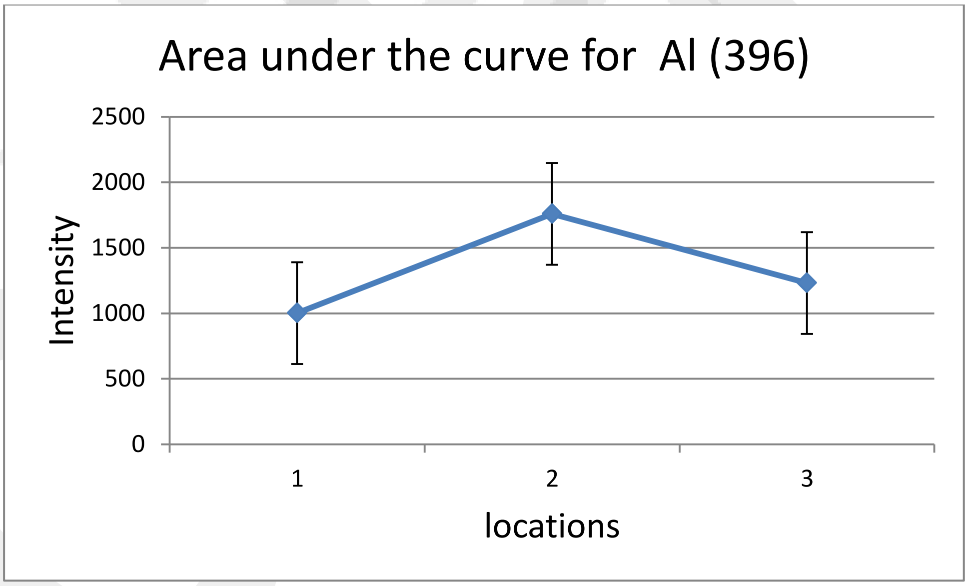


fig (4.27). Area under the curve for Al (396)

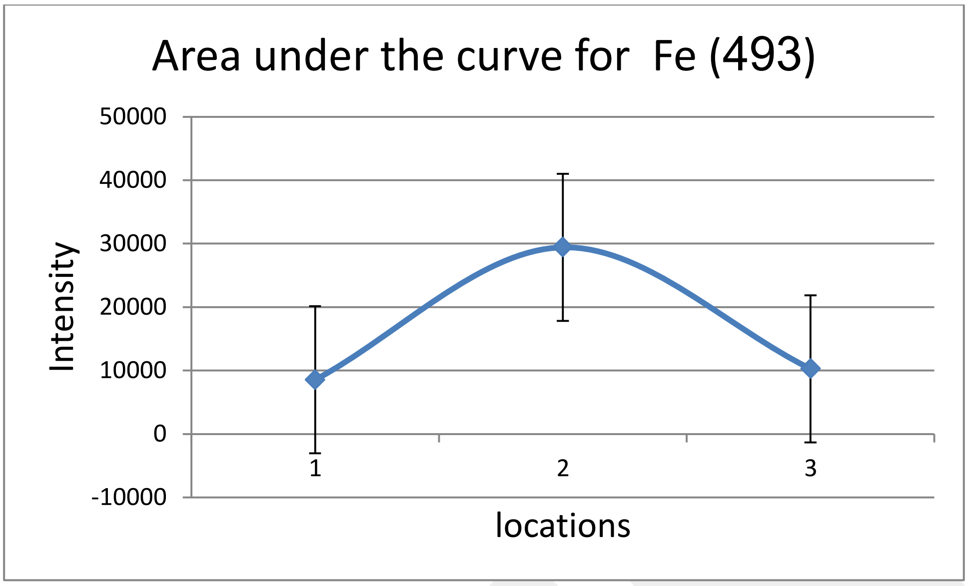


fig (4.28). Area under the curve for Fe (493)

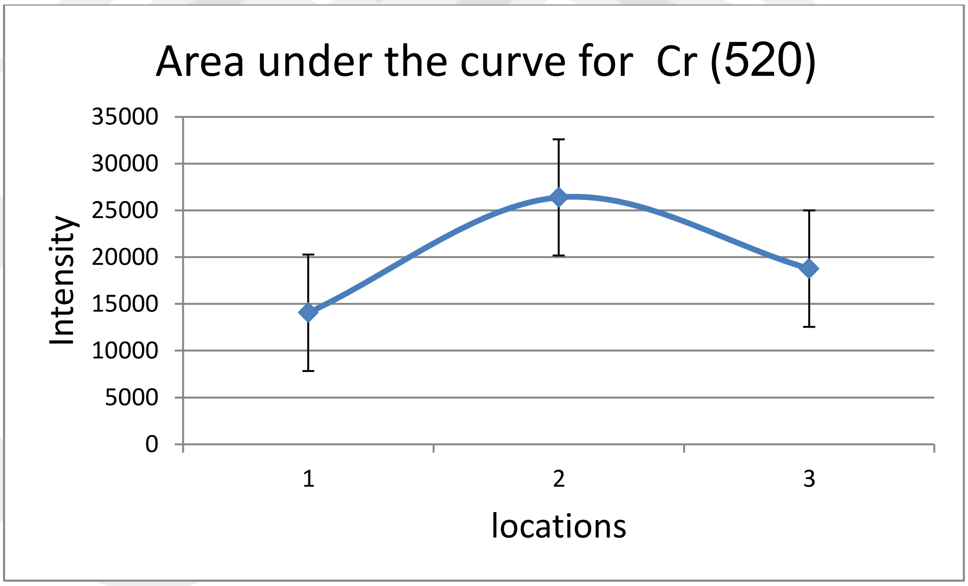


fig (4.29). Area under the curve for Cr (520)

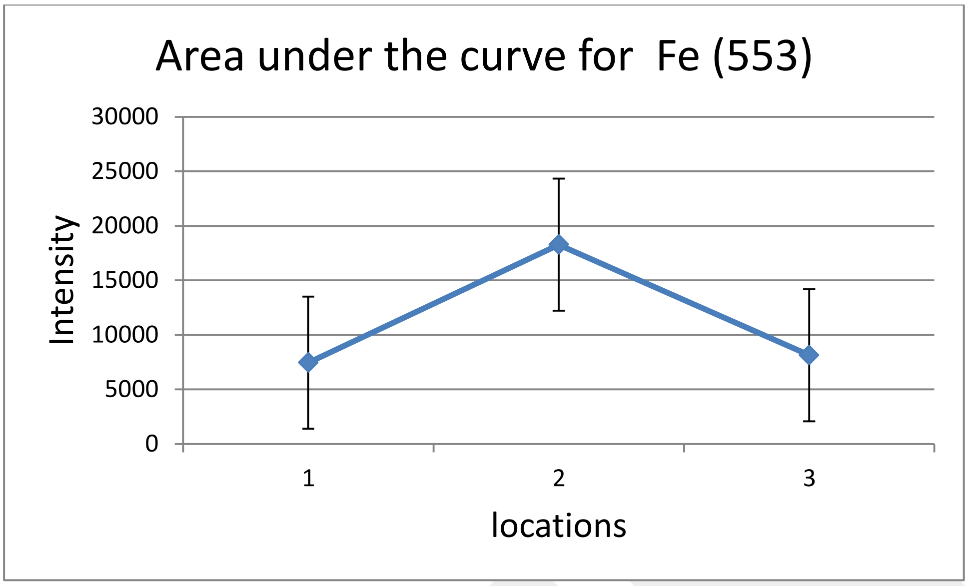


fig (4.30). Area under the curve for Fe (553)

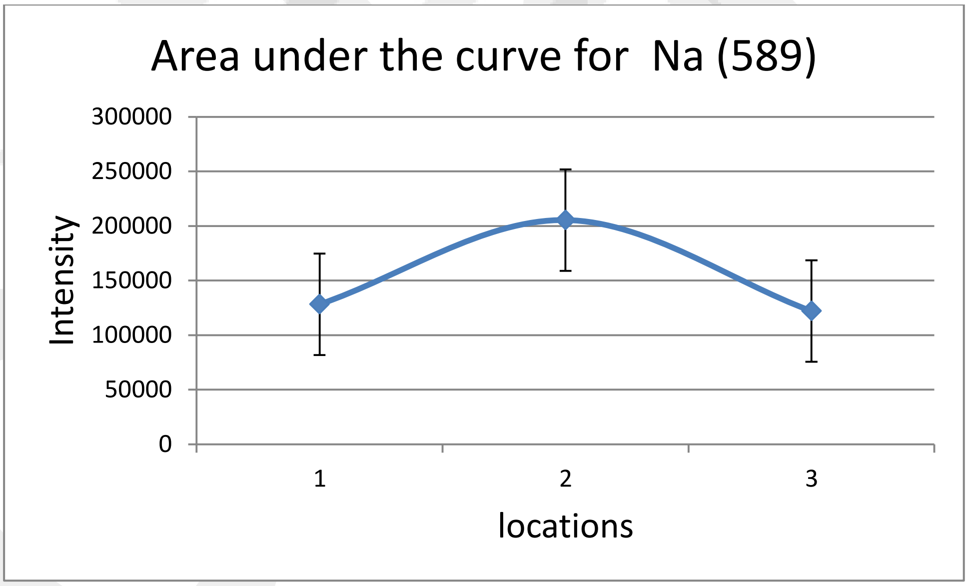


fig (4.31). Area under the curve for Na (589)

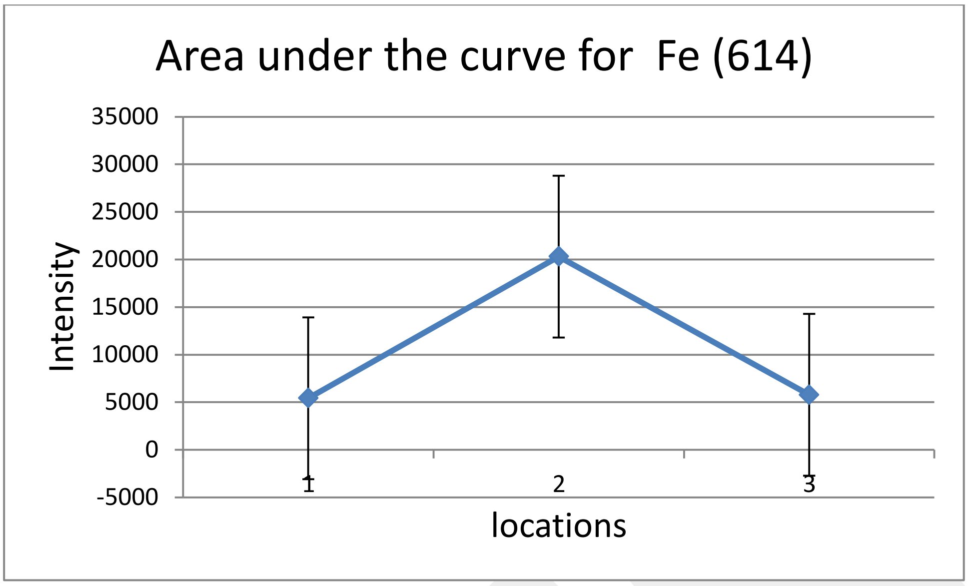


fig (4.32) Area under the curve for Fe (614)

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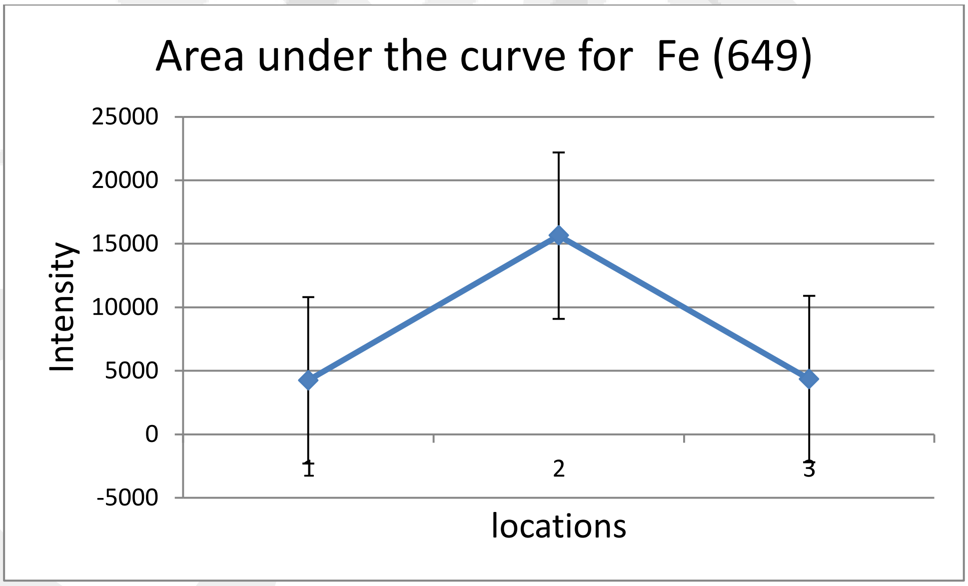

fig (4.33). Area under the curve for Fe (649)

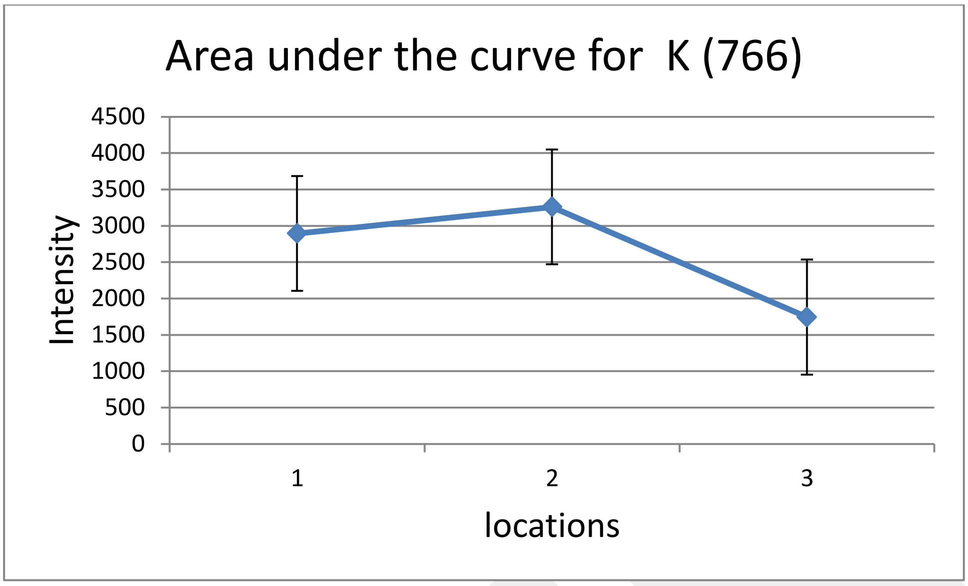

fig (4.34). Area under the curve for K (766)

After that we used 0,1 µs time delay, and power of 30 mJ, with frequency 10 hz, and 50 shots, then we have moved the alloy in three locations, and we got the following results as shown in the following figures.

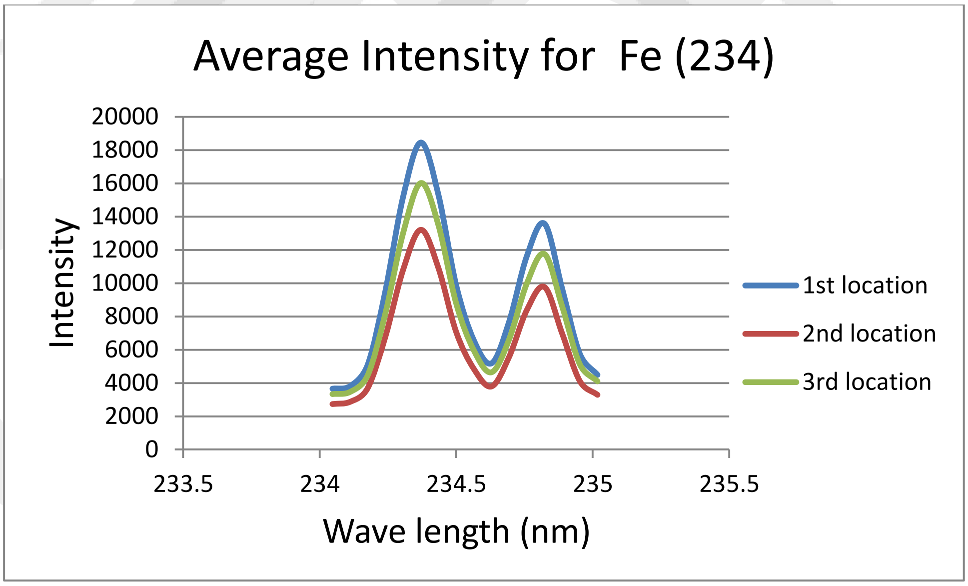

fig (4.35). Average Intensity for Fe (234)

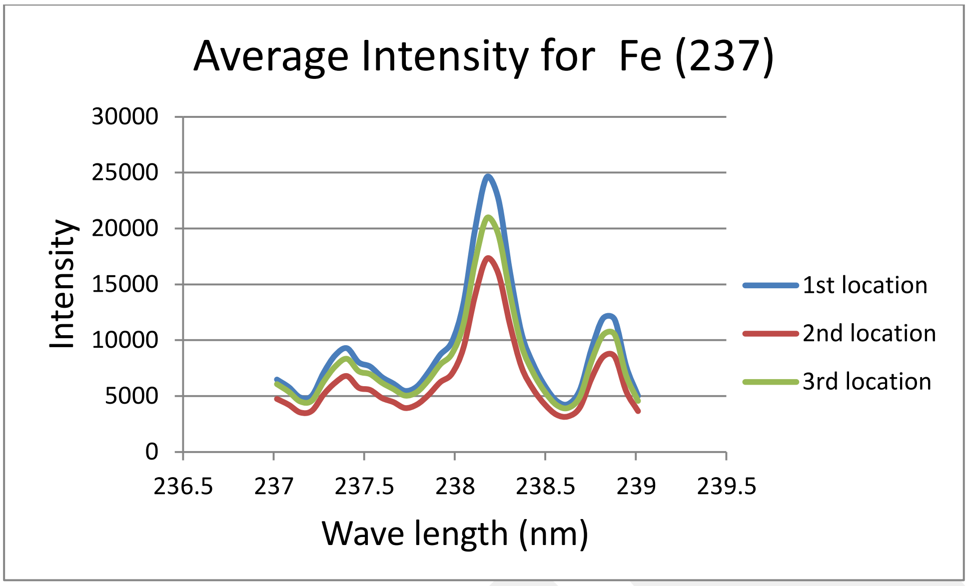

fig (4.36). Average Intensity for Fe (237)

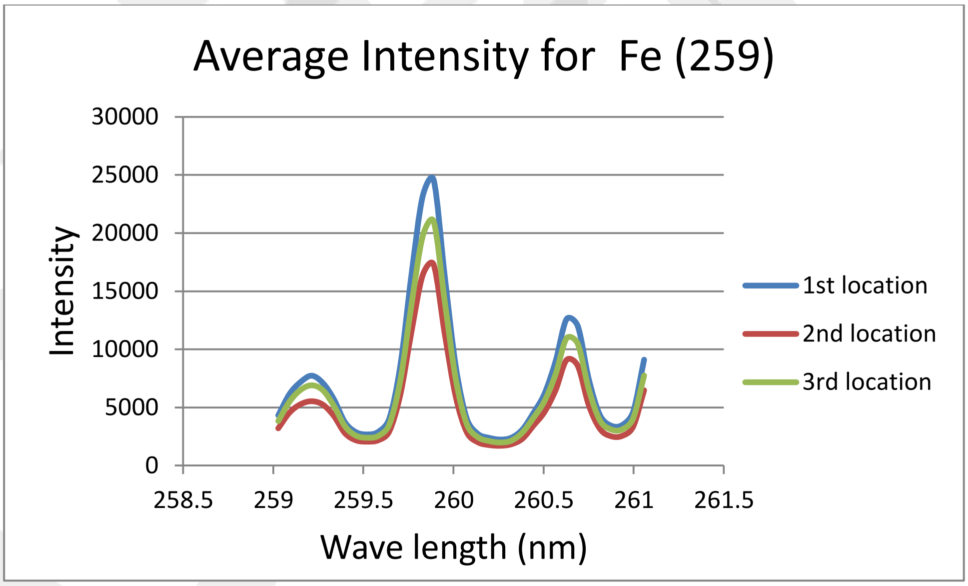

fig (4.37). Average Intensity for Fe (259)

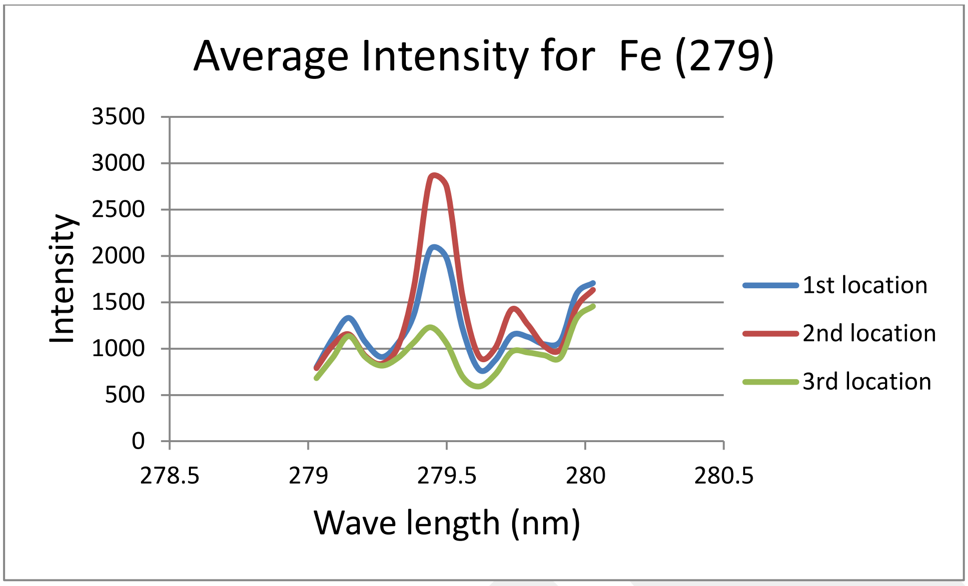

fig (4.38). Average Intensity for Fe (279)

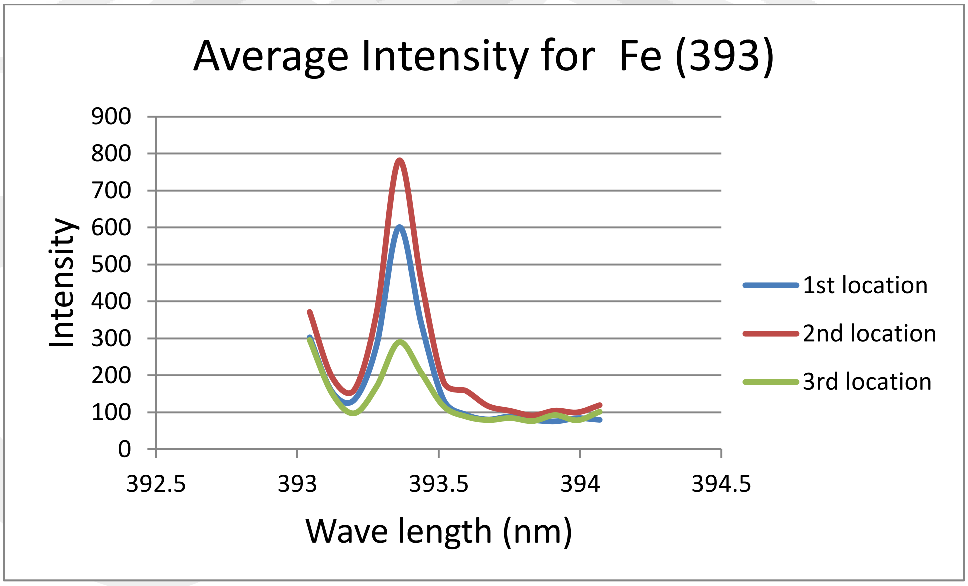

fig (4.39). Average Intensity for Fe (393)

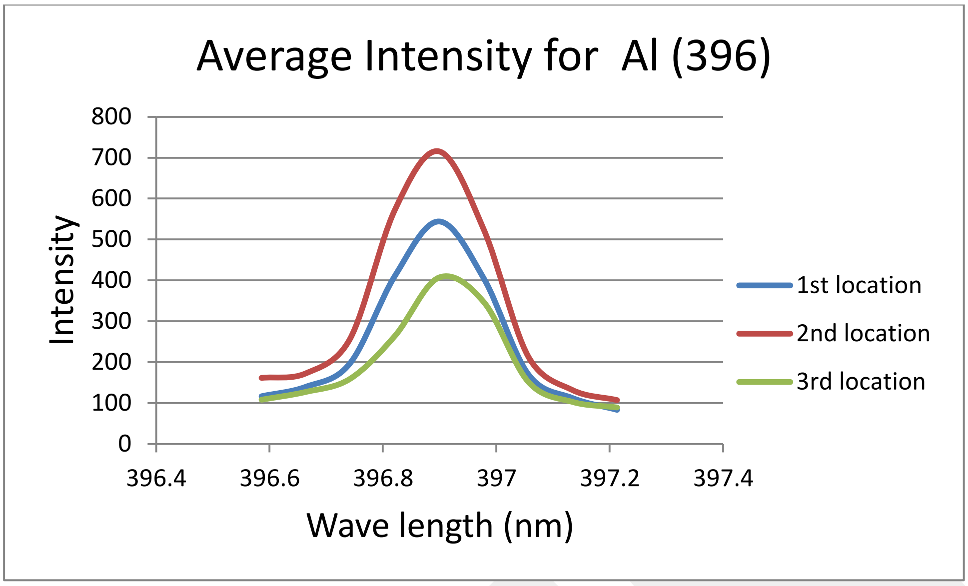

fig (4.40). Average Intensity for Al (396)

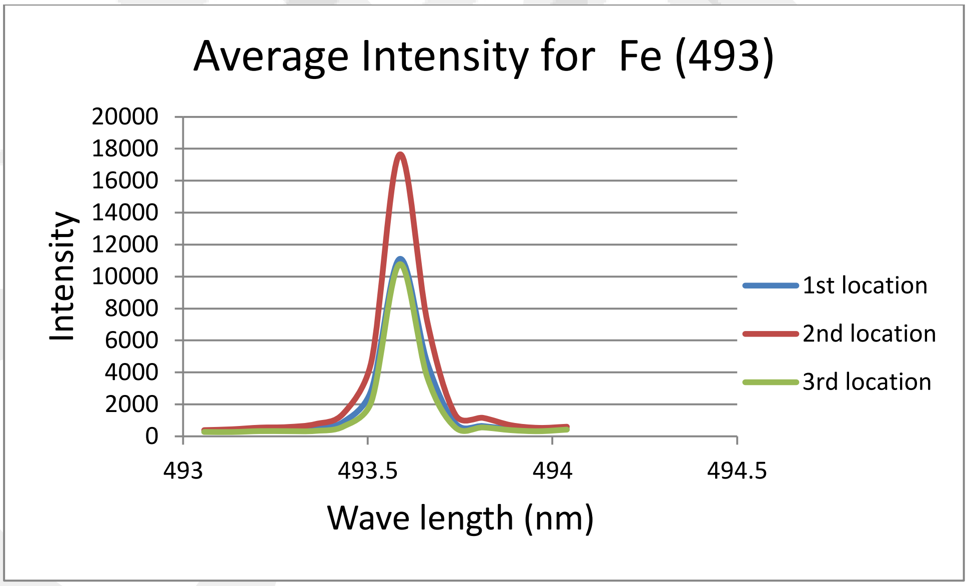

fig (4.41). Average Intensity for Fe (493)

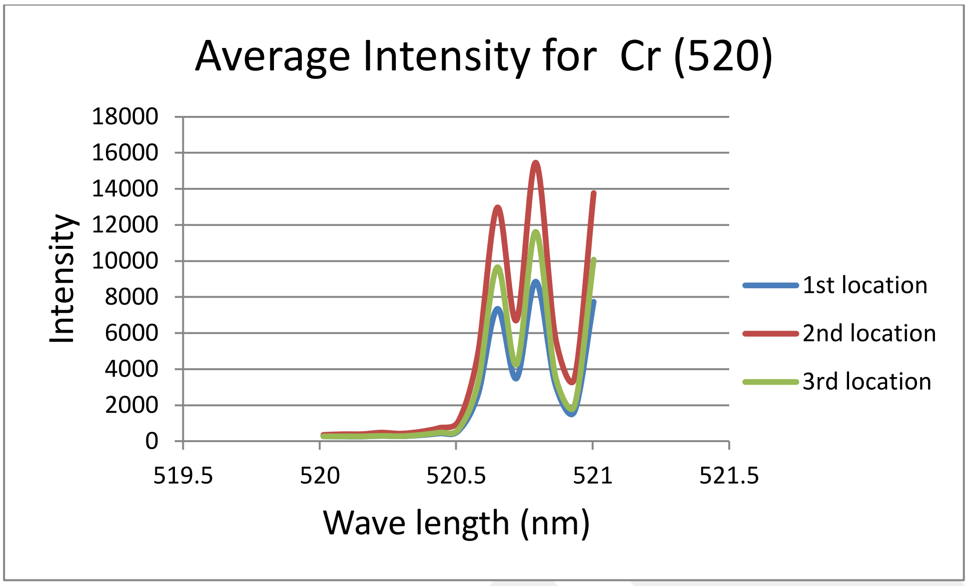

fig (4.42). Average Intensity for Cr (520)

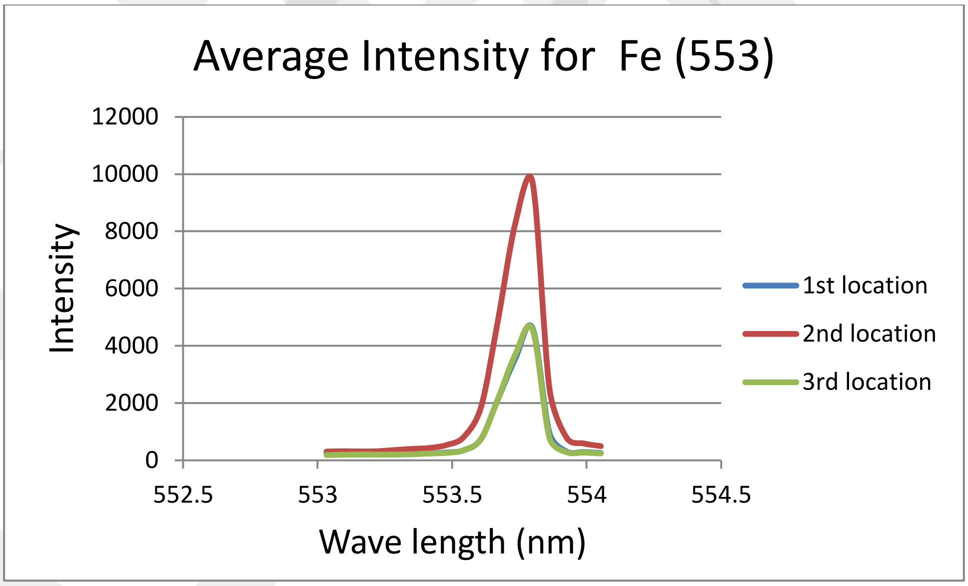

fig (4.43). Average Intensity for Fe (553)

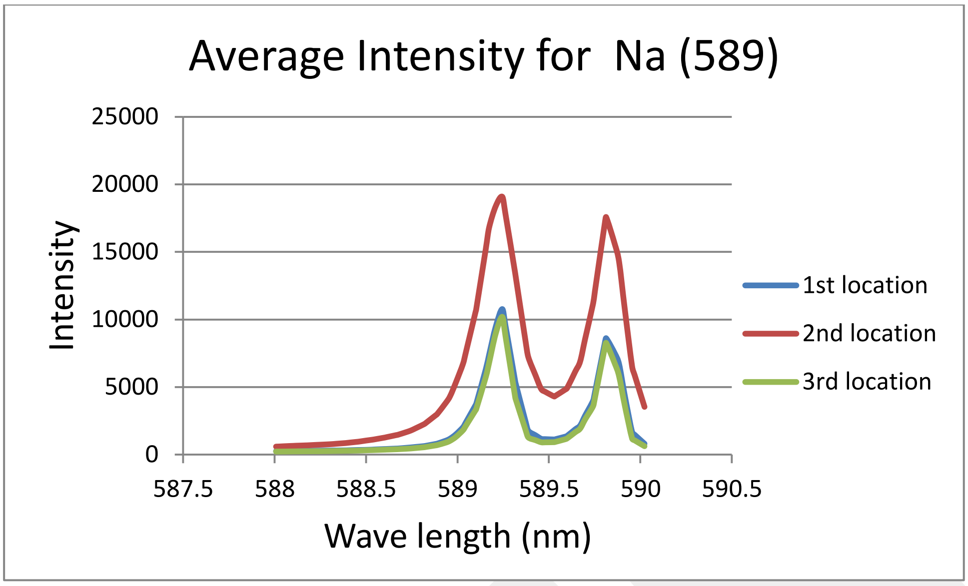

fig (4.44). Average Intensity for Na (589)

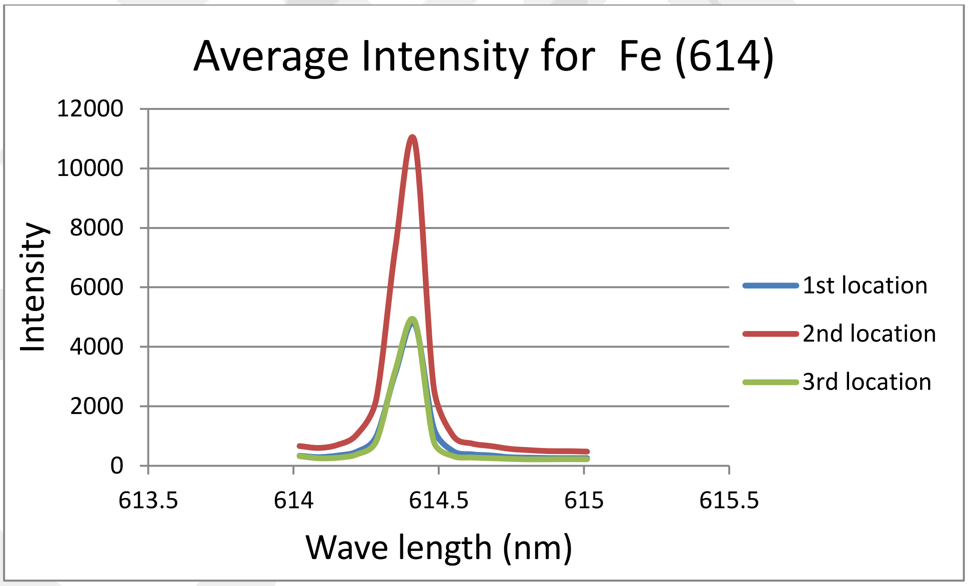

fig (4.45). Average Intensity for Fe (614)

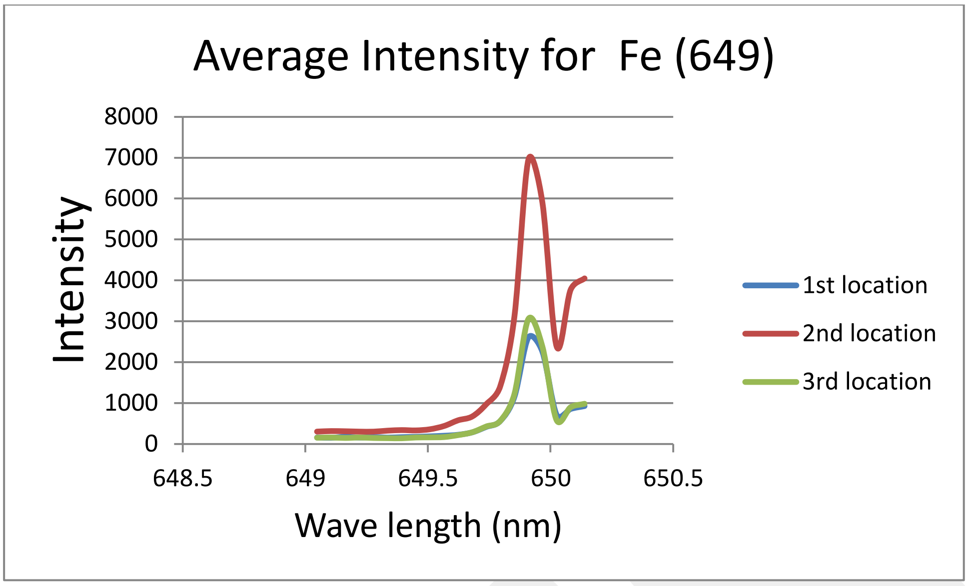

fig (4.46). Average Intensity for Fe (649)

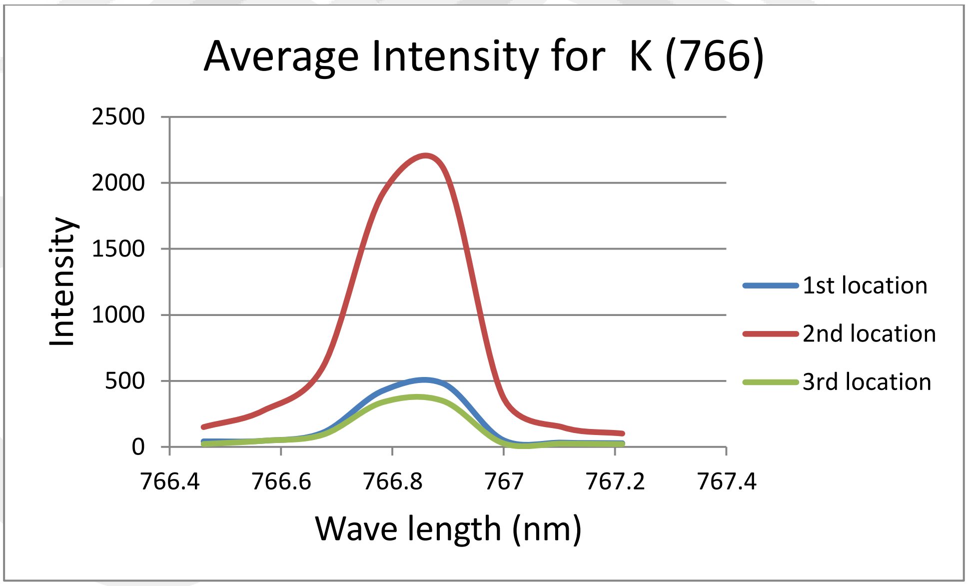

fig (4.47). Average Intensity for K (766)

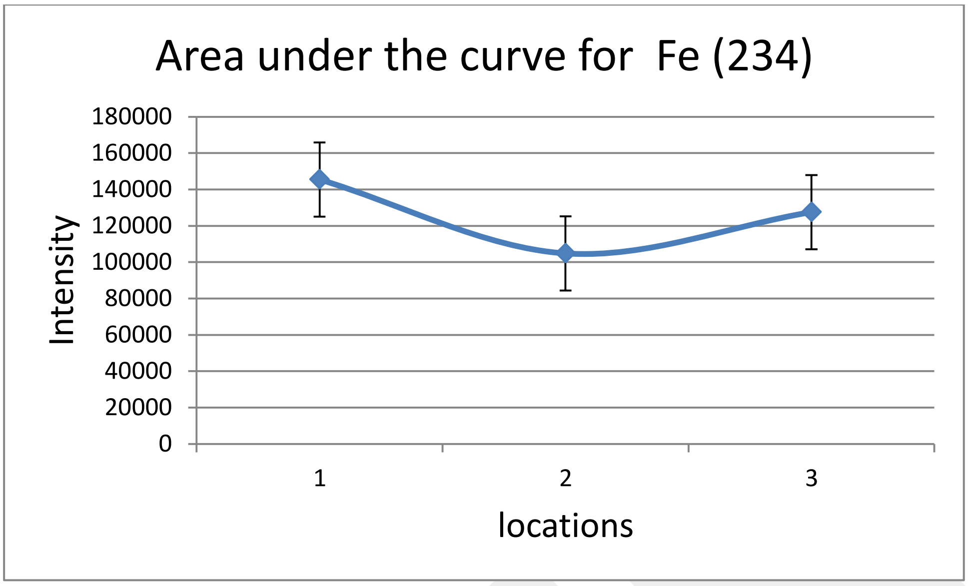

fig (4.48). Area under the curve for Fe (234)

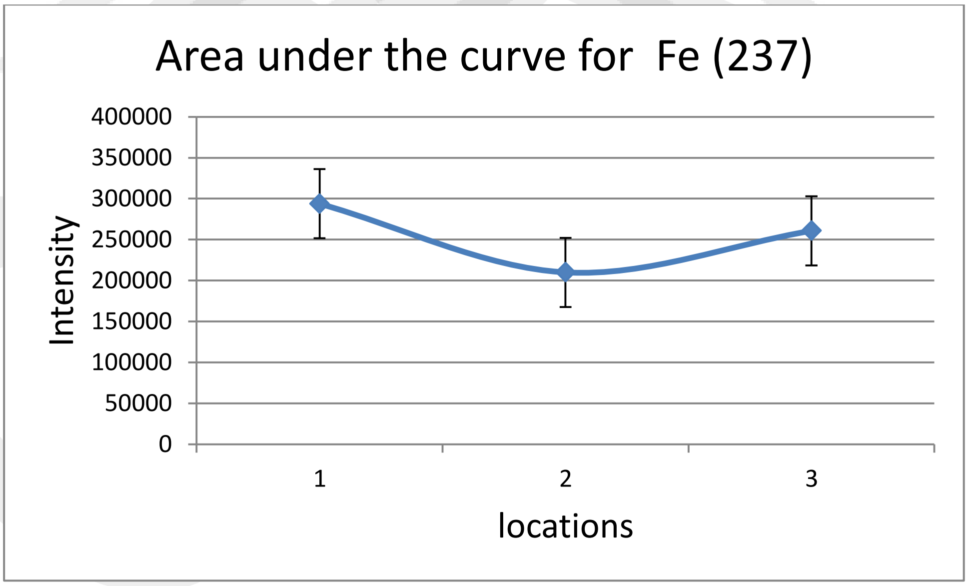

fig (4.49). Area under the curve for Fe (237)

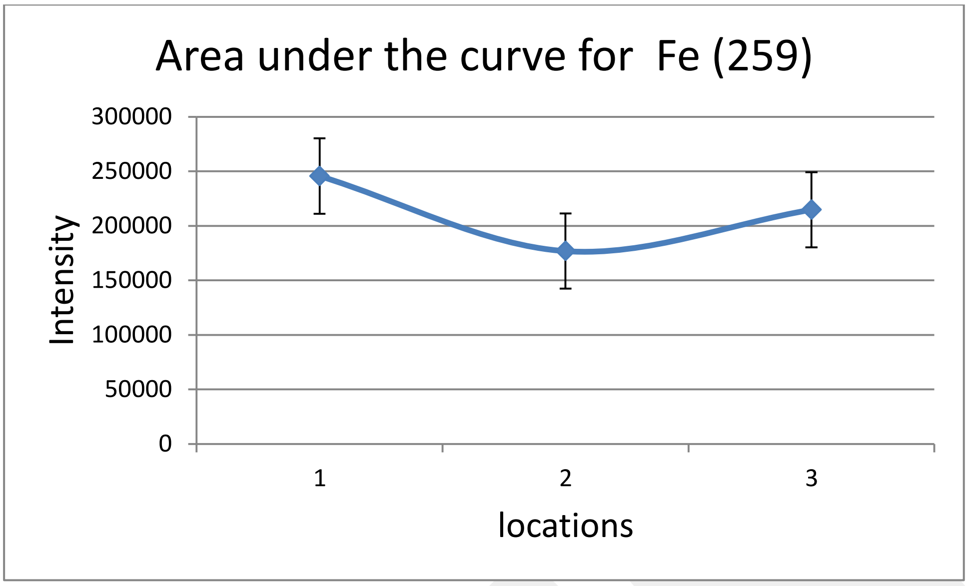

fig (4.50). Area under the curve for Fe (259)

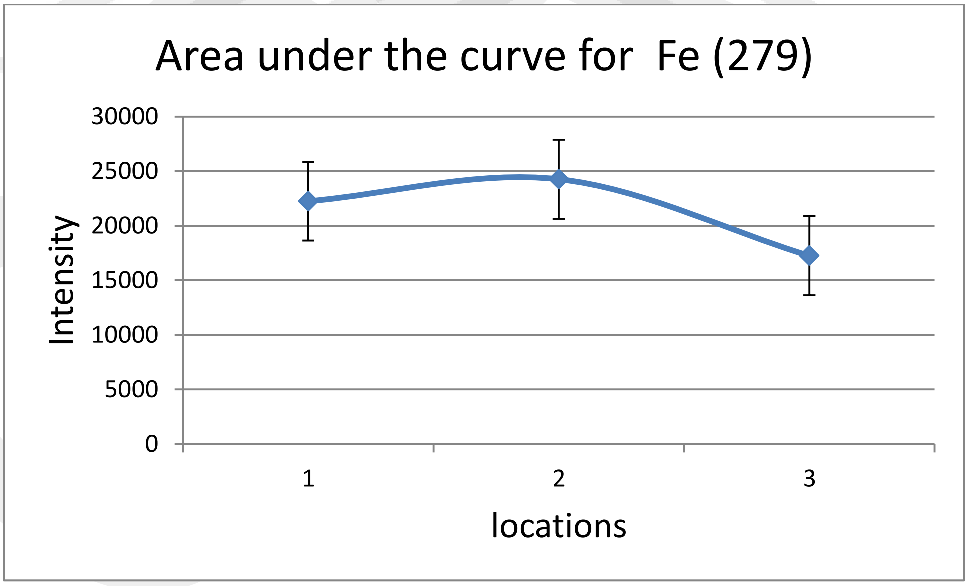

fig (4.51). Area under the curve for Fe (279)

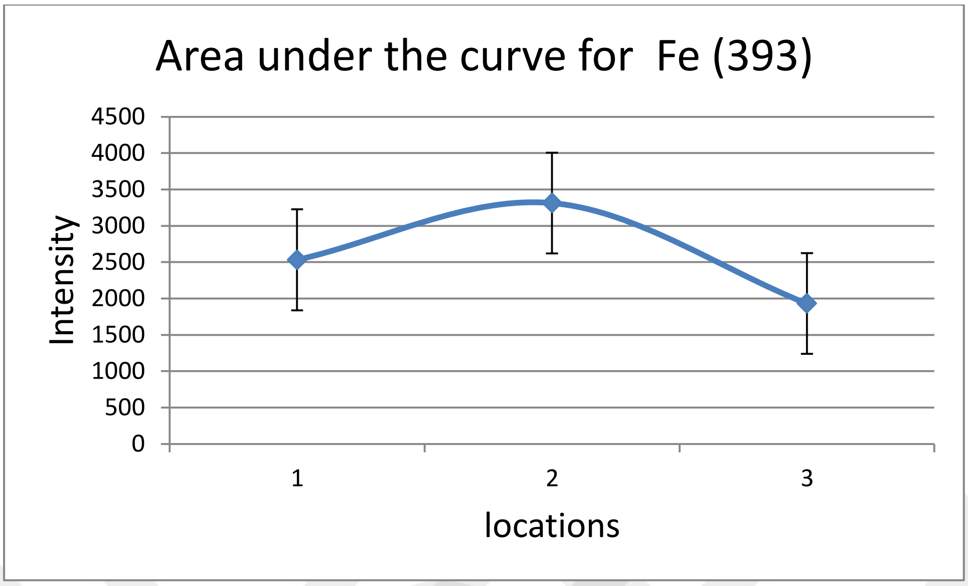

fig (4.52). Area under the curve for Fe (393)

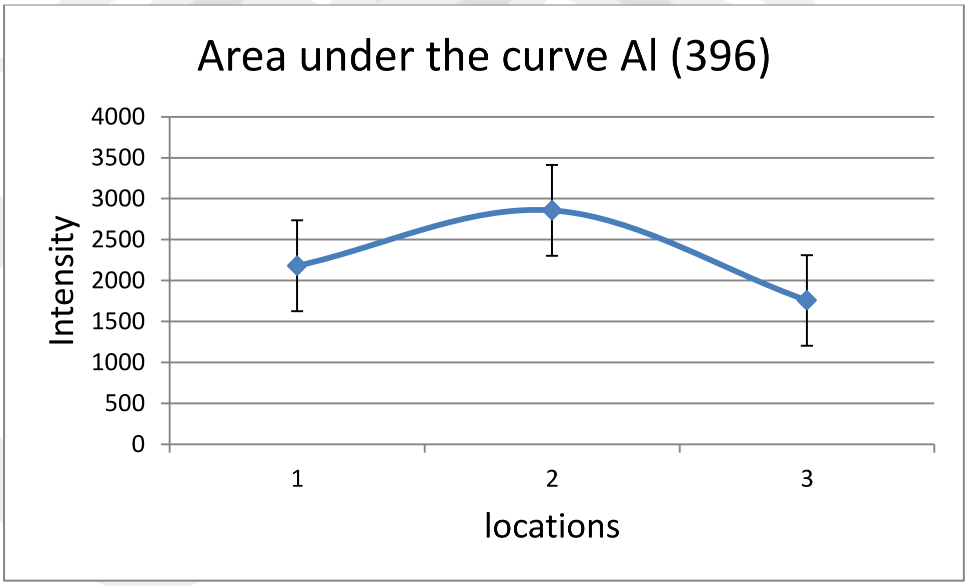

fig (4.53). Area under the curve for Al (396)

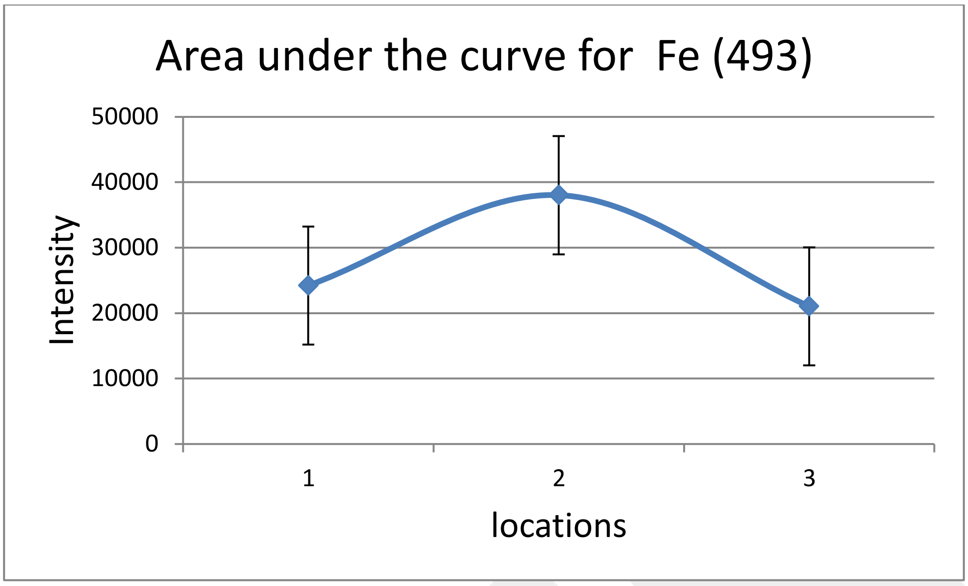

fig (4.54). Area under the curve for Fe (493)

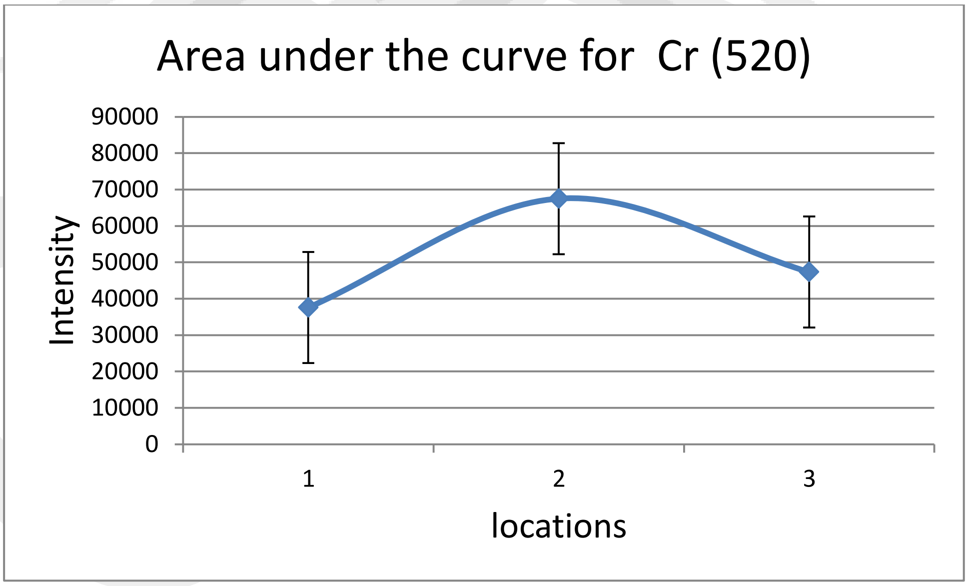

fig (4.55). Area under the curve for Cr (520)

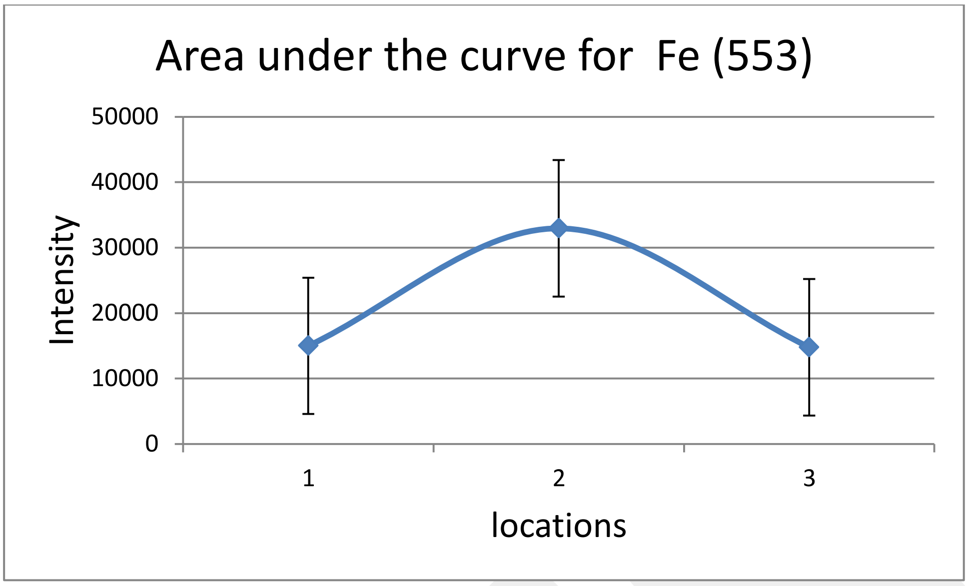

fig (4.56). Area under the curve for Fe (553)

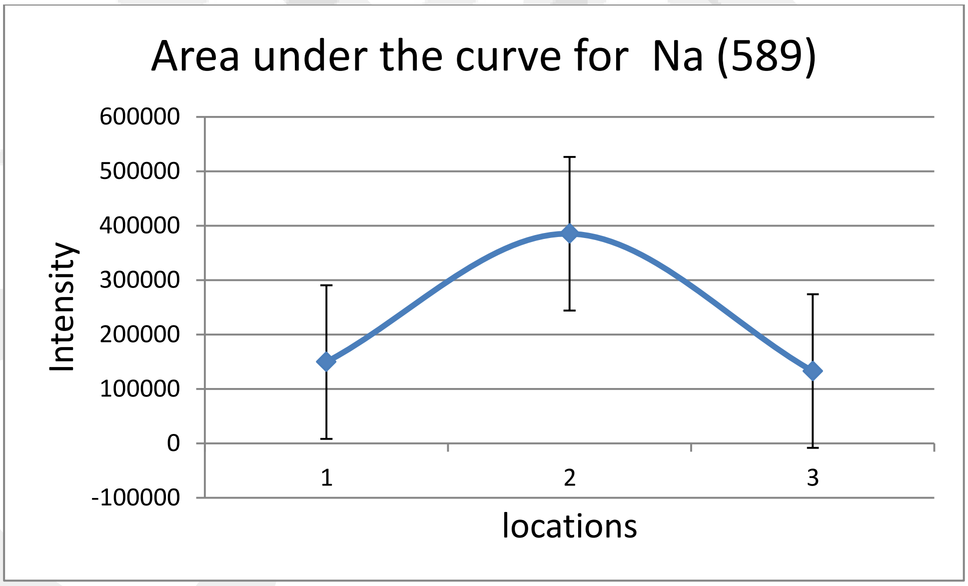

fig (4.57). Area under the curve for Na (589)

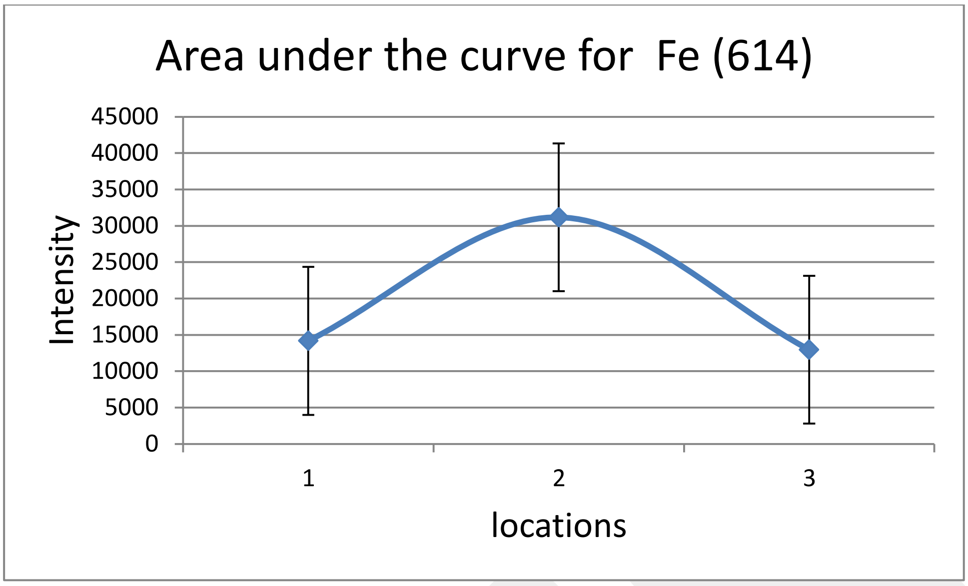

fig (4.58). Area under the curve for Fe (614)

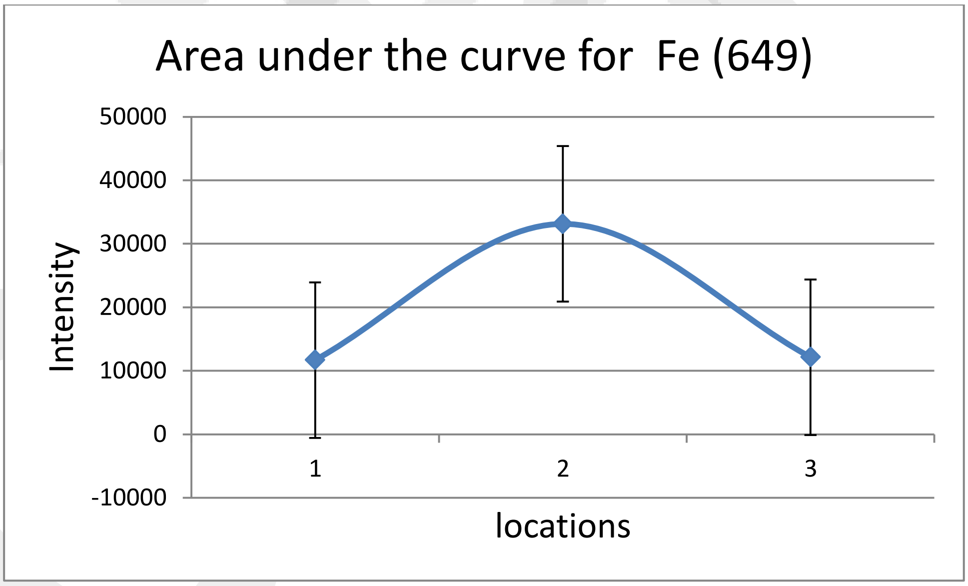

fig (4.59). Area under the curve for Fe (649)

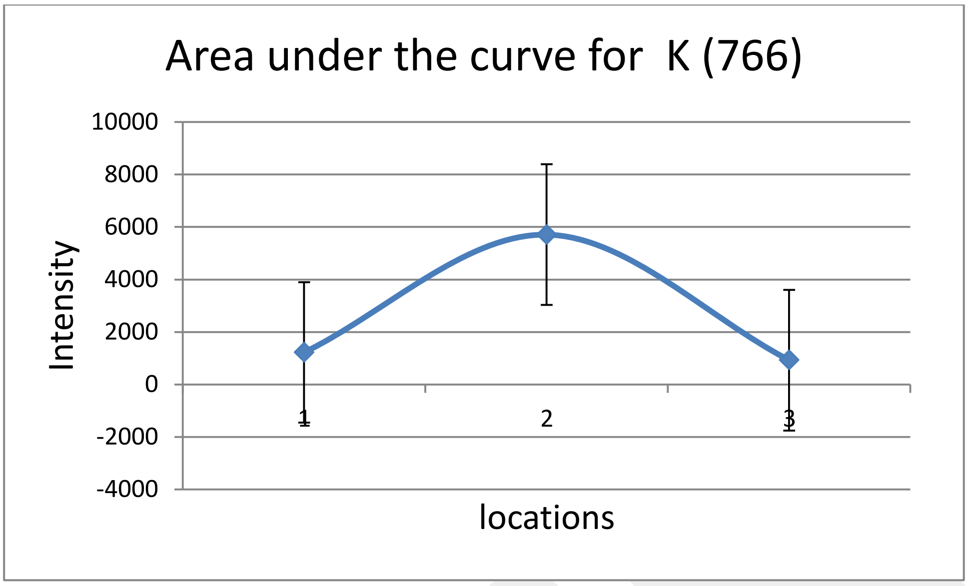

fig (4.60). Area under the curve for K (766)

After that we used 0,1 µs time delay, and power of 40 mJ, with frequency 10 hz, and 50 shots, then we have moved the alloy in three locations, and we got the following results as shown in the following figures.

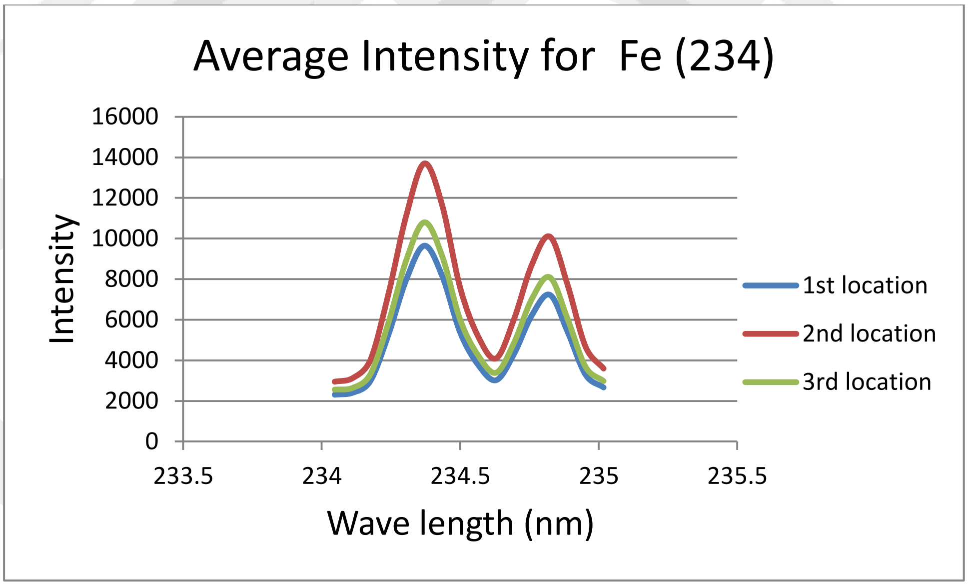

fig (4.61). Average Intensity for Fe (234)

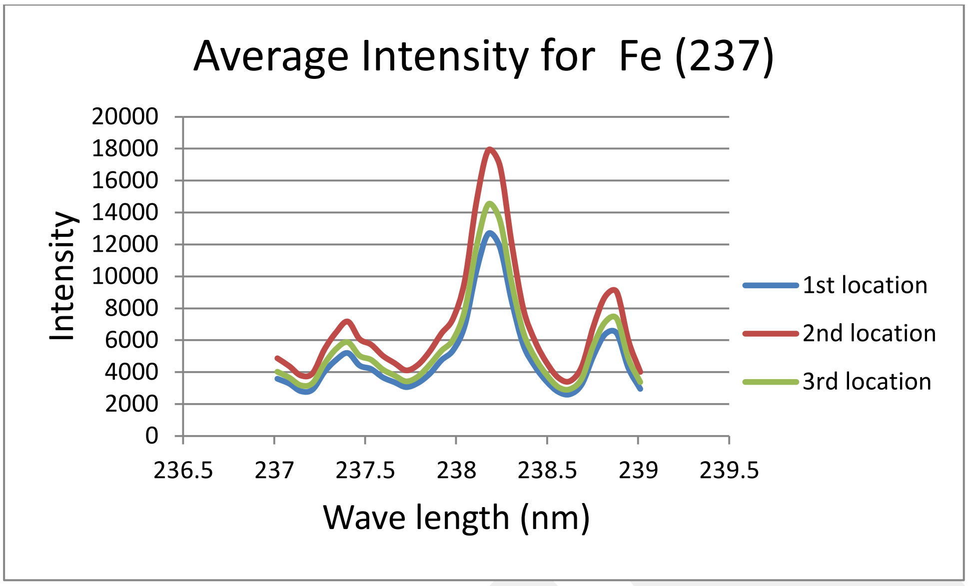

fig (4.62). Average Intensity for Fe (237)

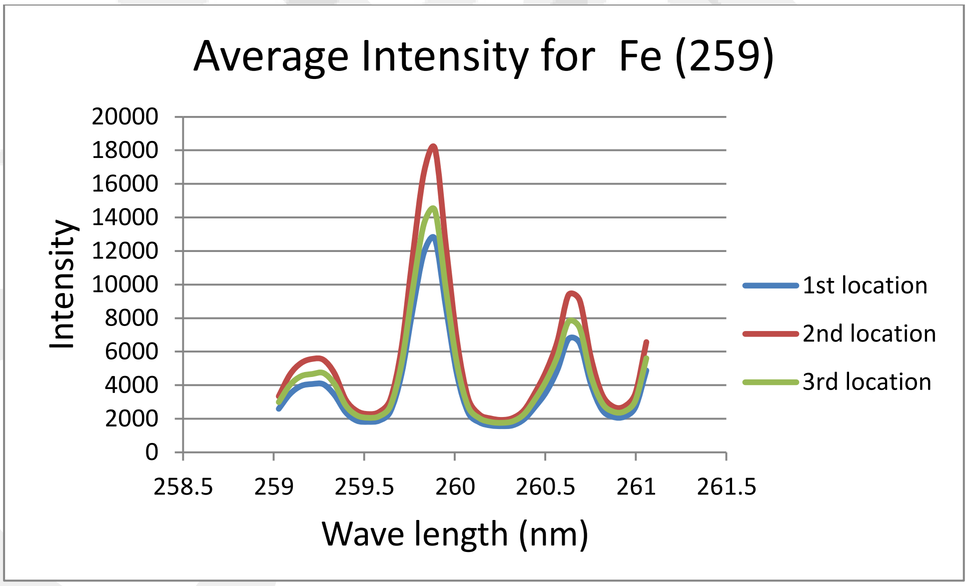

fig (4.63). Average Intensity for Fe (259)

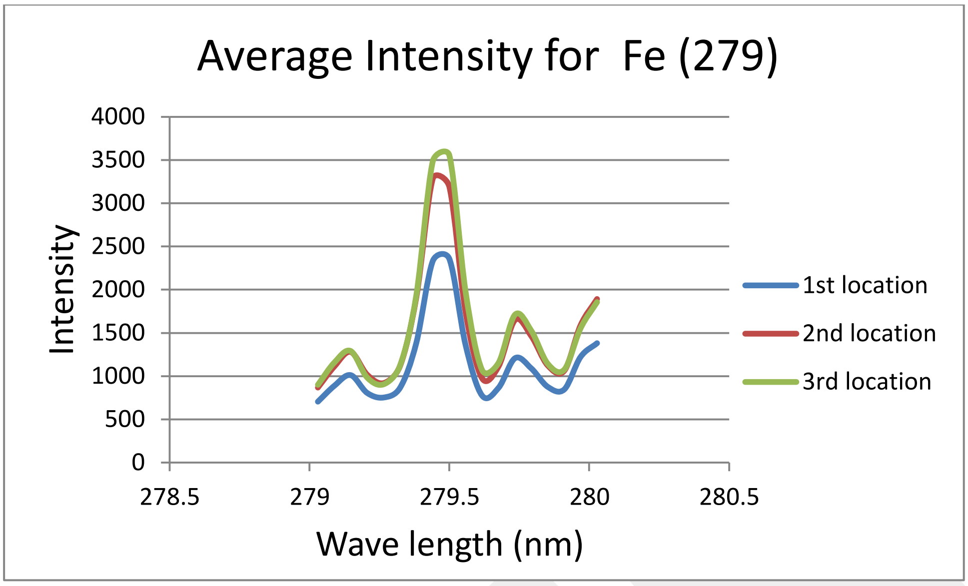

fig (4.64). Average Intensity for Fe (279)

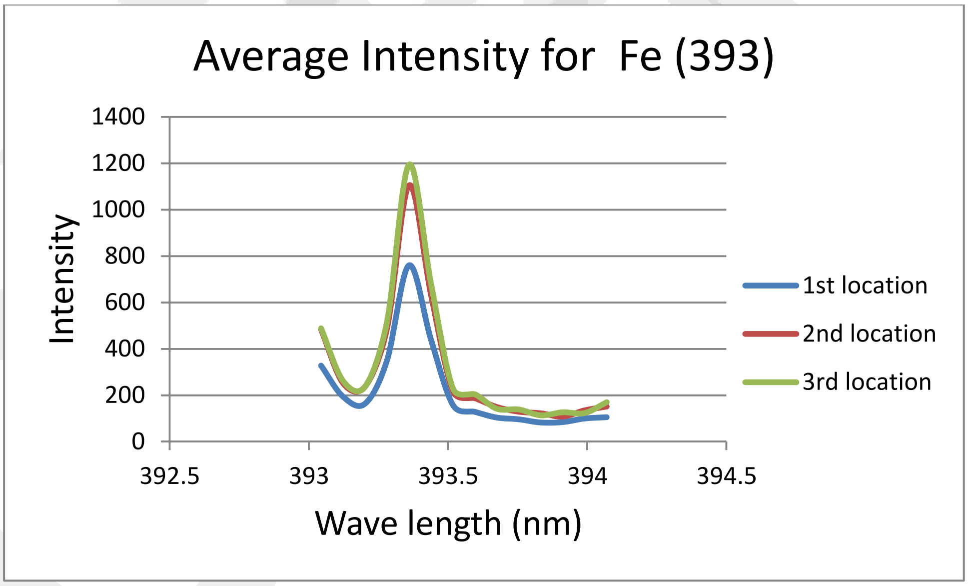

fig (4.65). Average Intensity for Fe (393)

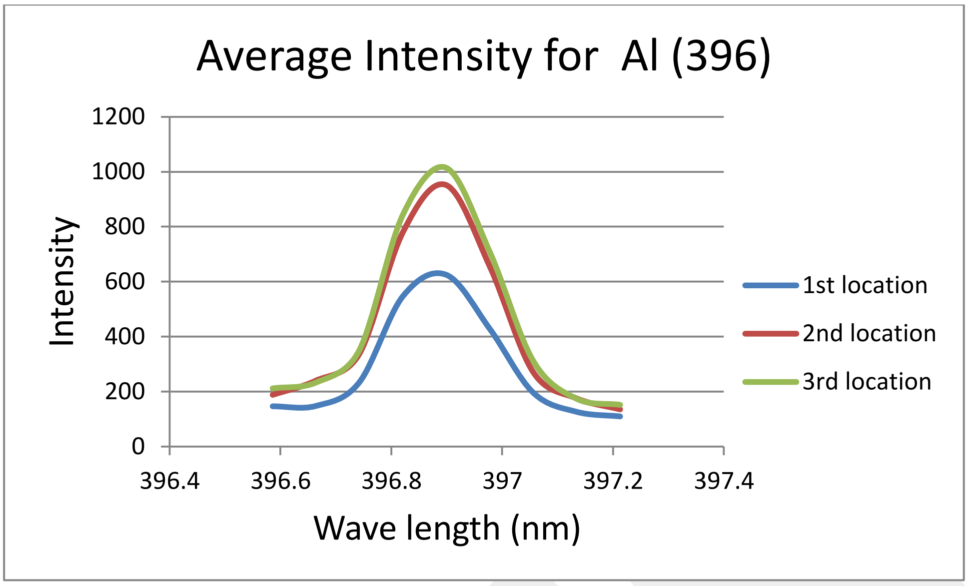

fig (4.66). Average Intensity for Al (396)

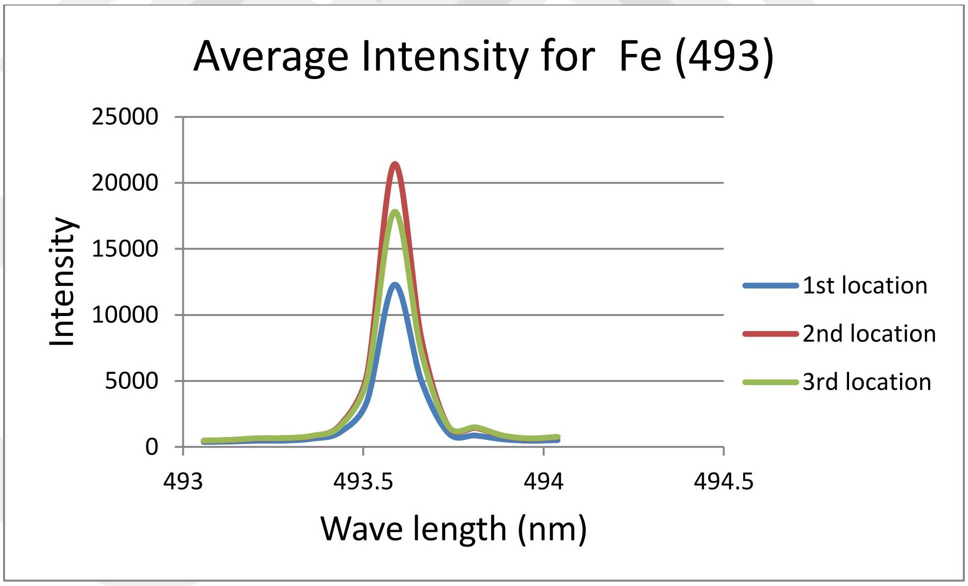

fig (4.67). Average Intensity for Fe (493)

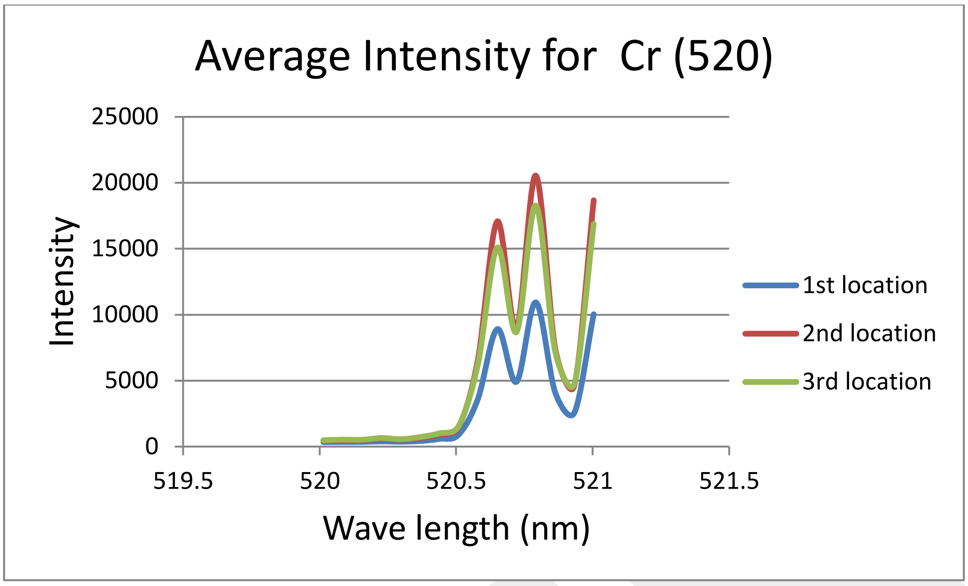

fig (4.68). Average Intensity for Cr (520)

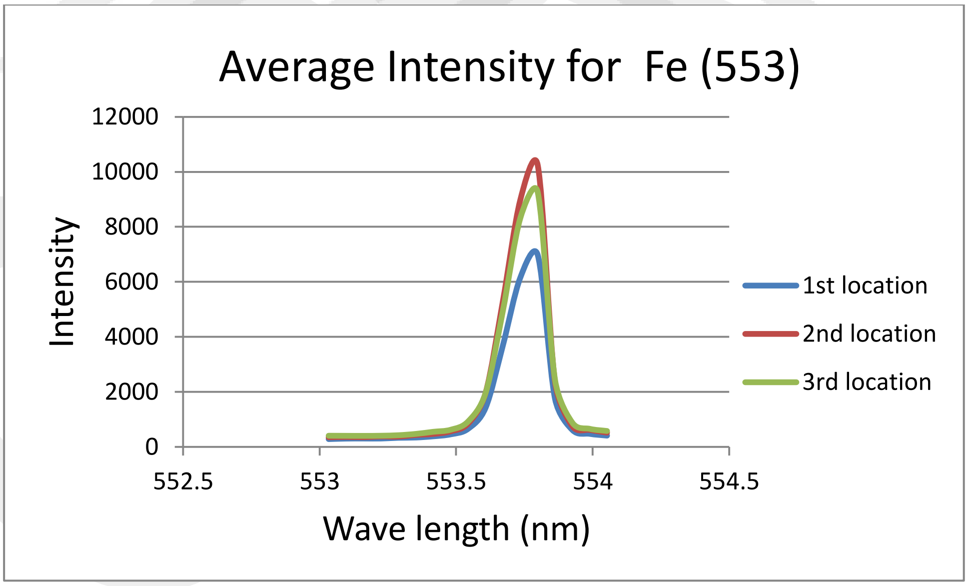

fig (4.69). Average Intensity for Fe (553)

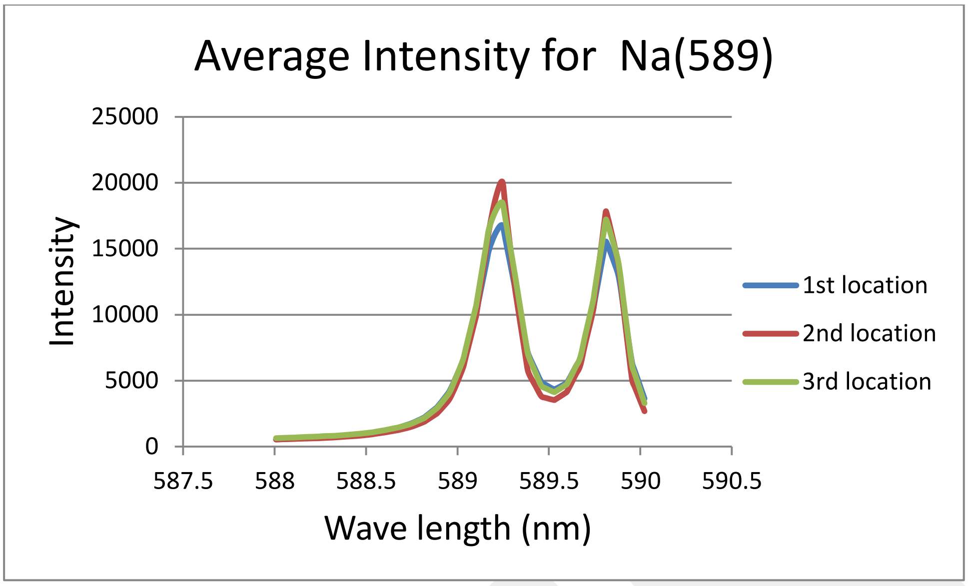

fig (4.70). Average Intensity for Na (589)

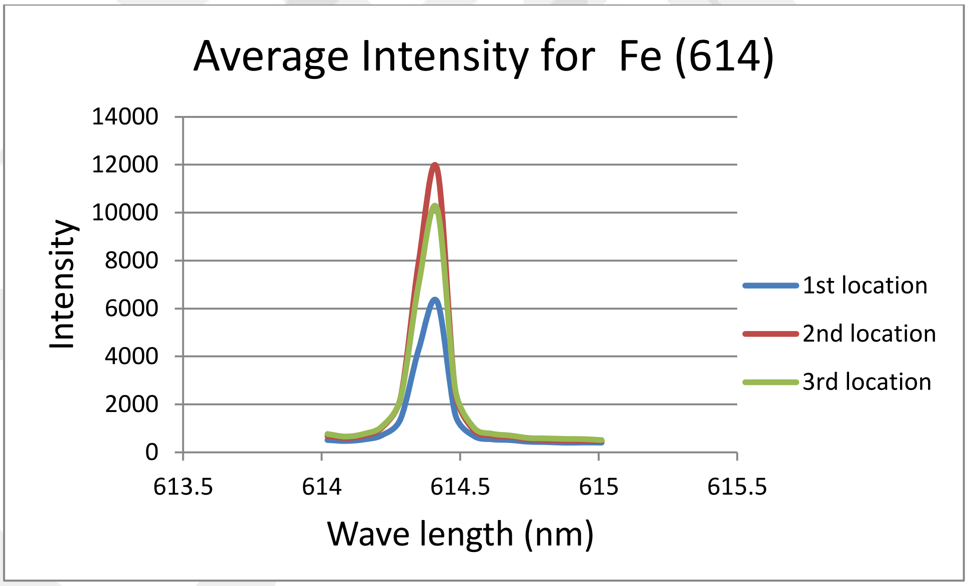

fig (4.71). Average Intensity for Fe (614)

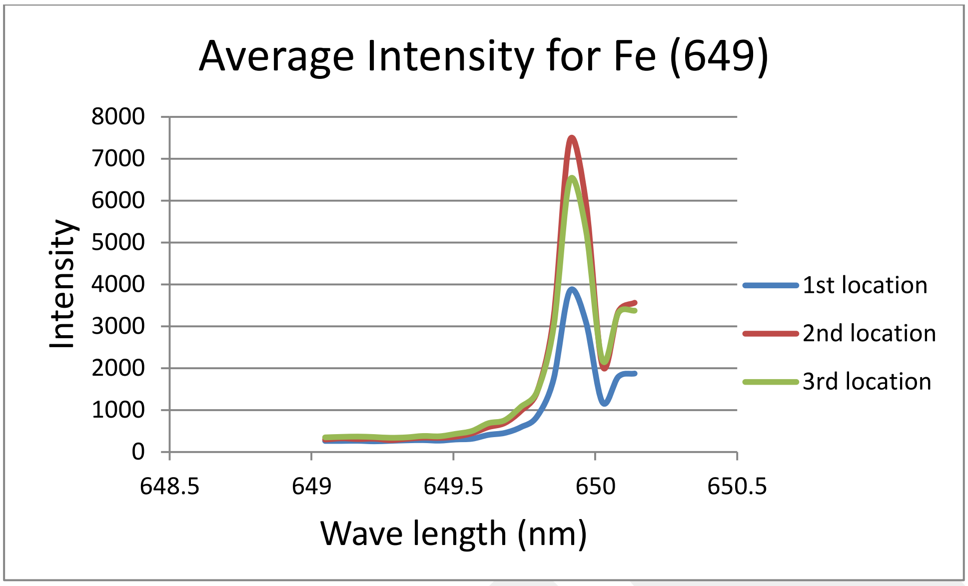

fig (4.72). Average Intensity for Fe (649)

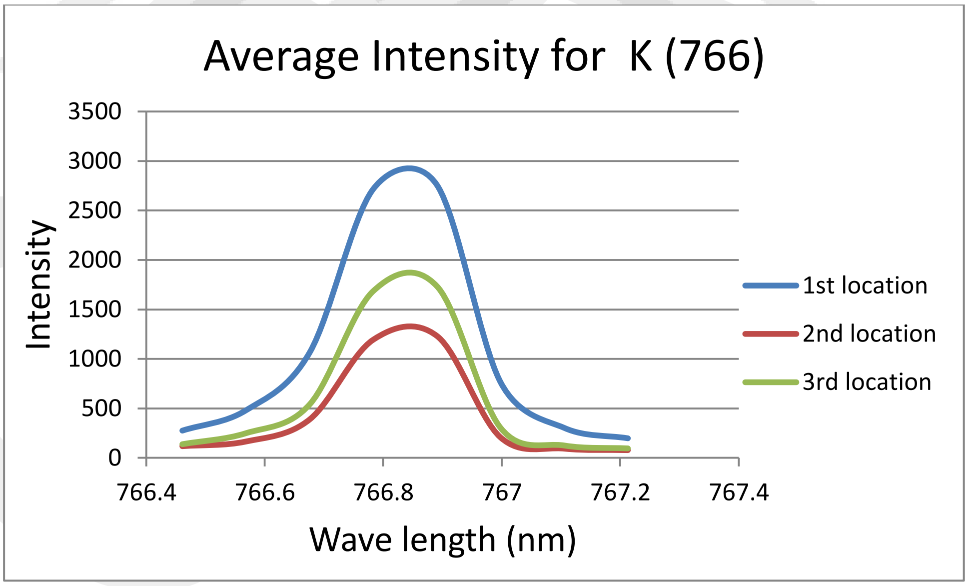

fig (4.73). Average Intensity for K (766)

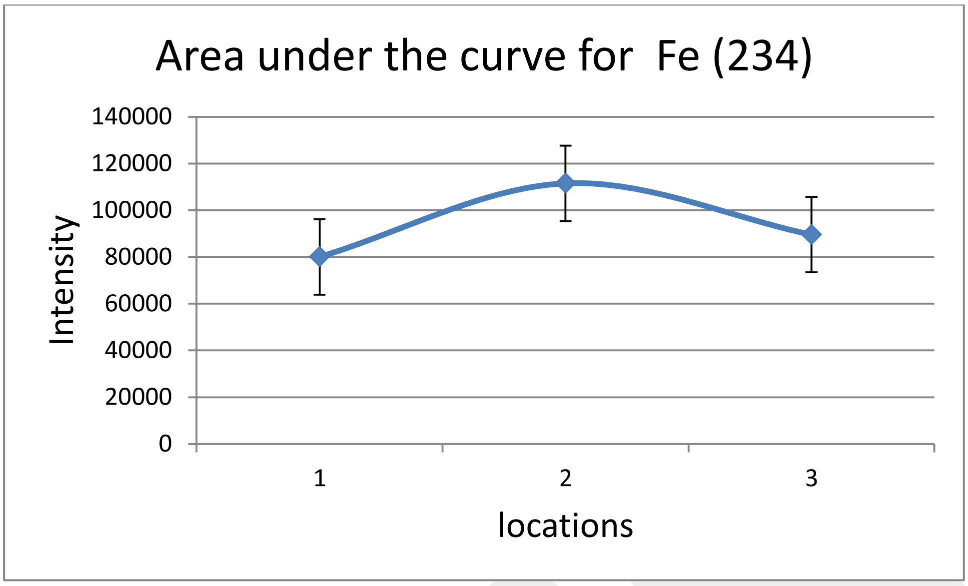

fig (4.74). Area under the curve for Fe (234)

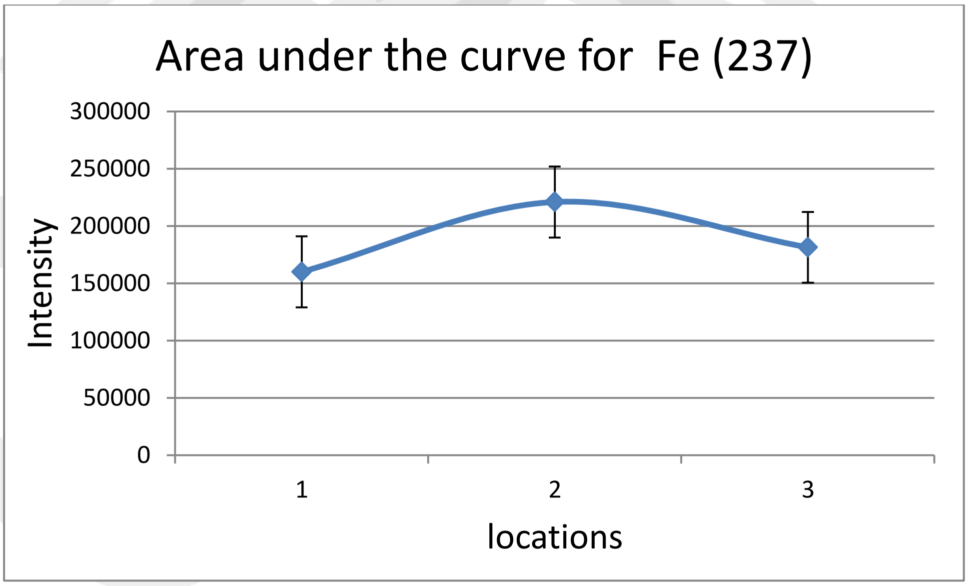

fig (4.75). Area under the curve for Fe (237)

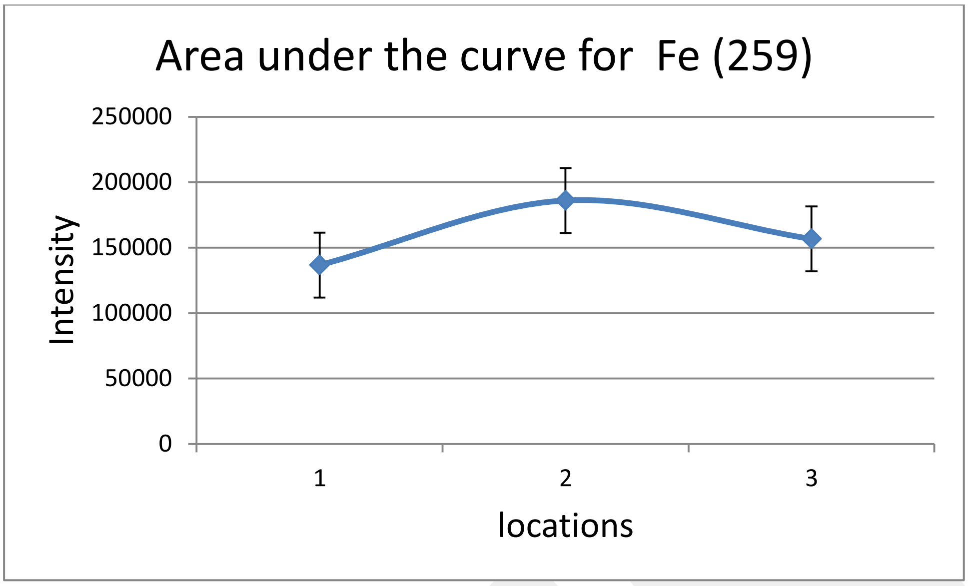

fig (4.76). Area under the curve for Fe (259)

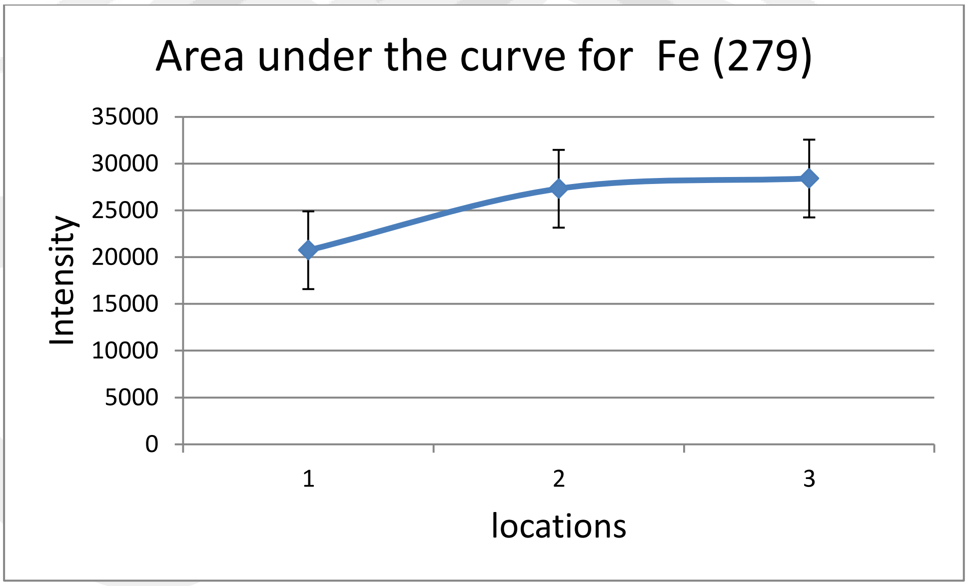

fig (4.77). Area under the curve for Fe (279)

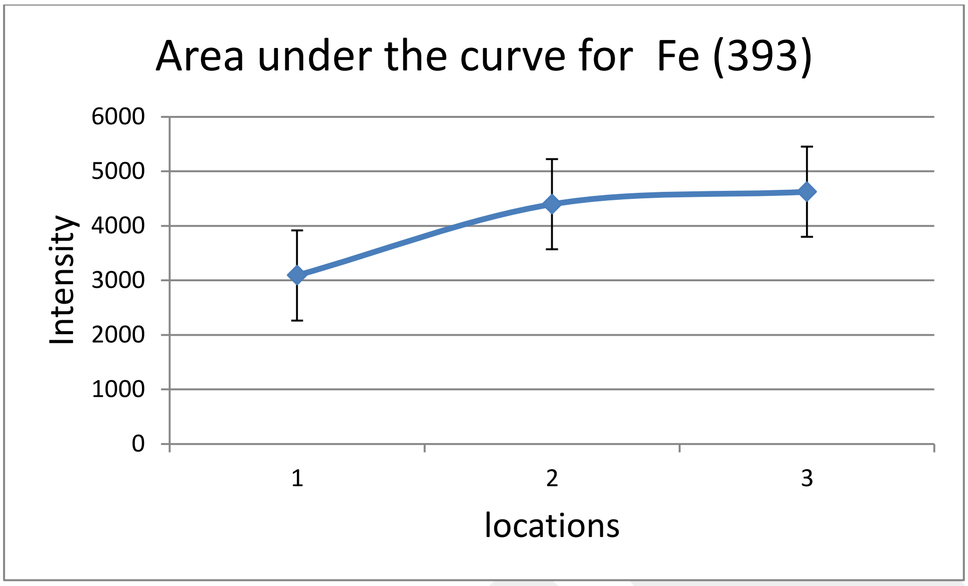

fig (4.78). Area under the curve for Fe (393)

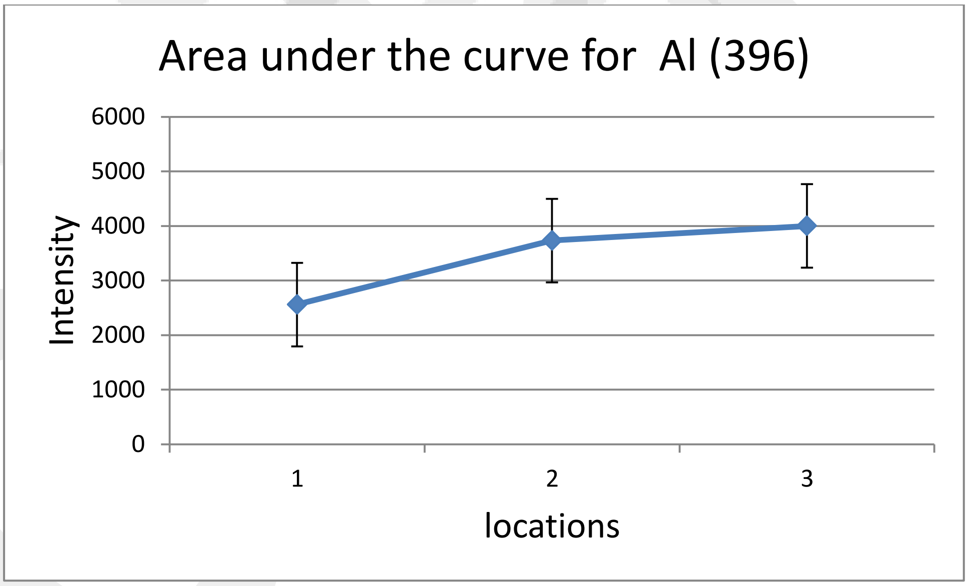

fig (4.79). Area under the curve for Al (396)

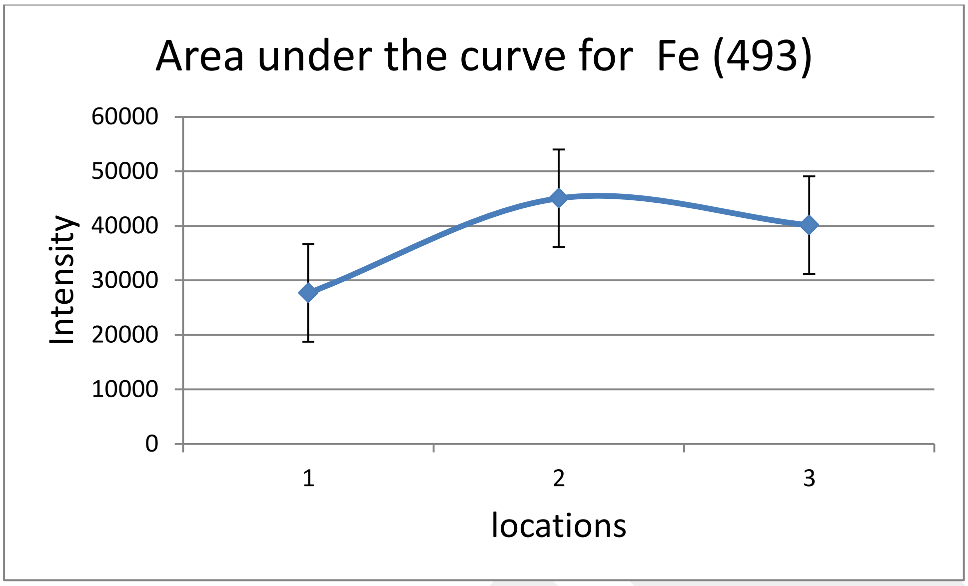

fig (4.80). Area under the curve for Fe (493)

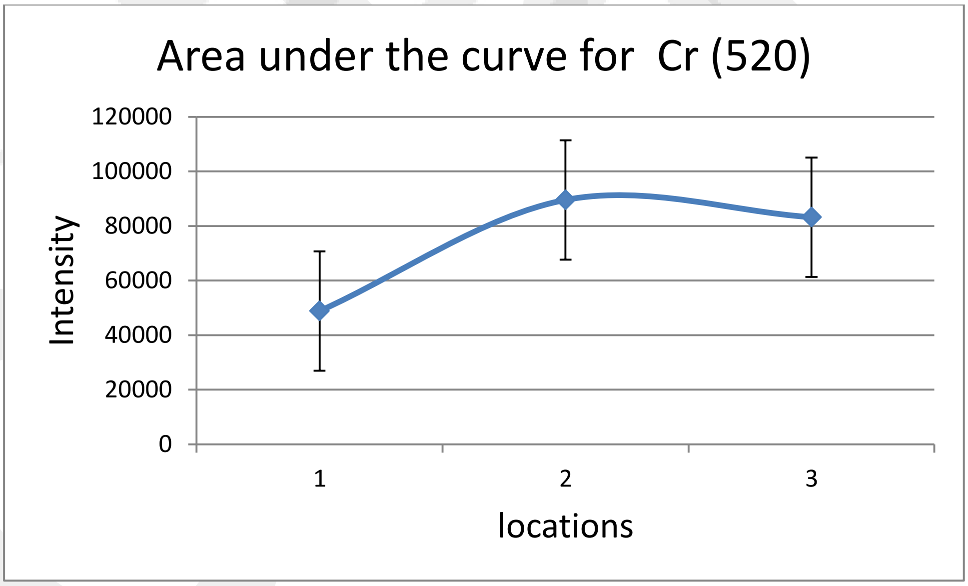

fig (4.81). Area under the curve for Cr (520)

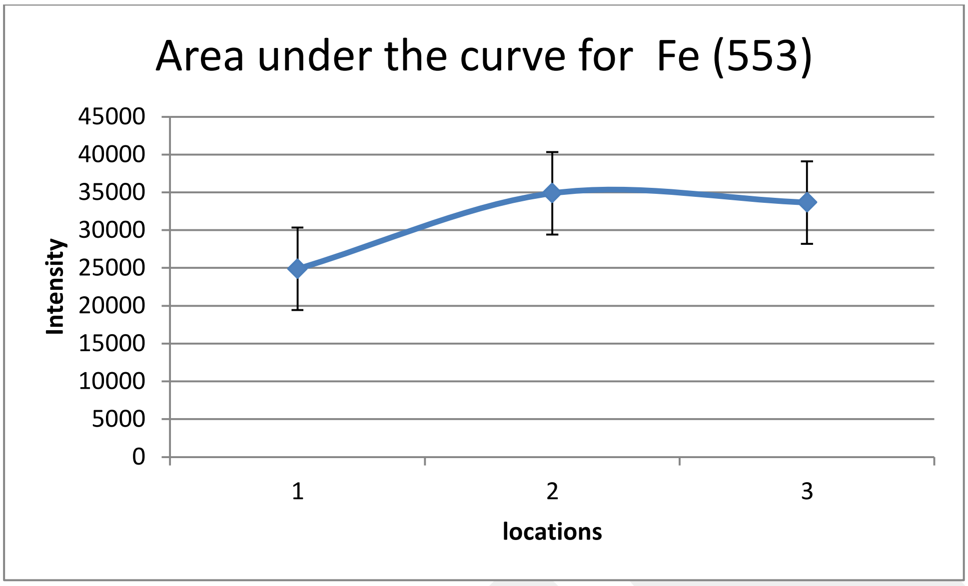

fig (4.82). Area under the curve for Fe (553)

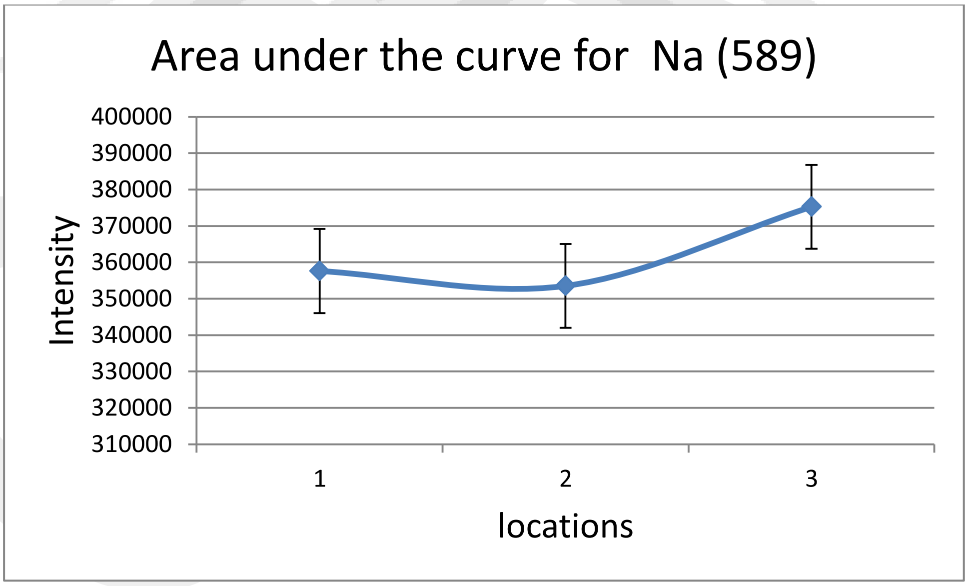

fig (4.83). Area under the curve for Na (589)

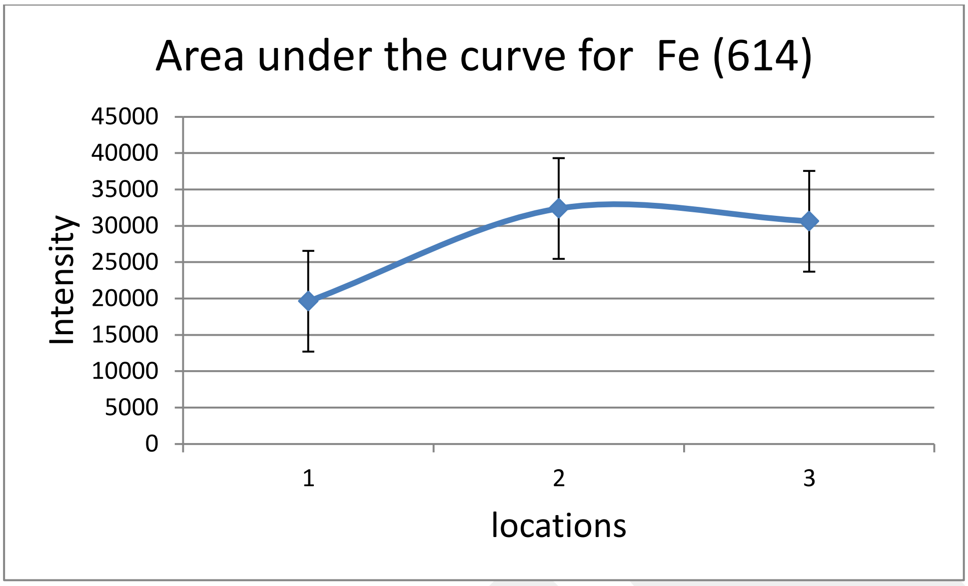

fig (4.84). Area under the curve for Fe (614)

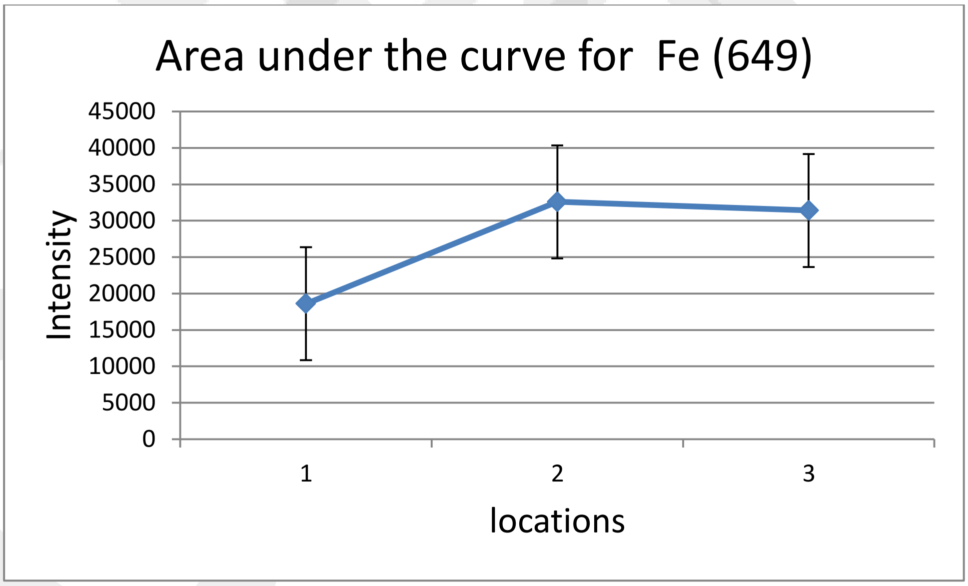

fig (4.85). Area under the curve for Fe (649)

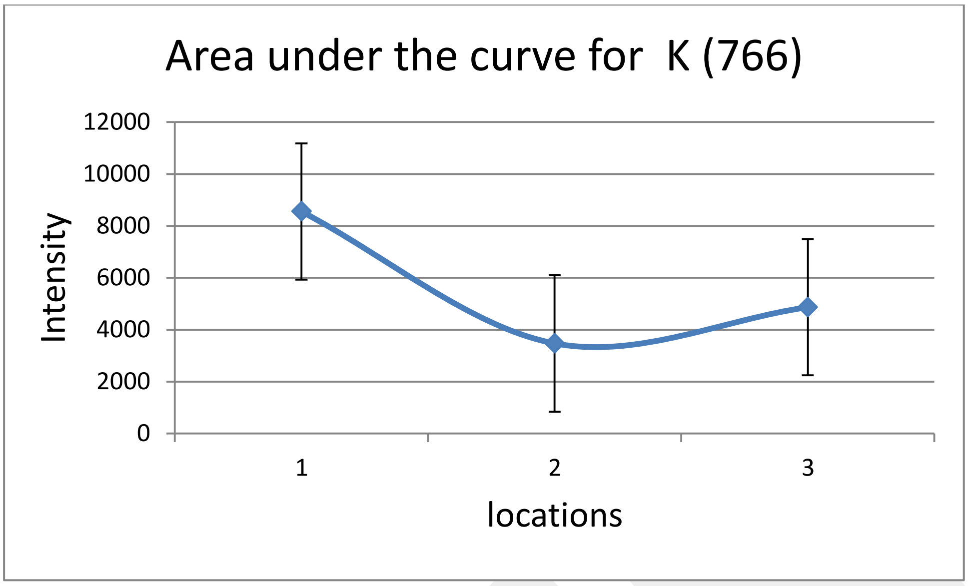

fig (4.86). Area under the curve for K (766)

After that we used 0,3 μs time delay, and power of 20 mJ, with frequency 10 hz, and 50 shots, then we have moved the alloy in three locations, and we got the following results as shown in the following figures.

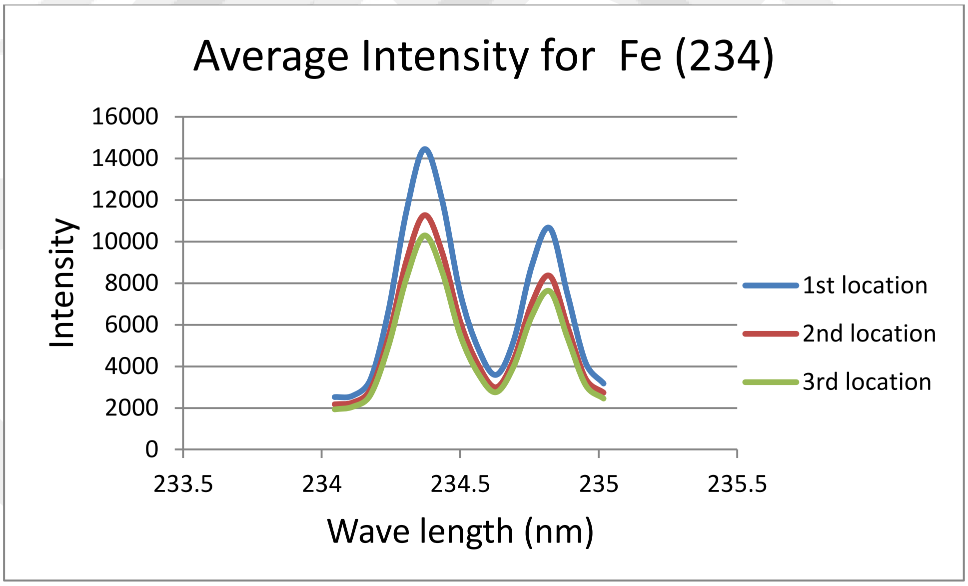

fig (4.87). Average Intensity for Fe (234)

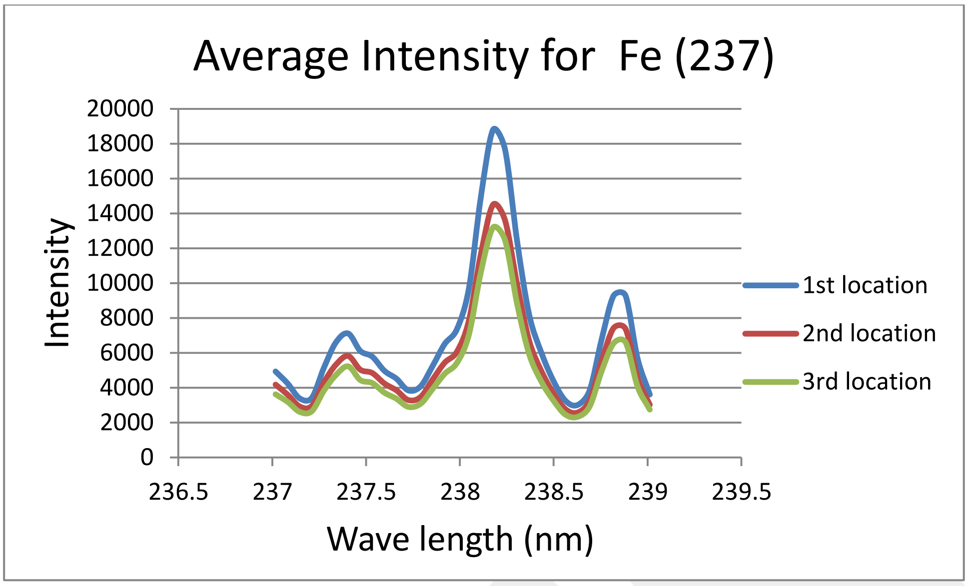

fig (4.88). Average Intensity for Fe (237)

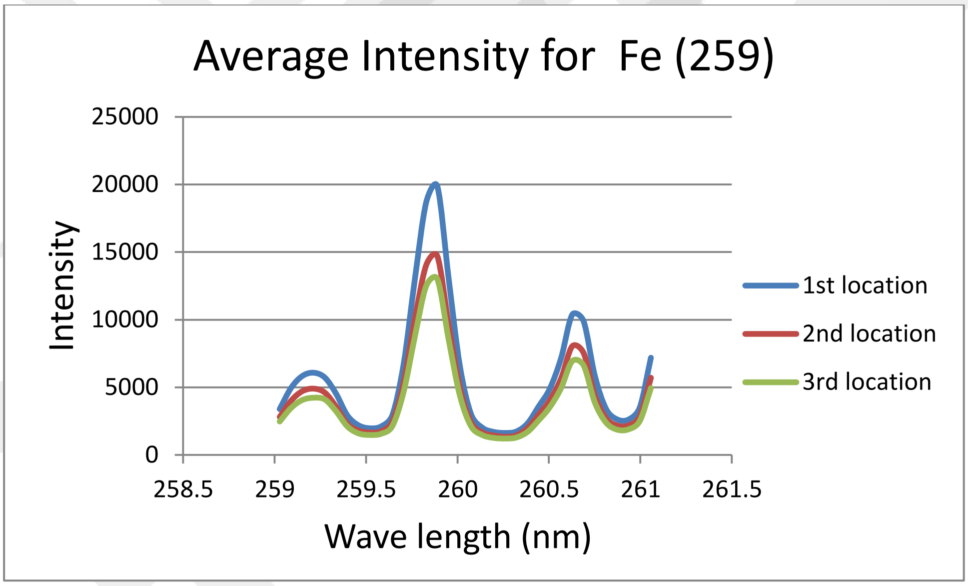

fig (4.89). Average Intensity for Fe (259)

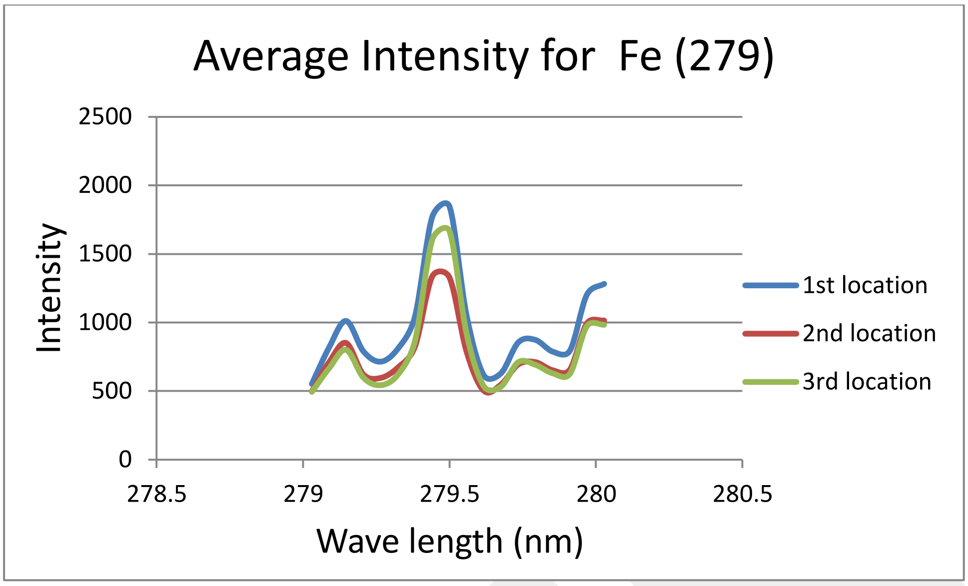

fig (4.90). Average Intensity for Fe (279)

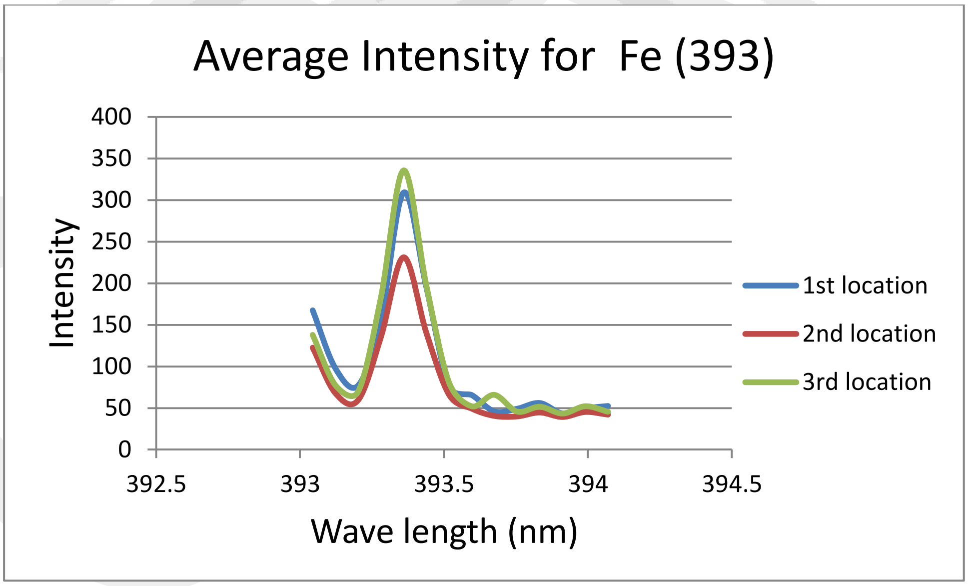

fig (4.91). Average Intensity for Fe (393)

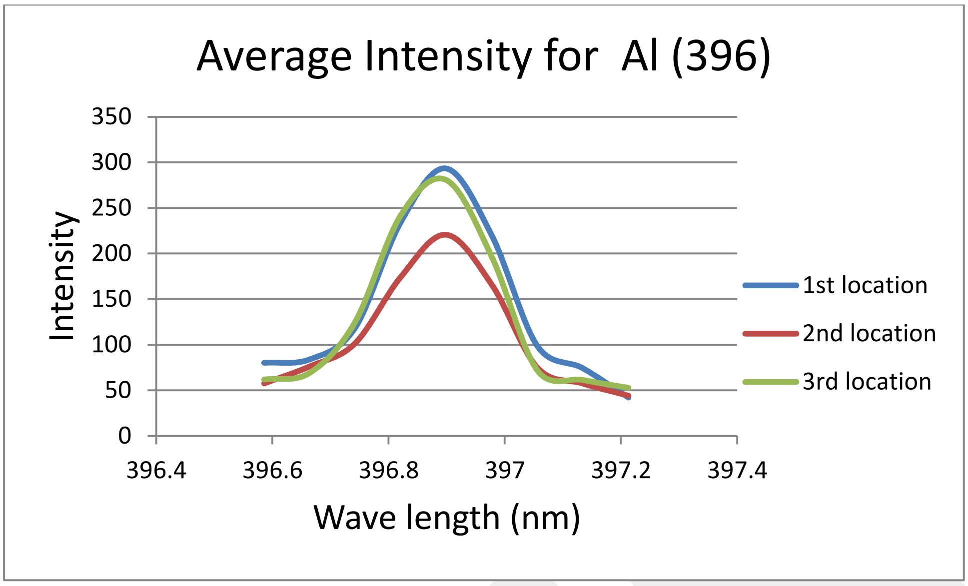

fig (4.92). Average Intensity for Al (396)

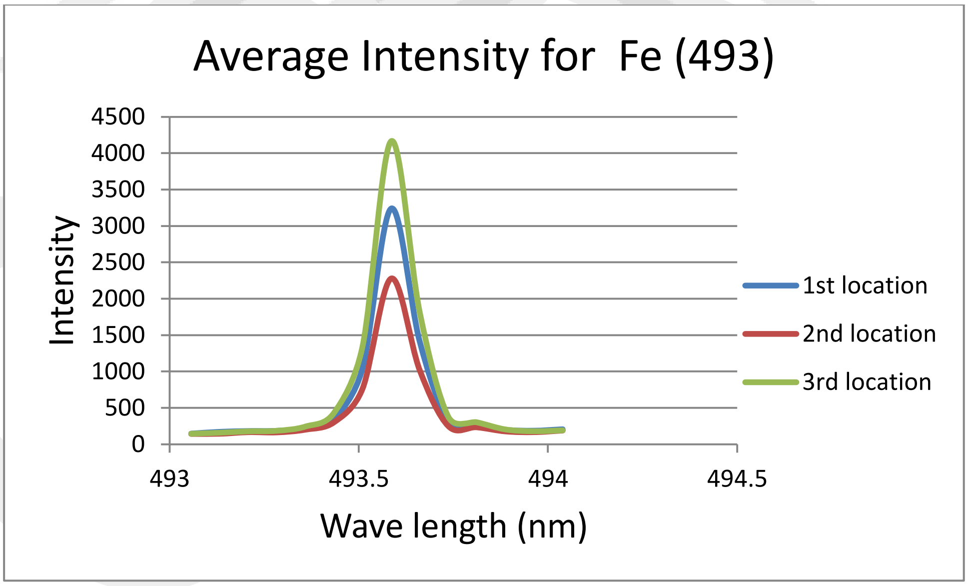

fig (4.93). Average Intensity for Fe (493)

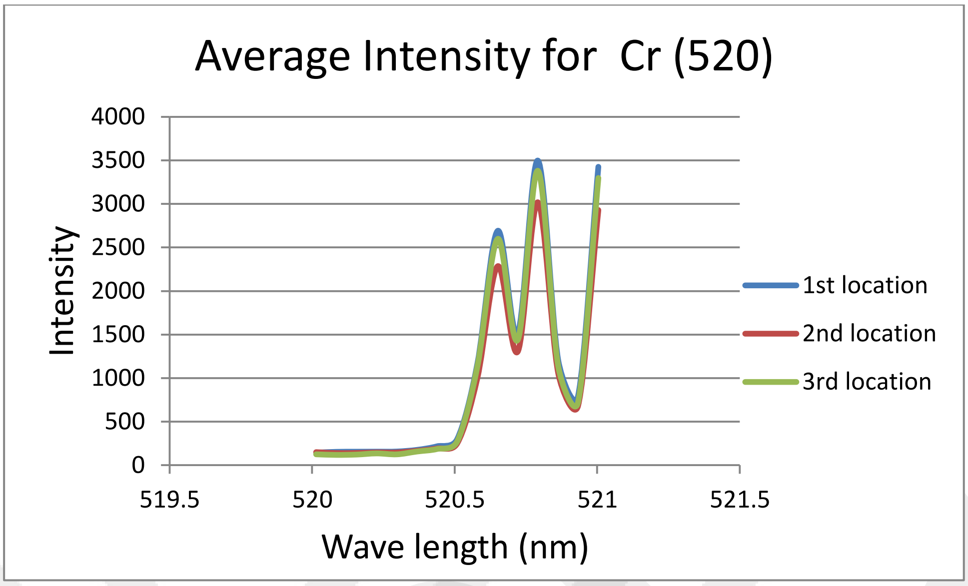

fig (4.94). Average Intensity for Cr (520)

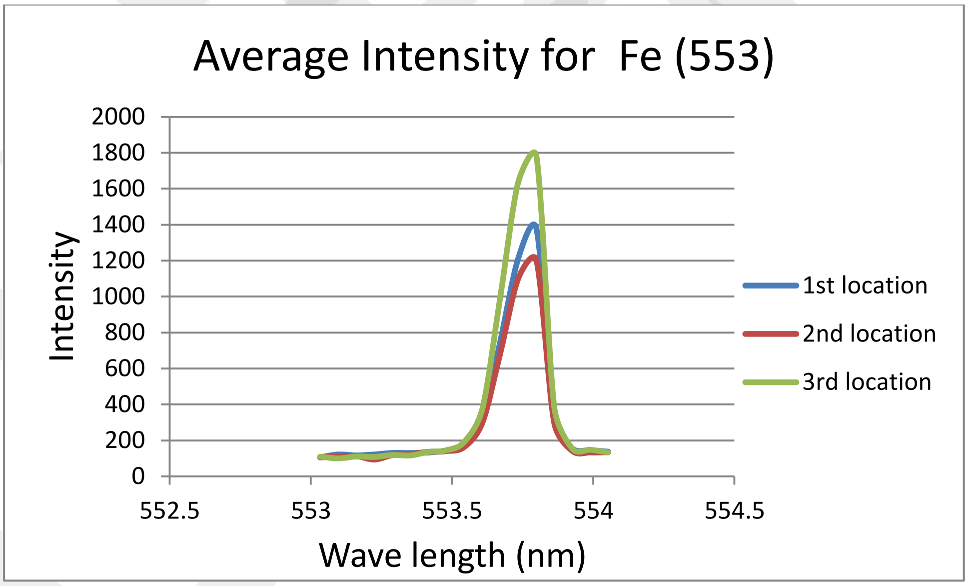

fig (4.95). Average Intensity for Fe (553)

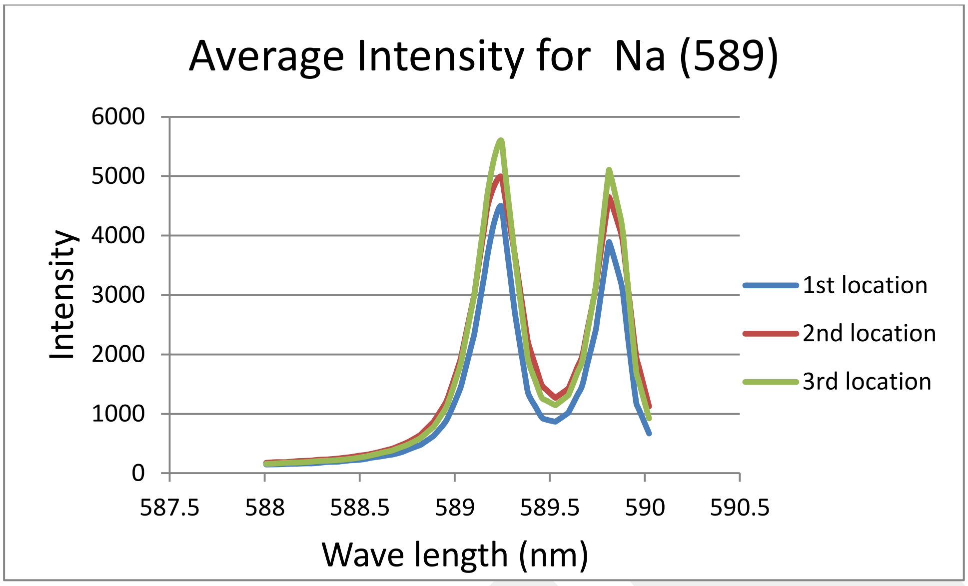

fig (4.96). Average Intensity for Na (589)

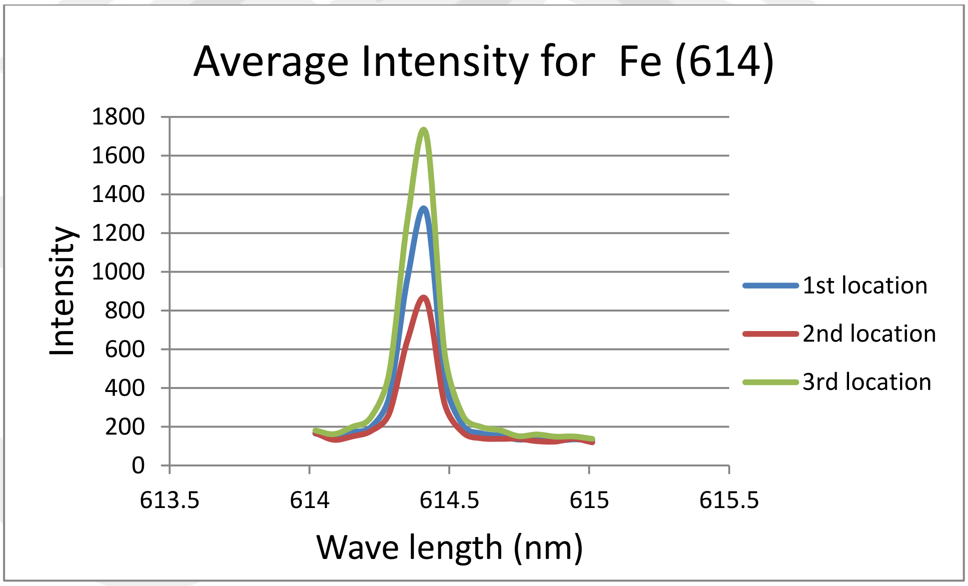

fig (4.97). Average Intensity for Fe (614)

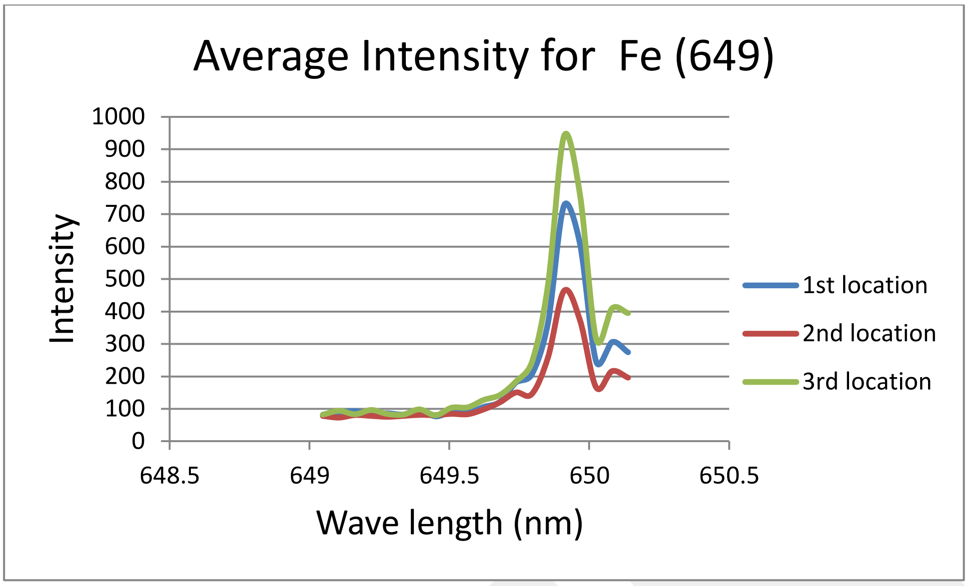

fig (4.98). Average Intensity for Fe (649)

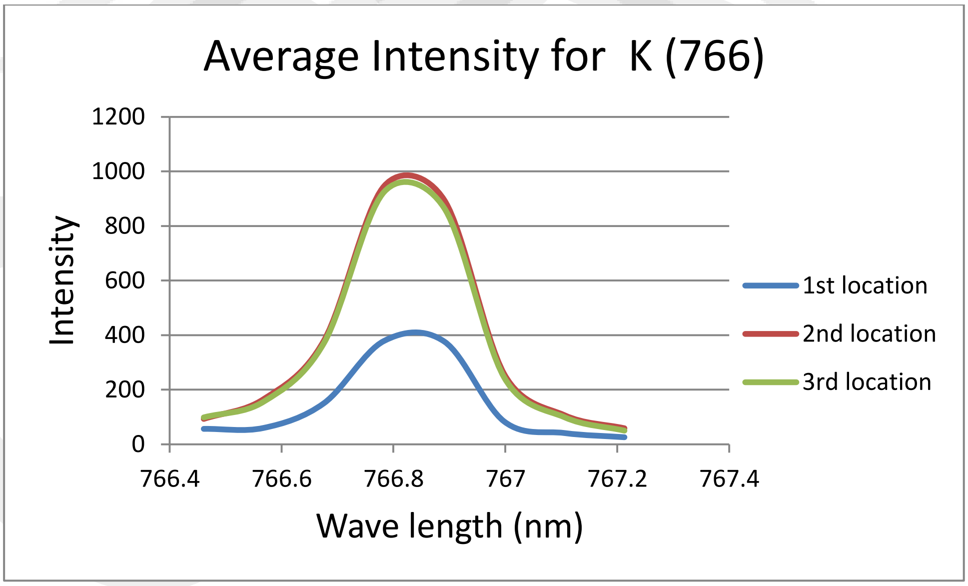

fig (4.99). Average Intensity for K (766)

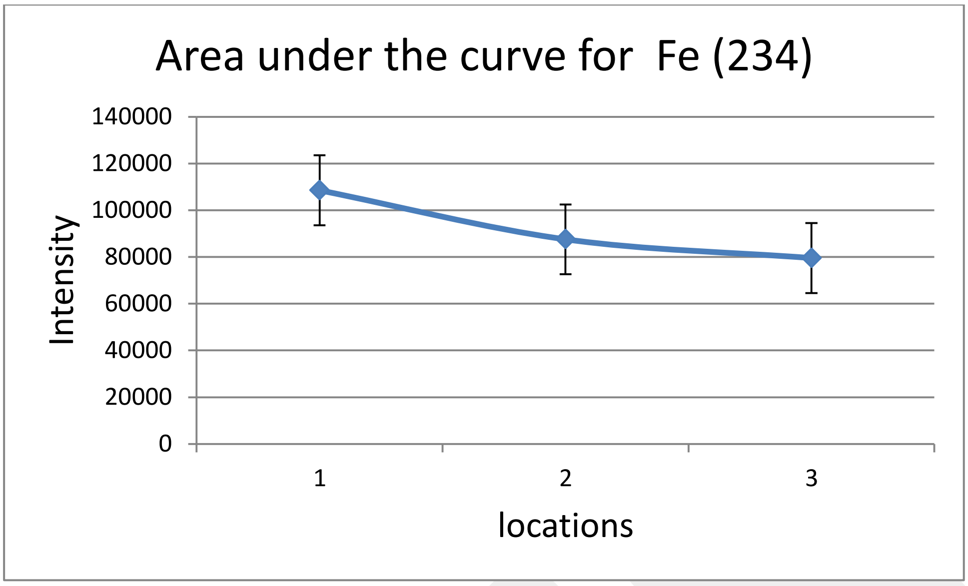

fig (4.100). Area under the curve for Fe (234)

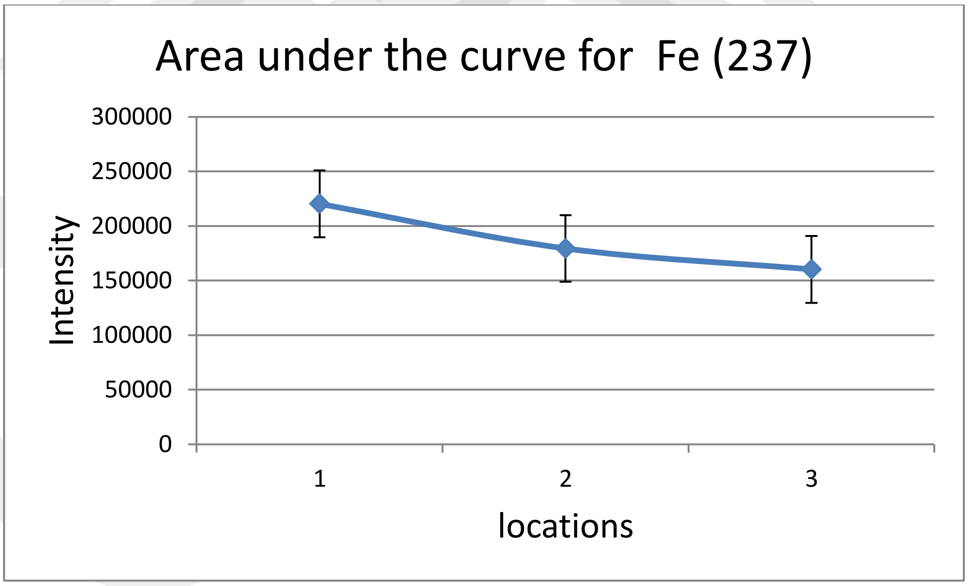

fig (4.101). Area under the curve for Fe (237)

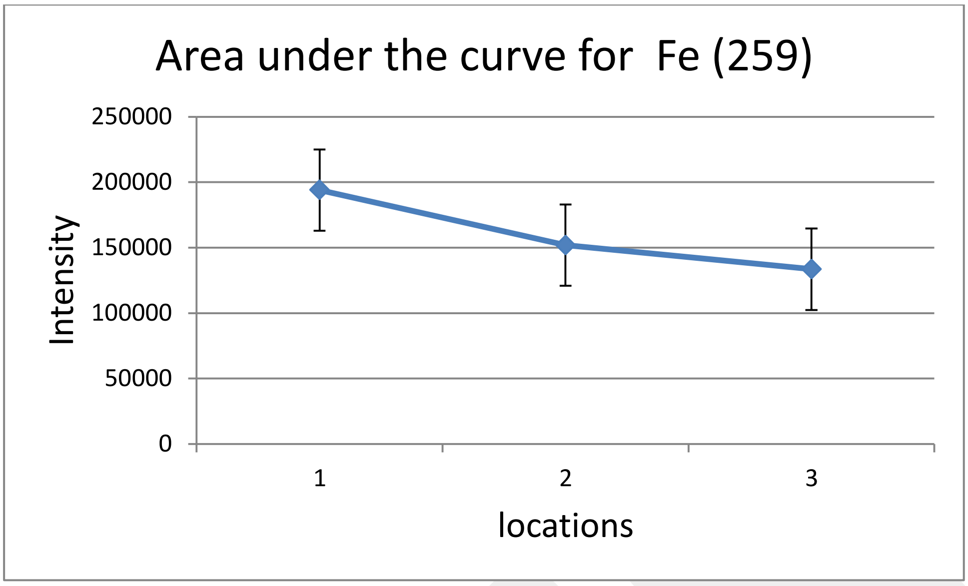

fig (4.102). Area under the curve for Fe (259)

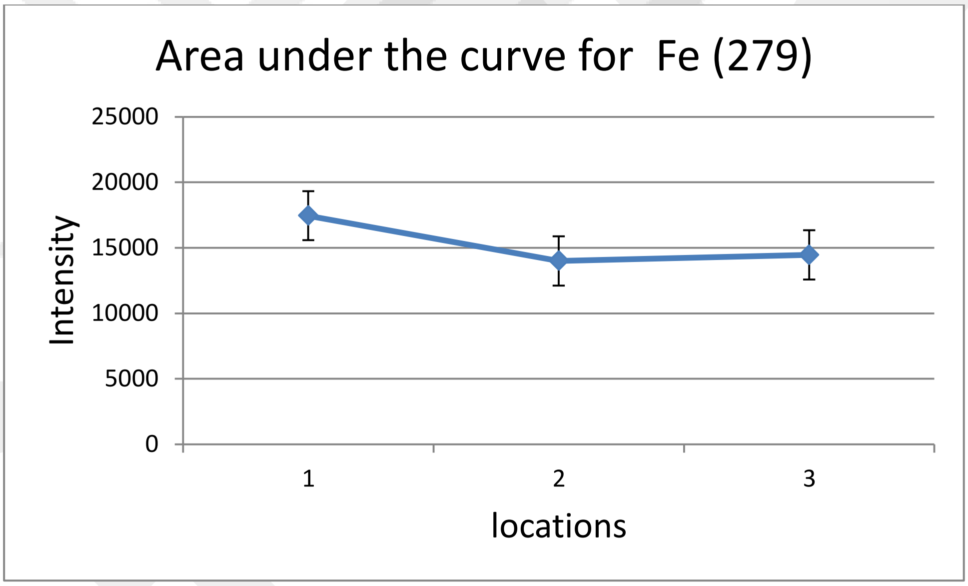

fig (4.103). Area under the curve for Fe (279)

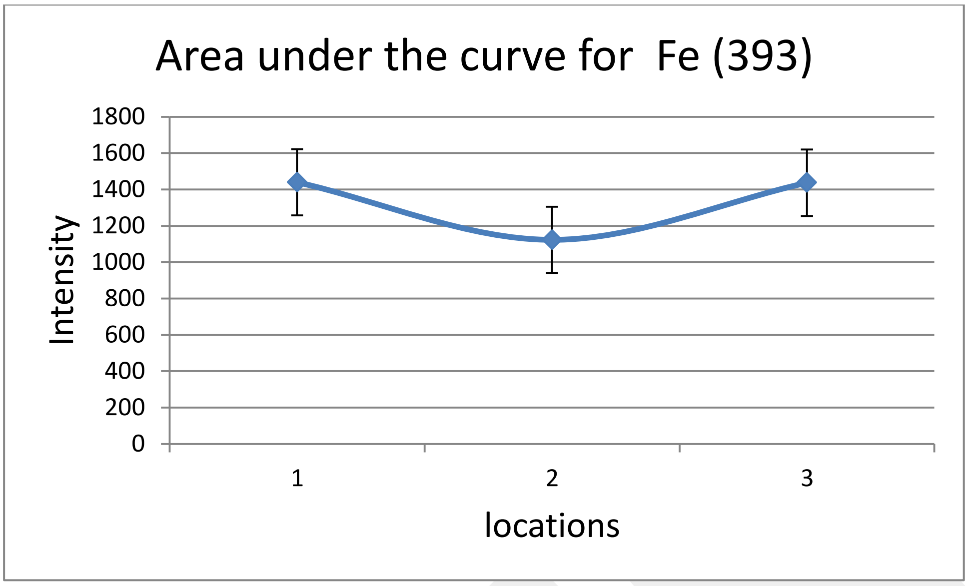

fig (4.104). Area under the curve for Fe (393)

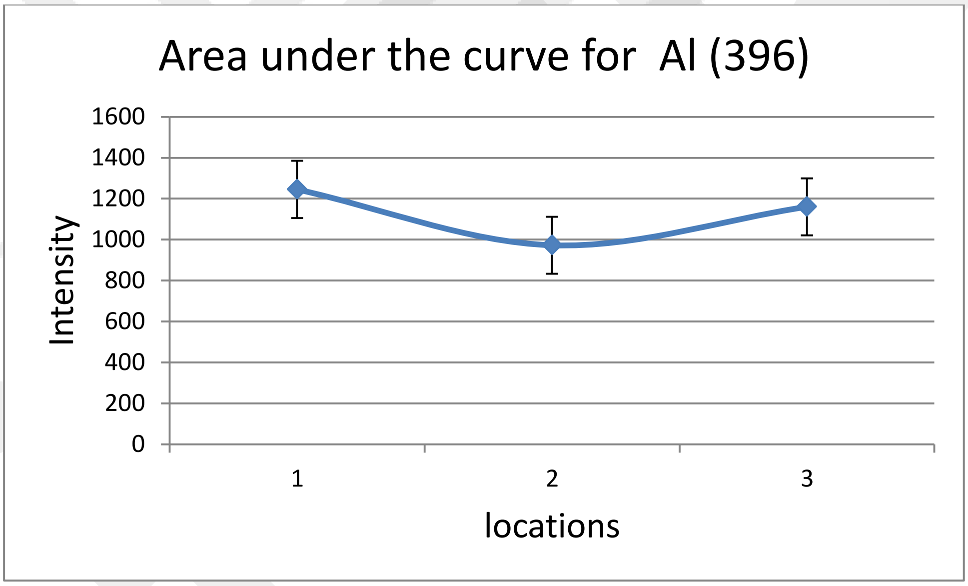

fig (4.105). Area under the curve for Al (396)

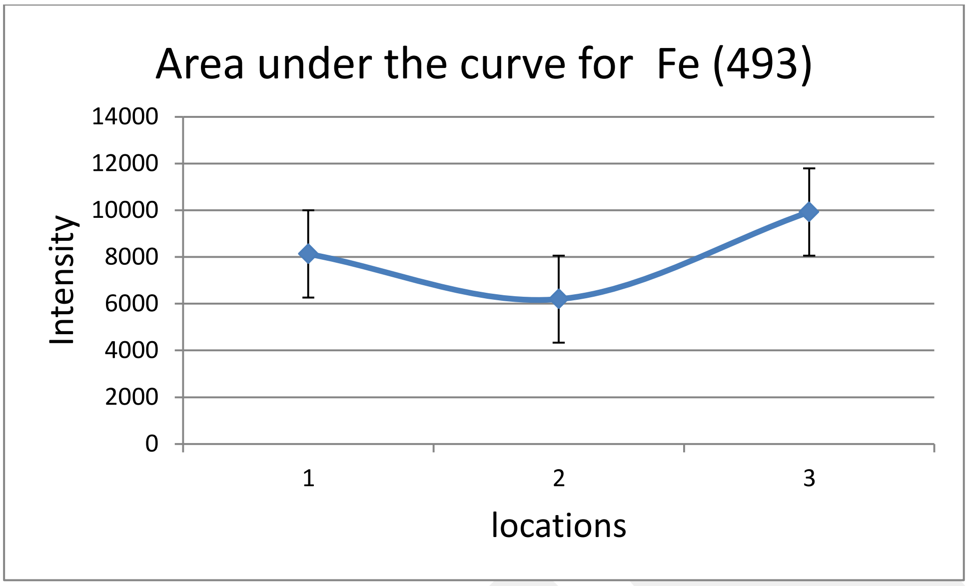

fig (4.106). Area under the curve for Fe (493)

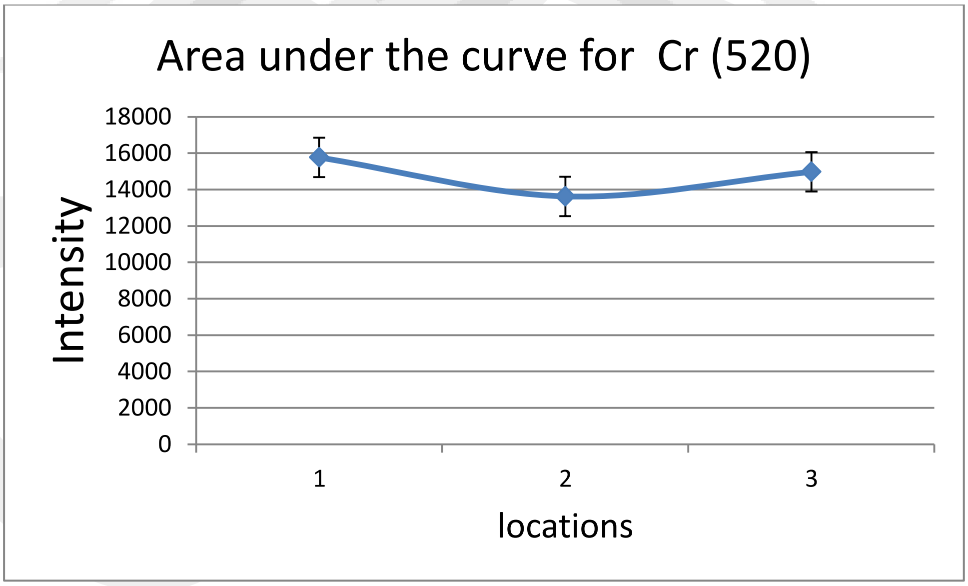

fig (4.107). Area under the curve for Cr (520)

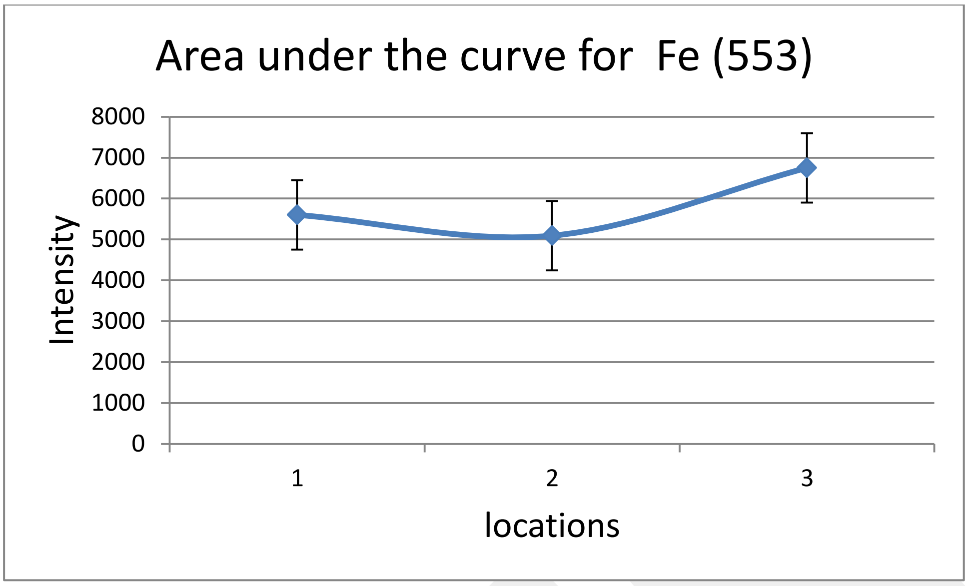

fig (4.108). Area under the curve for Fe (553)

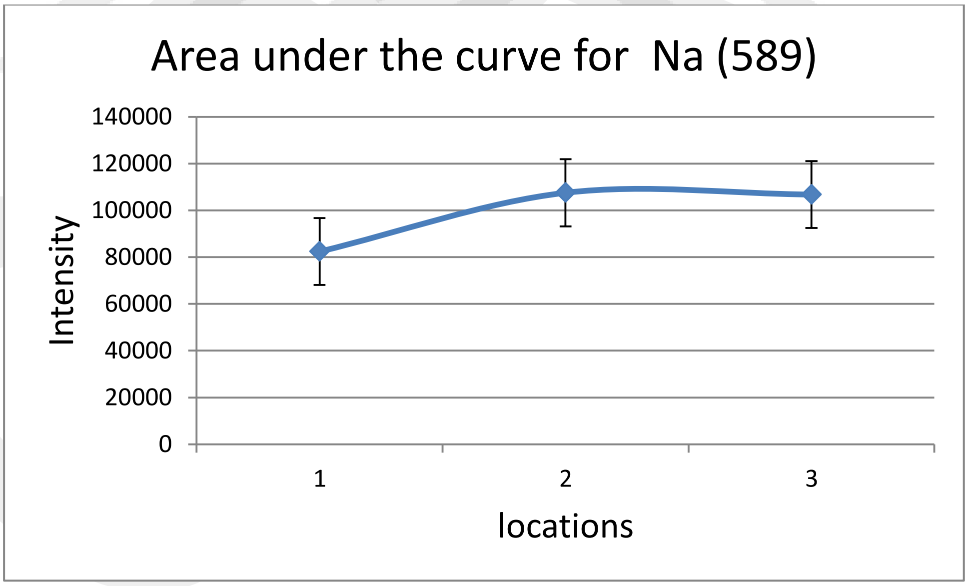

fig (4.109). Area under the curve for Na (589)

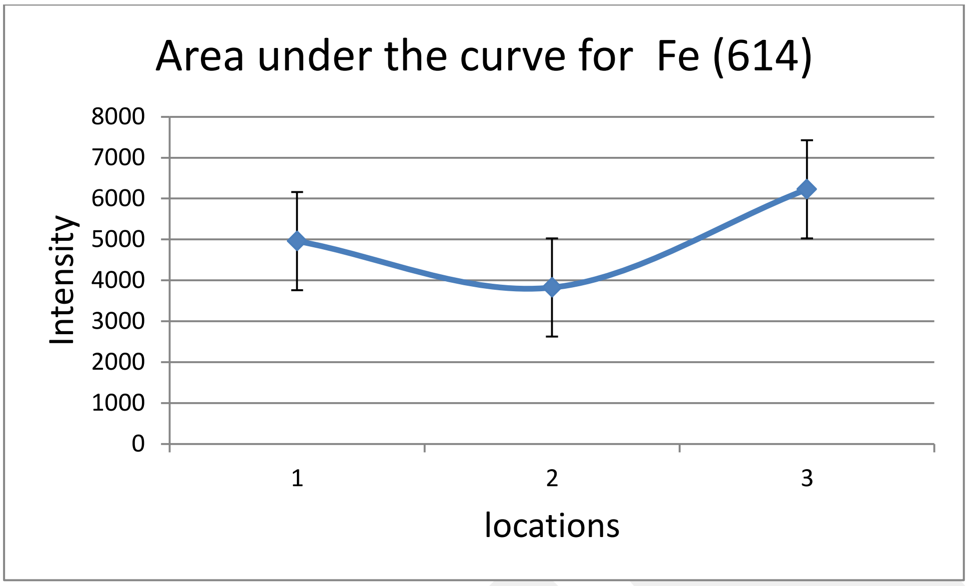

fig (4.110). Area under the curve for Fe (614)

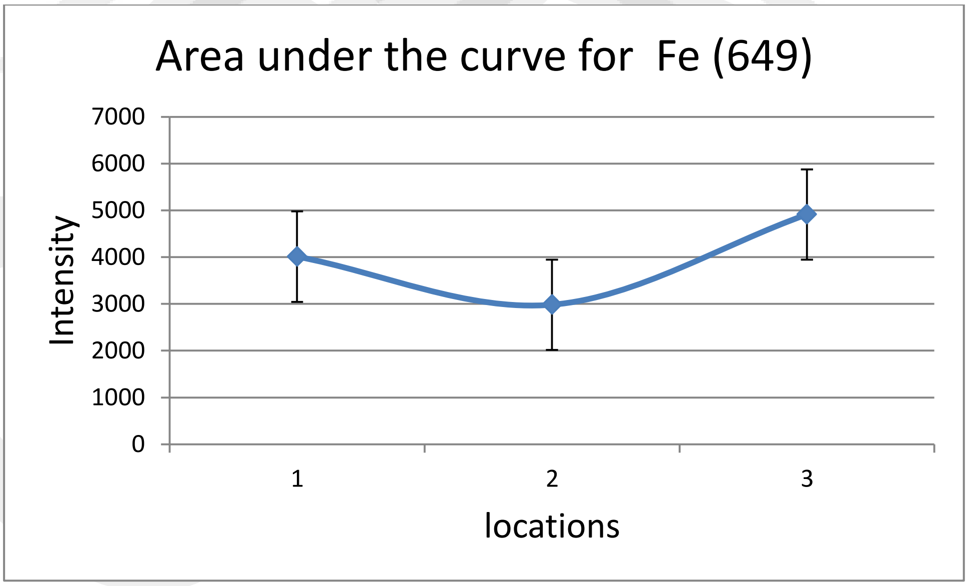

fig (4.111). Area under the curve for Fe (649)

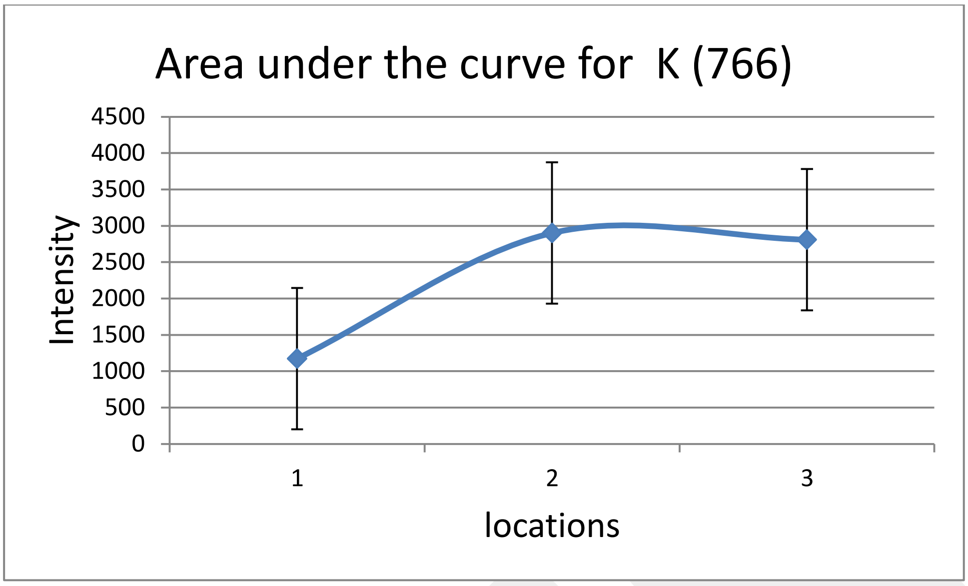

fig (4.112). Area under the curve for K (766)

After that we used 0,3 µs time delay, and power of 30 mJ, with frequency 10 hz, and 50 shots, then we have moved the alloy in three locations, and we got the following results as shown in the following figures.

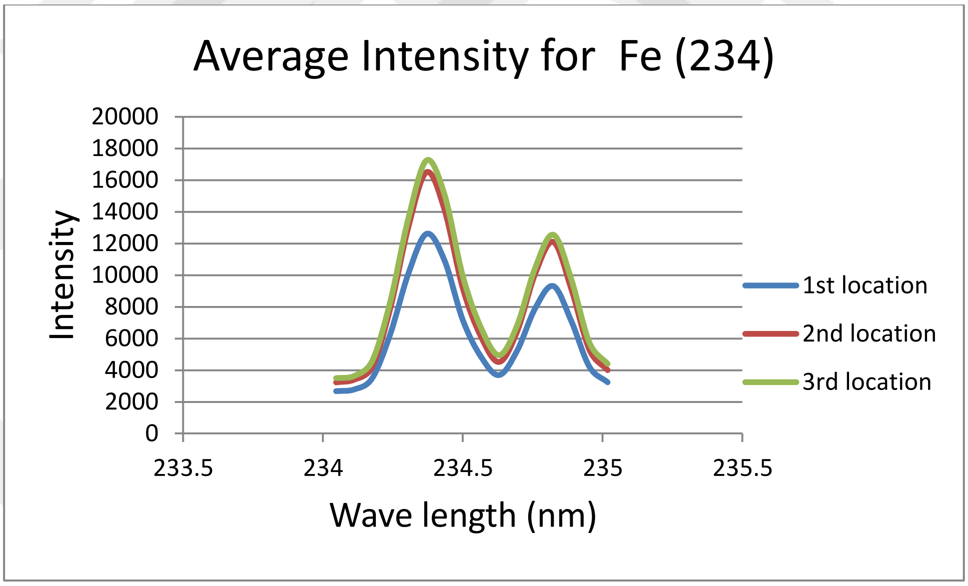

fig (4.113). Average Intensity for Fe (234)

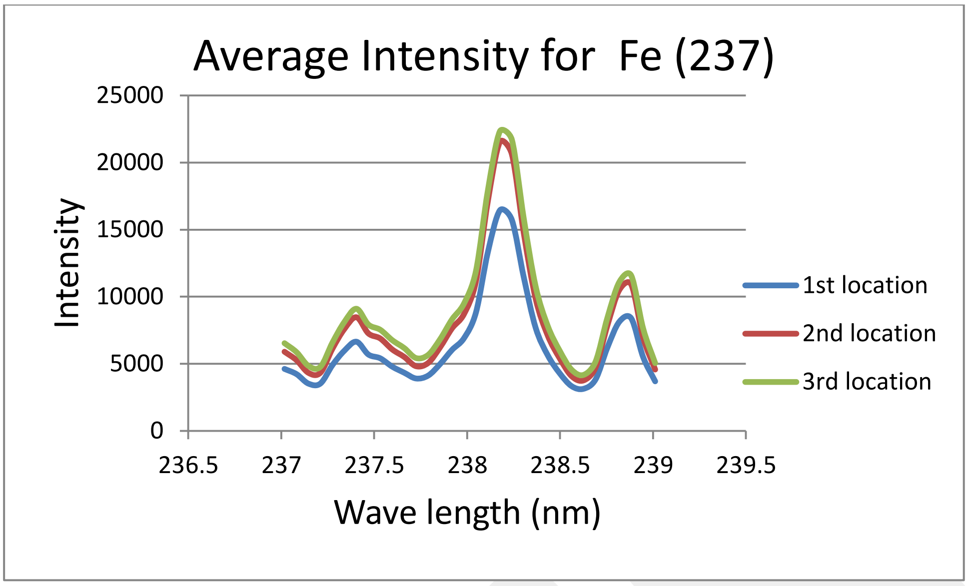

fig (4.114). Average Intensity for Fe (237)

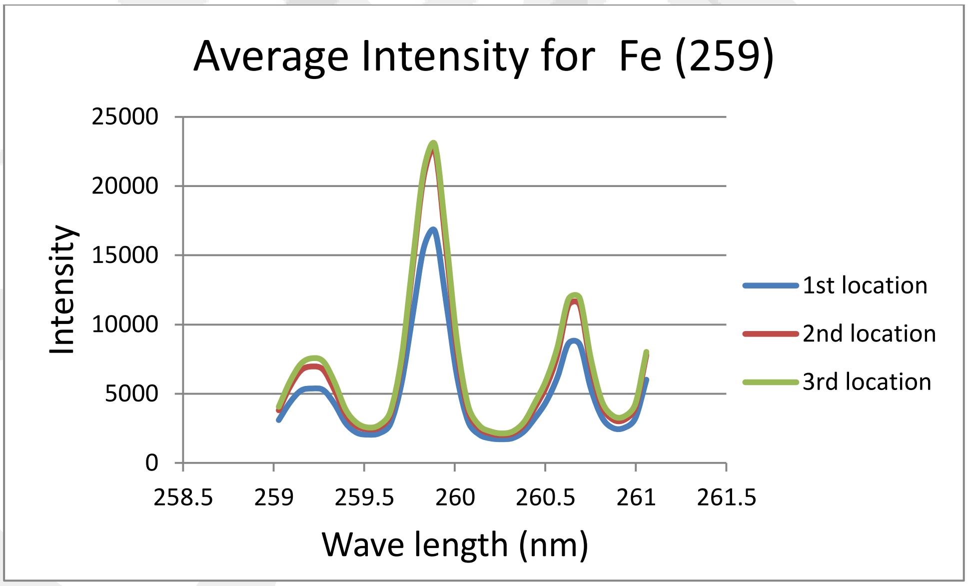

fig (4.115). Average Intensity for Fe (259)

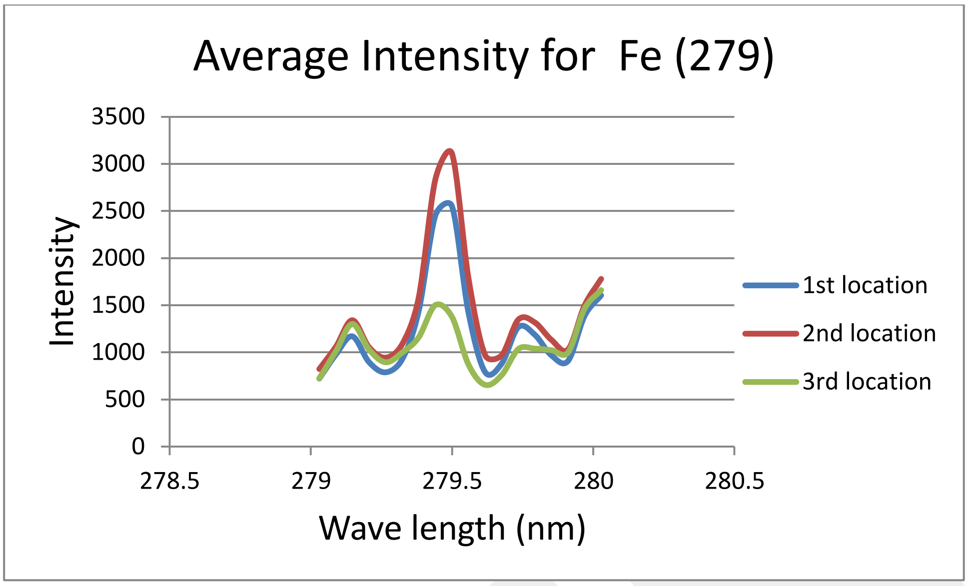

fig (4.116). Average Intensity for Fe (279)

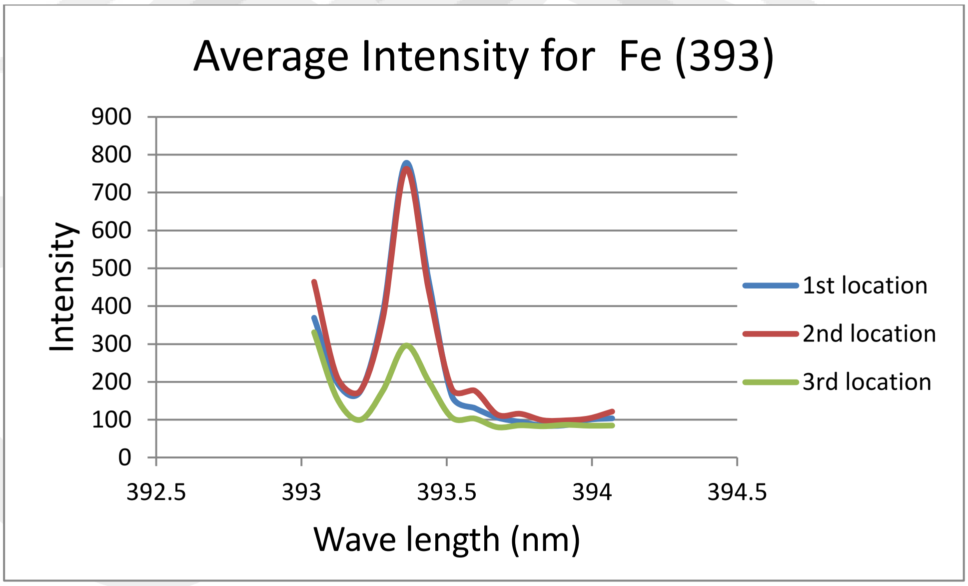

fig (4.117). Average Intensity for Fe (393)

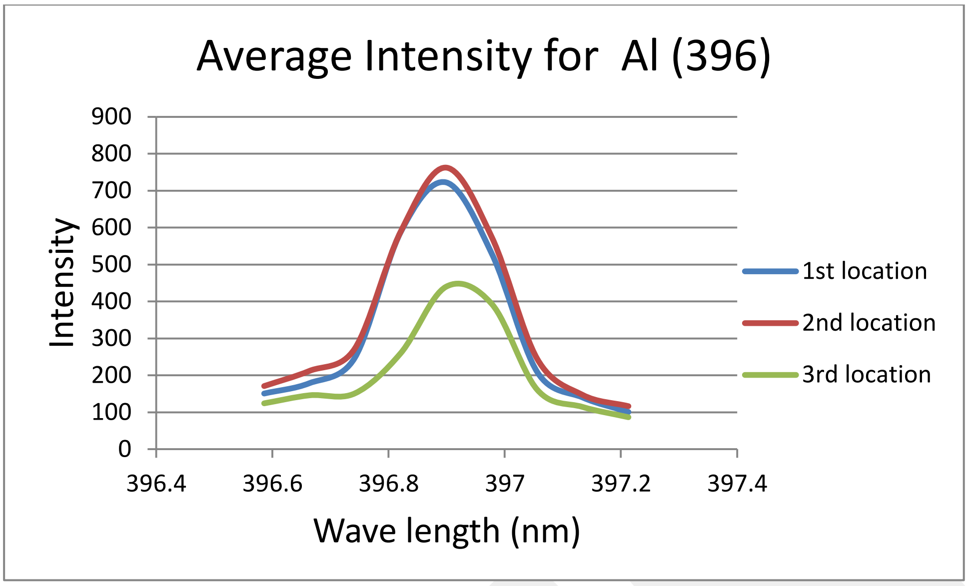

fig (4.118). Average Intensity for Al (396)

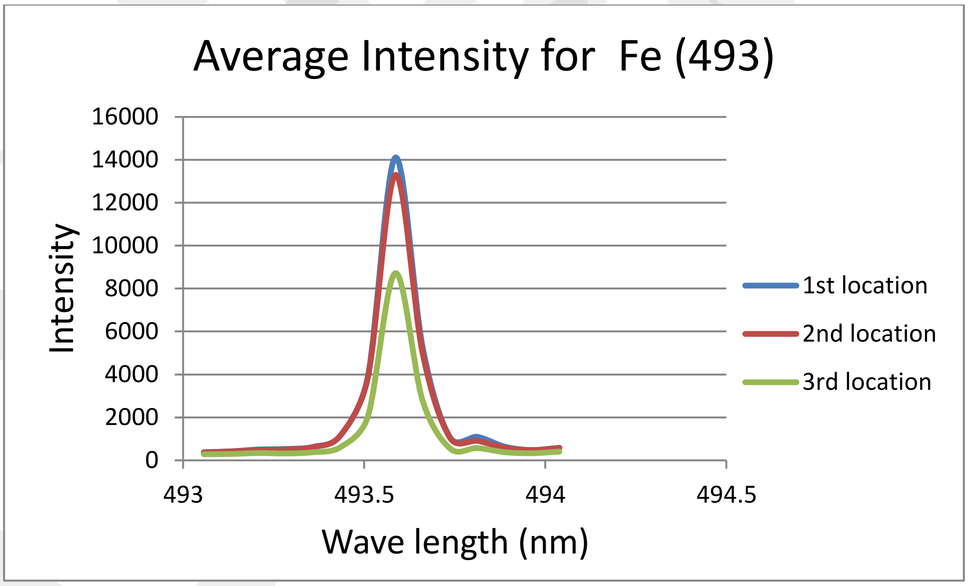

fig (4.119). Average Intensity for Al (396)

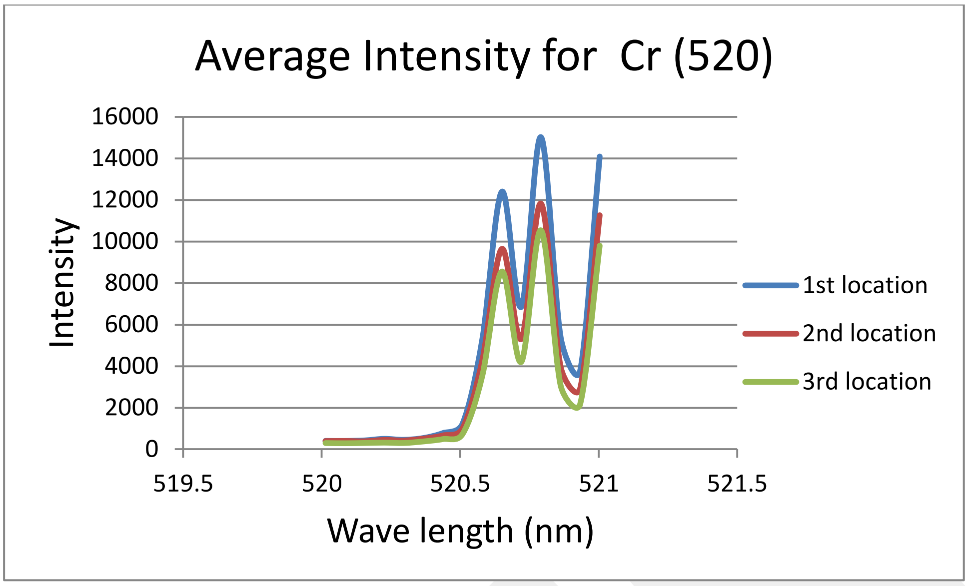

fig (4.120). Average Intensity for Cr (520)

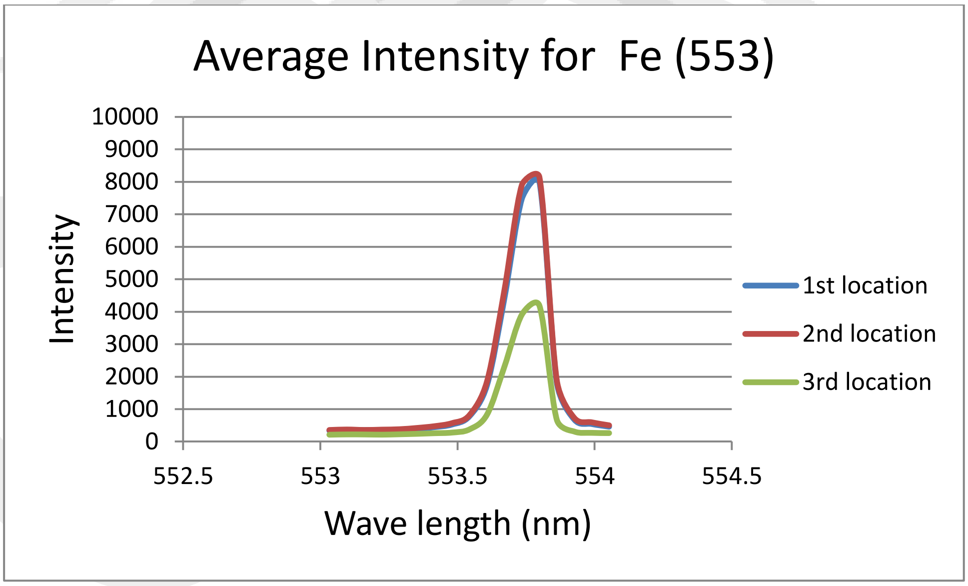

fig (4.121). Average Intensity for Fe (553)

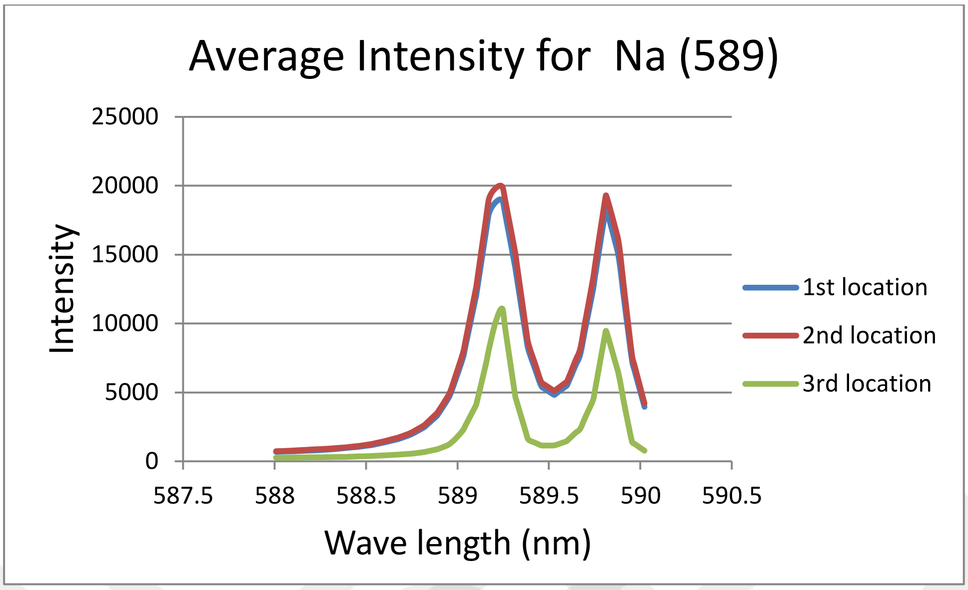

fig (4.122). Average Intensity for Na (589)

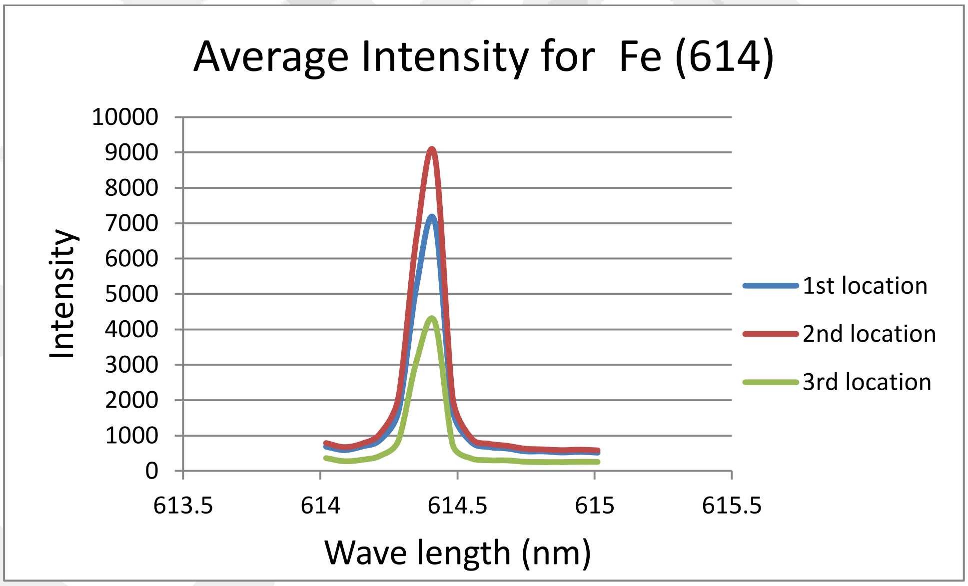

fig (4.123). Average Intensity for Fe (614)

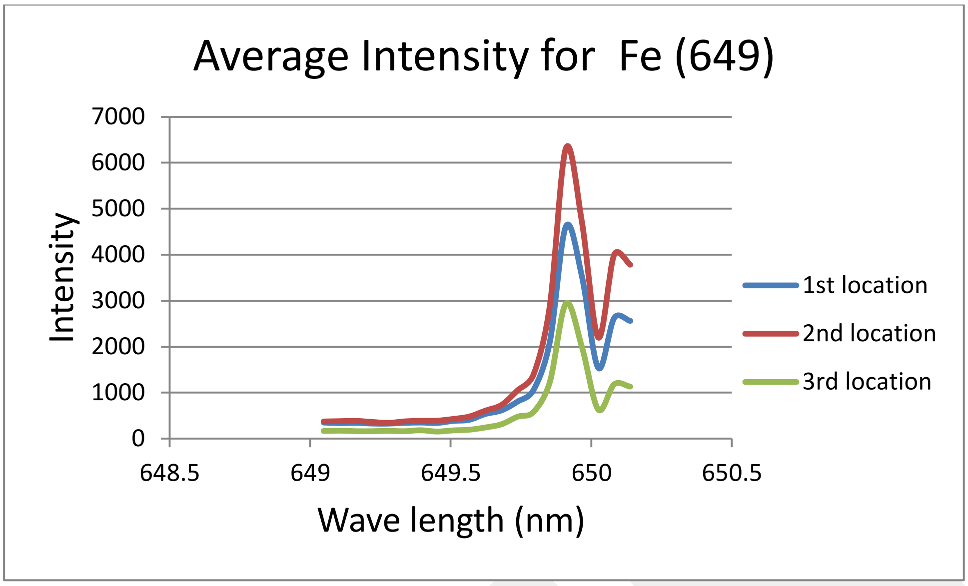

fig (4.124). Average Intensity for Fe (649)

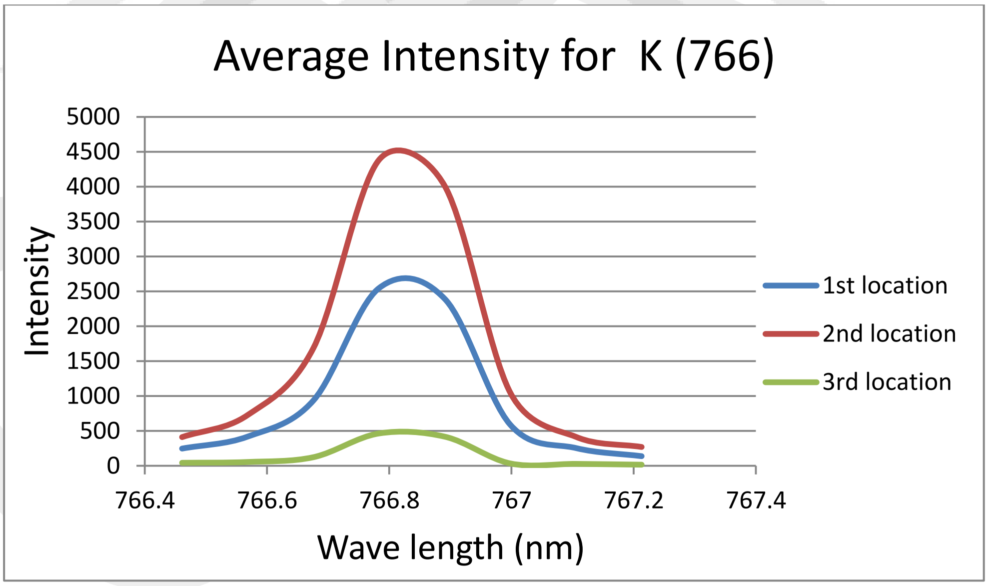

fig (4.125). Average Intensity for K (766)

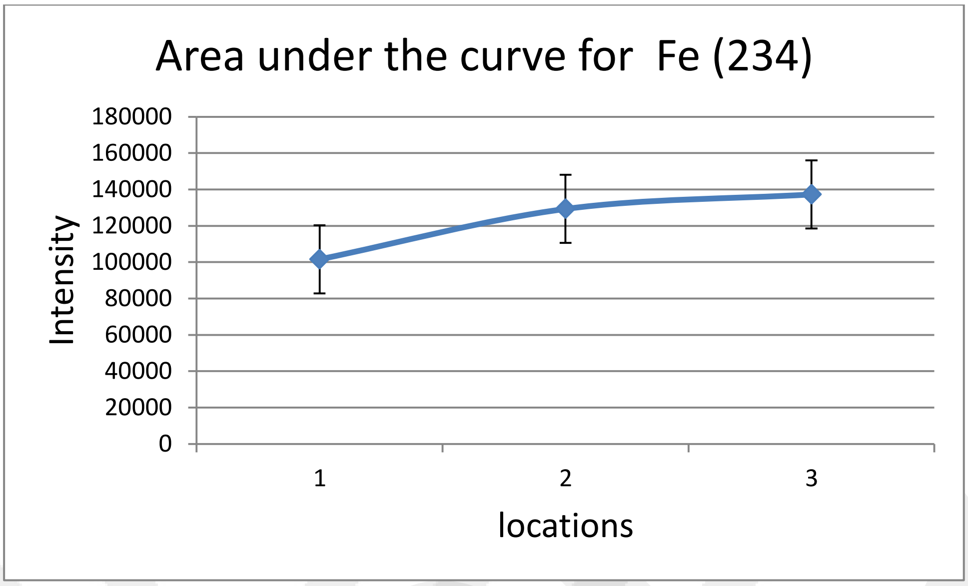

fig (4.126). Area under the curve for Fe (234)

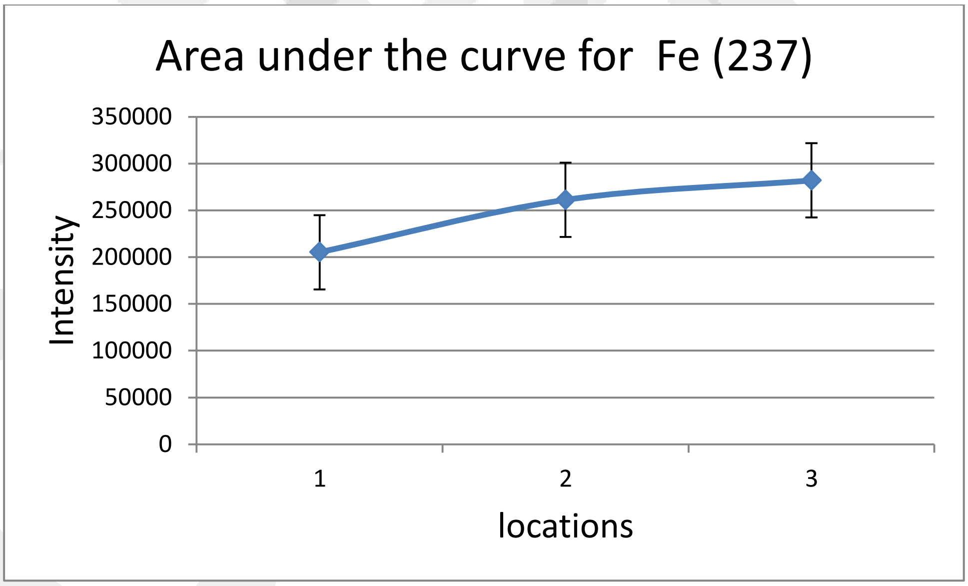

fig (4.127). Area under the curve for Fe (237)

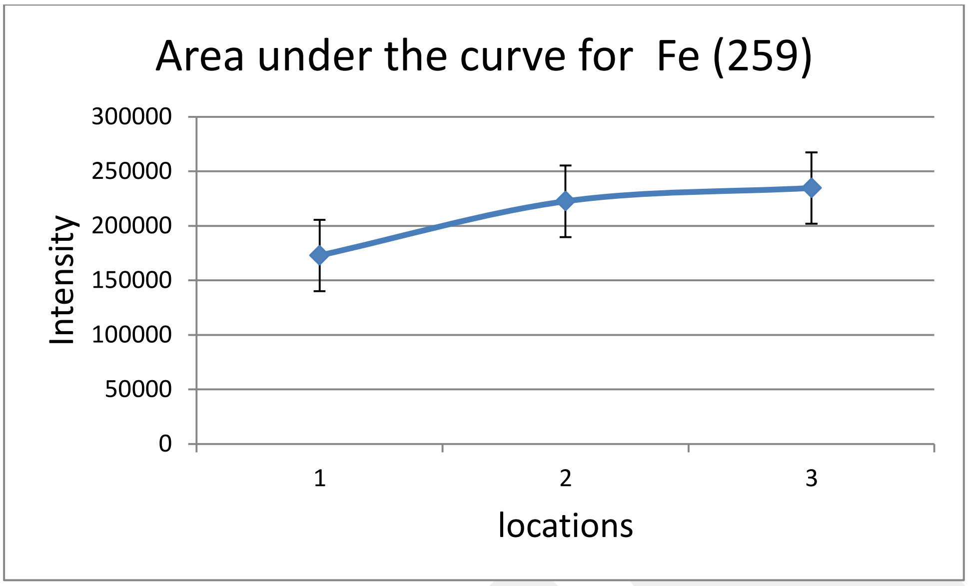

fig (4.128). Area under the curve for Fe (259)

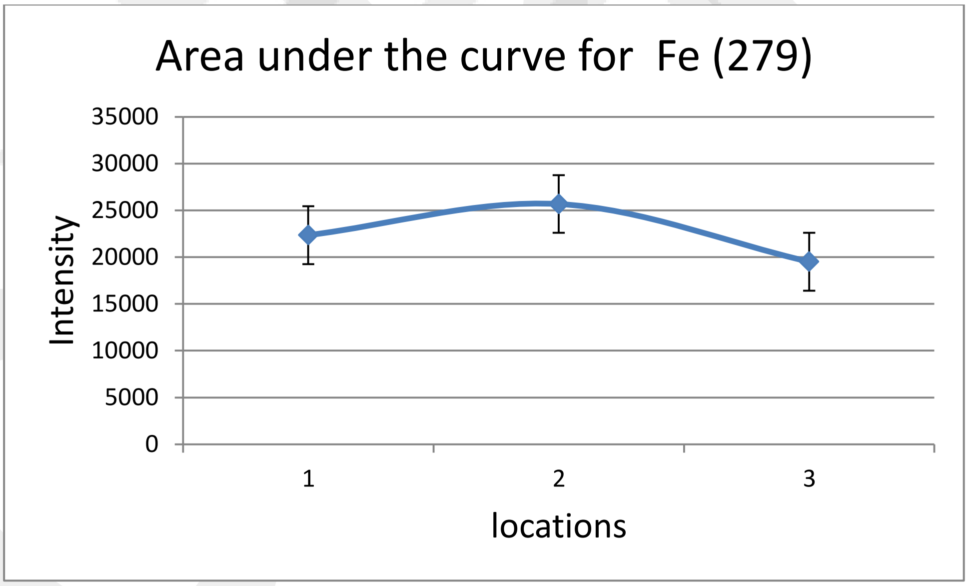

fig (4.129). Area under the curve for Fe (279)

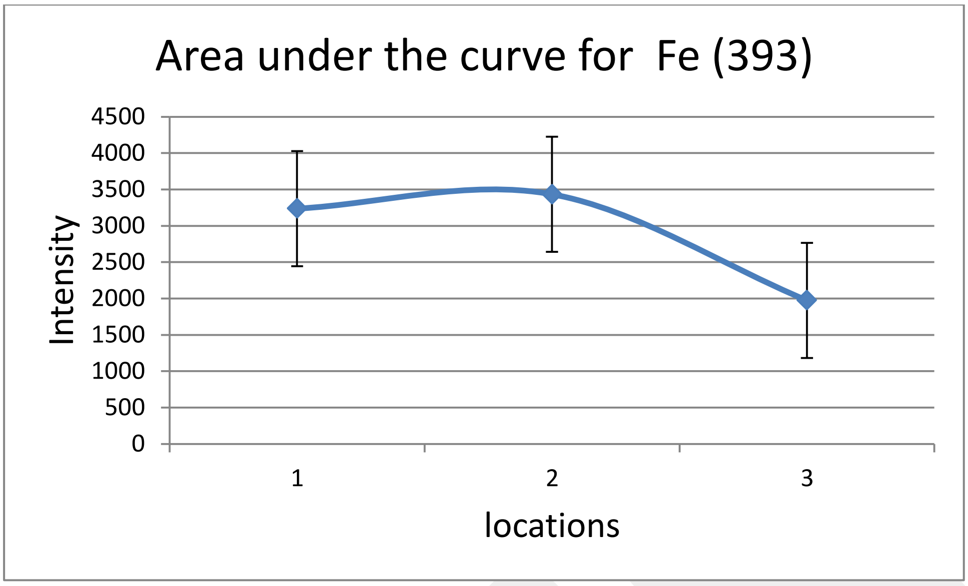

fig (4.130). Area under the curve for Fe (393)

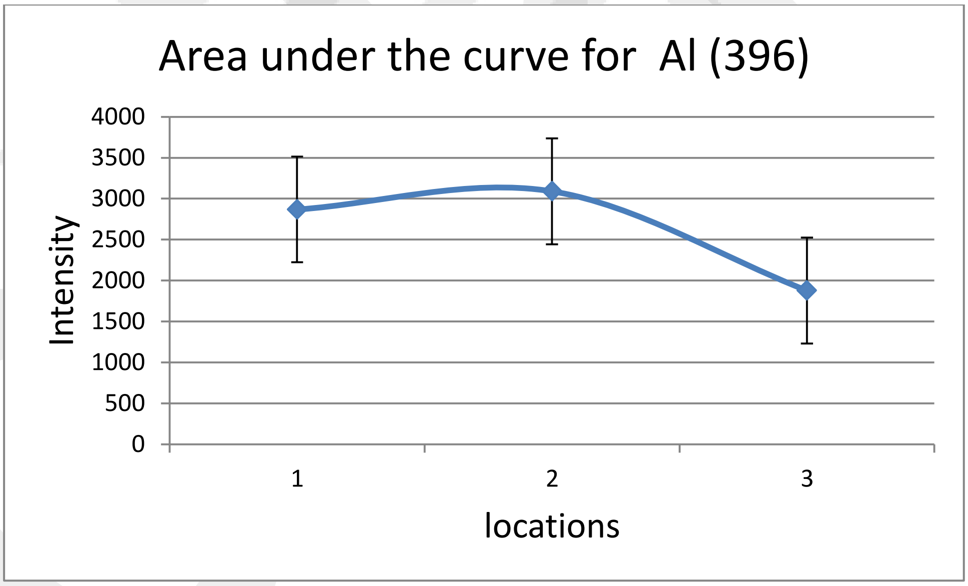

fig (4.131). Area under the curve for Al (396)

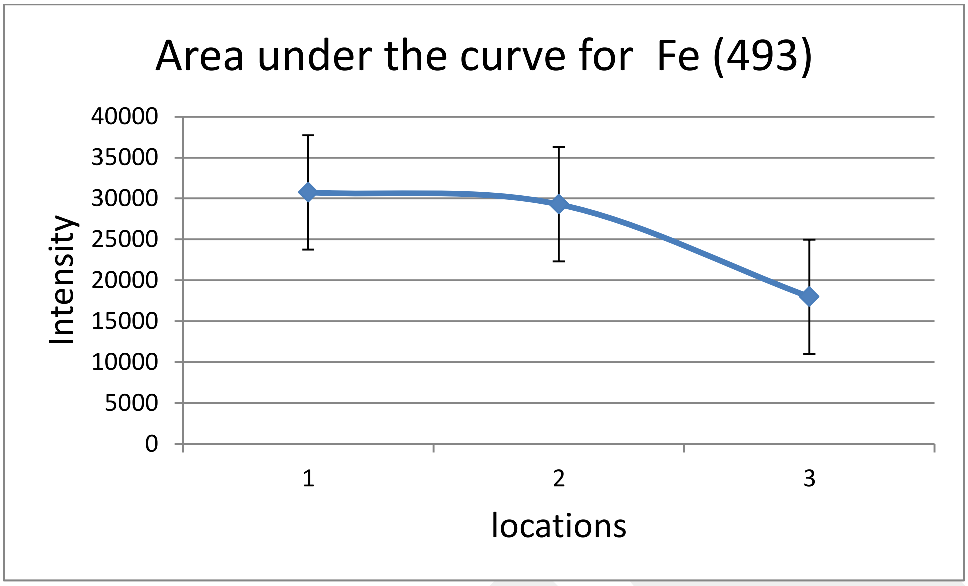

fig (4.132). Area under the curve for Fe (493)

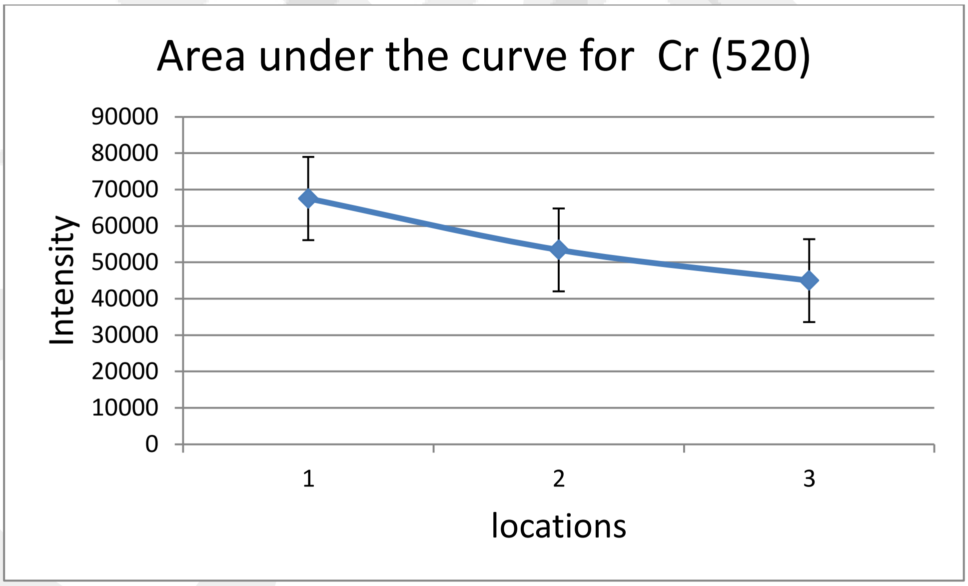

fig (4.133). Area under the curve for Cr (520)

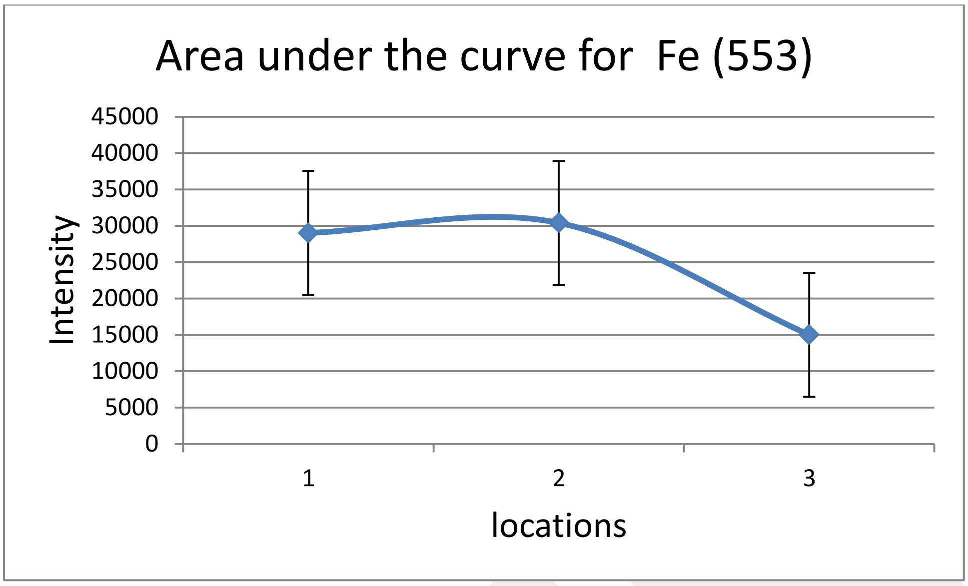

fig (4.134). Area under the curve for Fe (553)

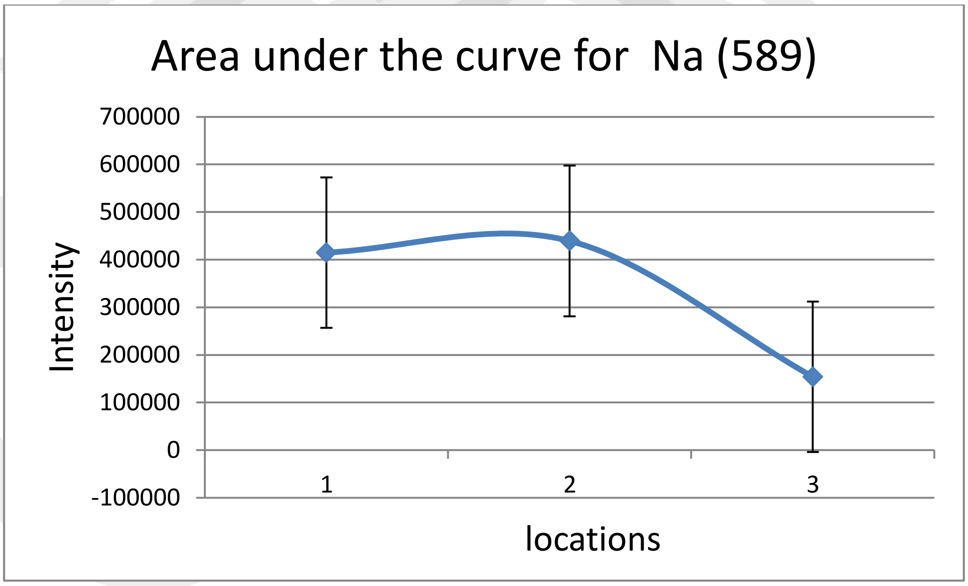

fig (4.135). Area under the curve for Na (589)

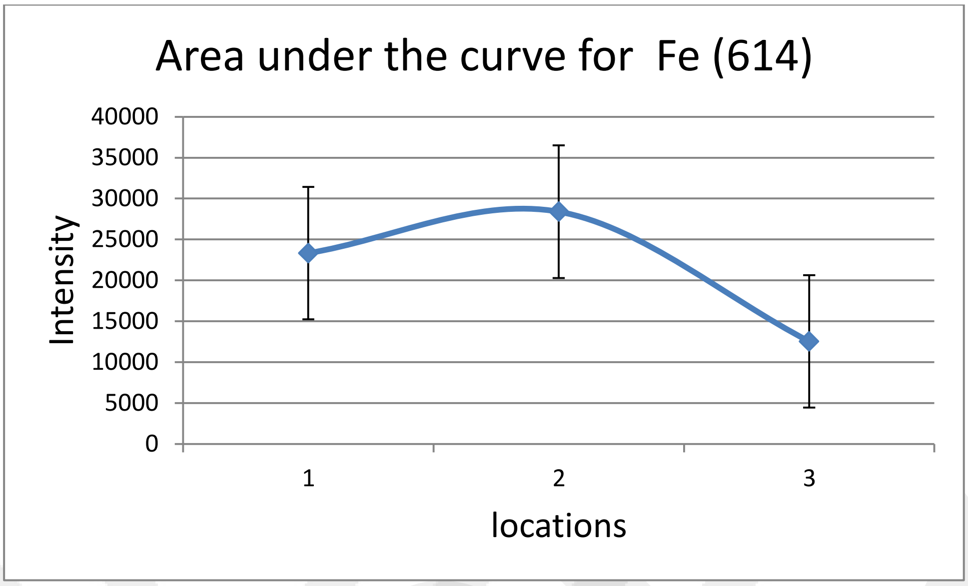

fig (4.136). Area under the curve for Fe (614)

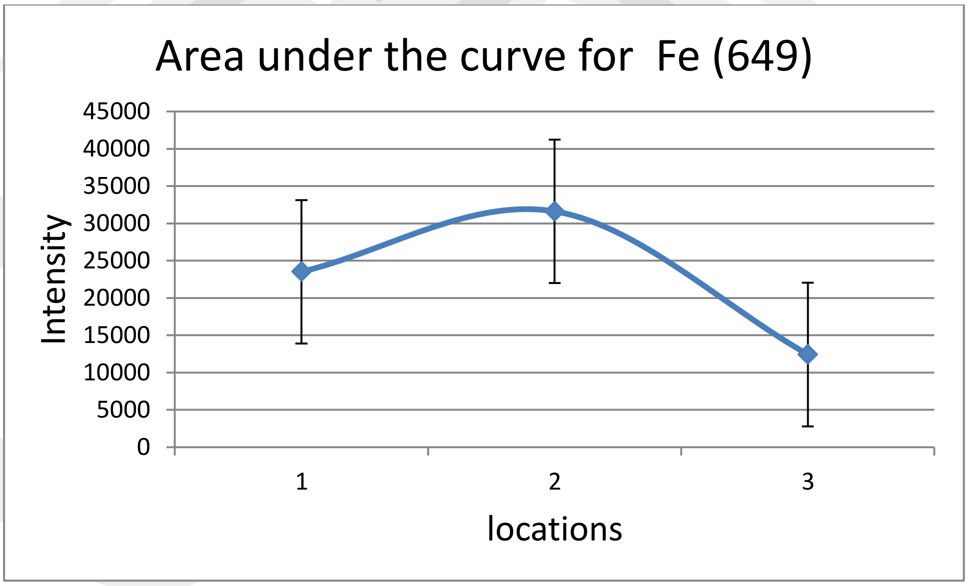

fig (4.137). Area under the curve for Fe (649)

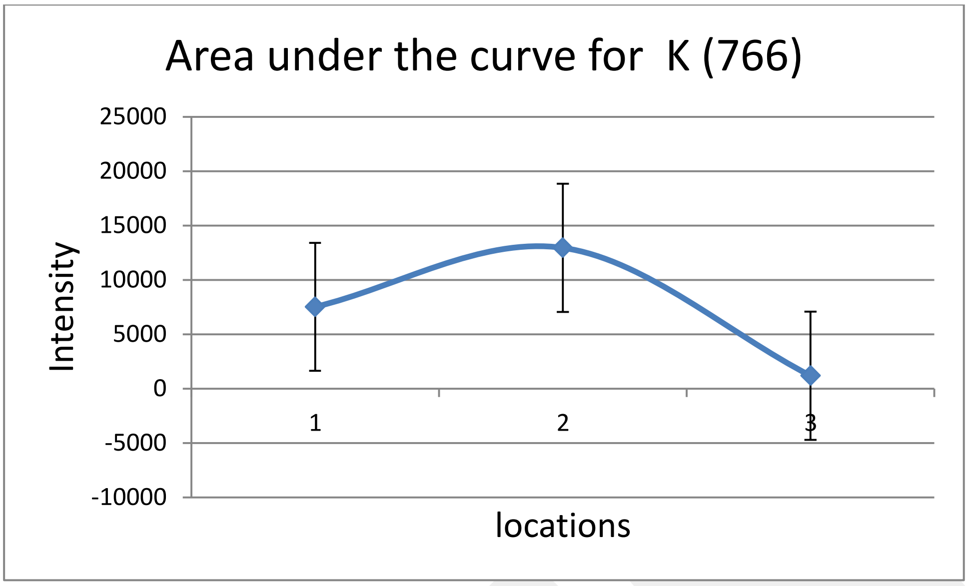

fig (4.138). Area under the curve for K (766)

After that we used 0,3 µs time delay, and power of 40 mJ, with frequency 10 hz, and 50 shots, then we have moved the alloy in three locations, and we got the following results as shown in the following figures.

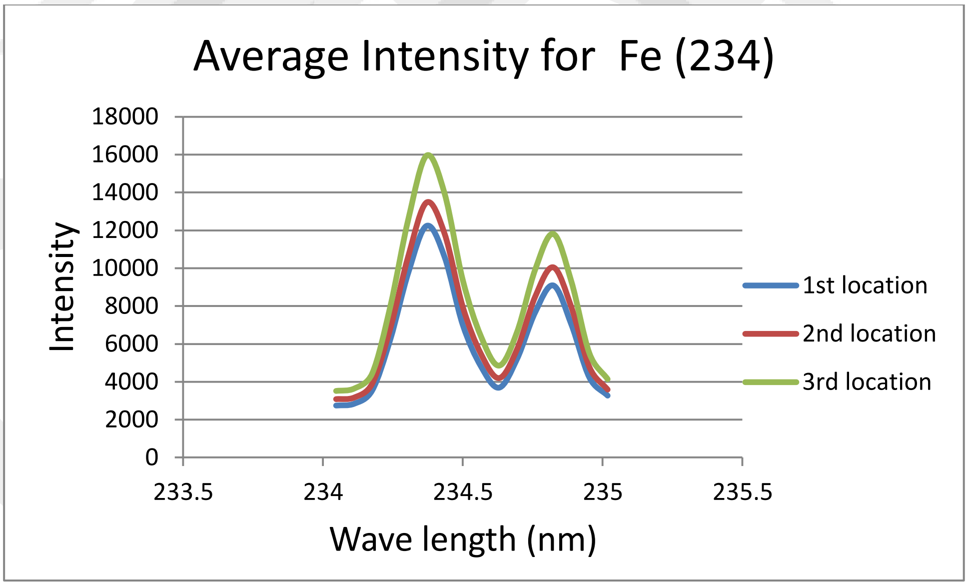

fig (4.139). Average Intensity for Fe (234)

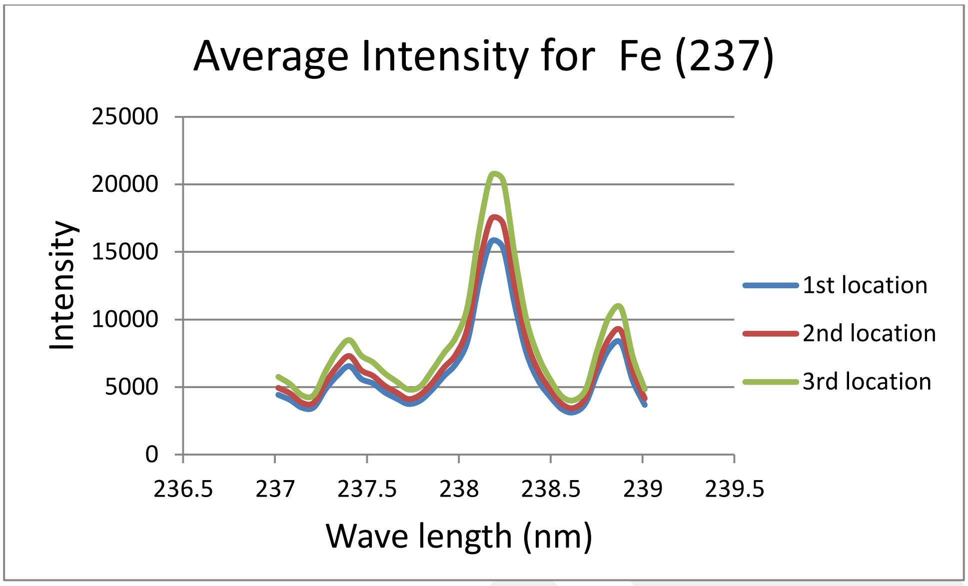

fig (4.140). Average Intensity for Fe (237)

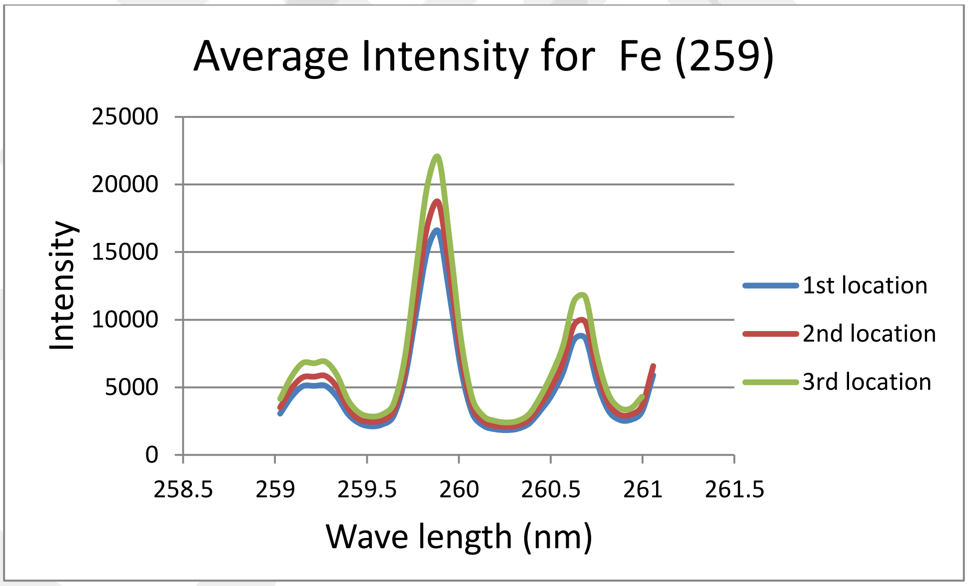

fig (4.141). Average Intensity for Fe (259)

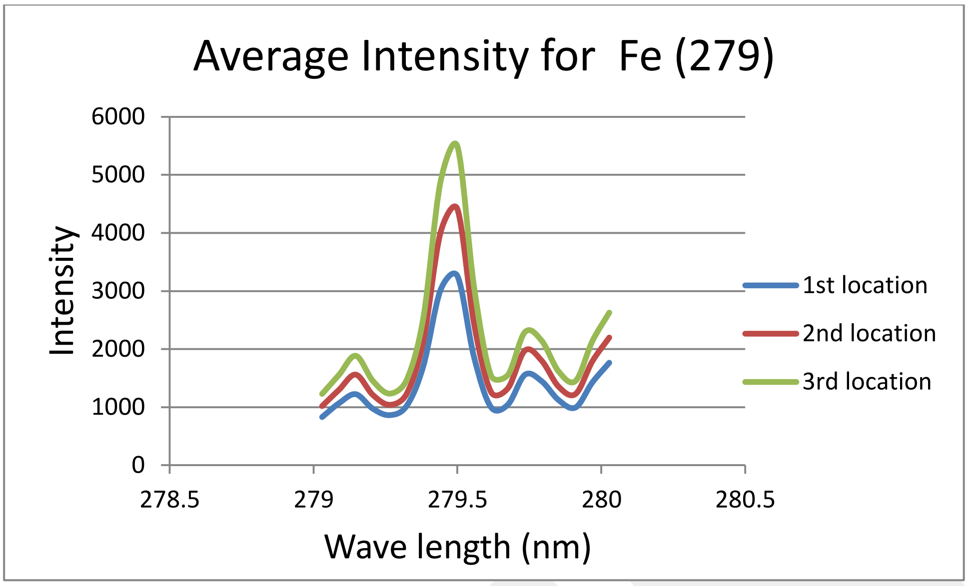

fig (4.142). Average Intensity for Fe (279)

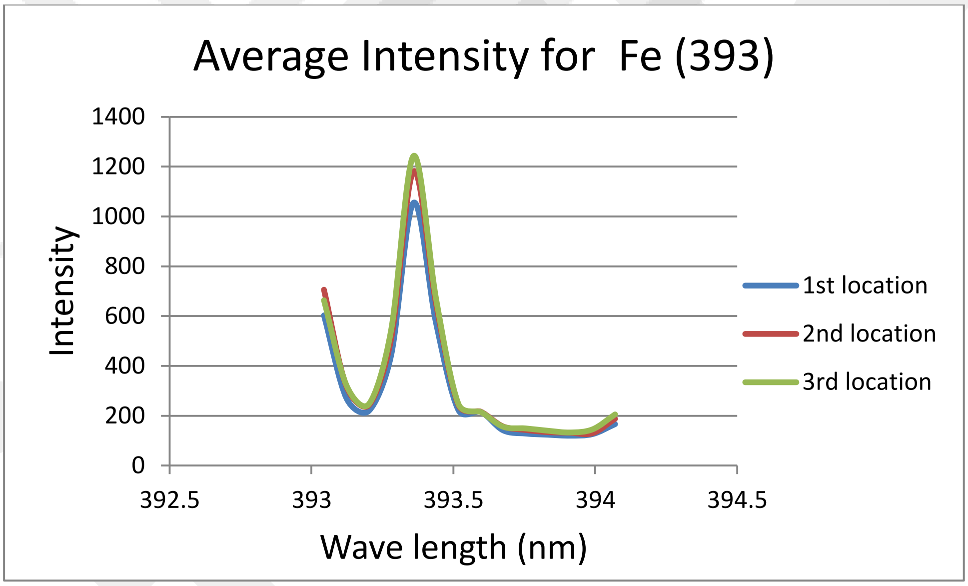

fig (4.143). Average Intensity for Fe (393)

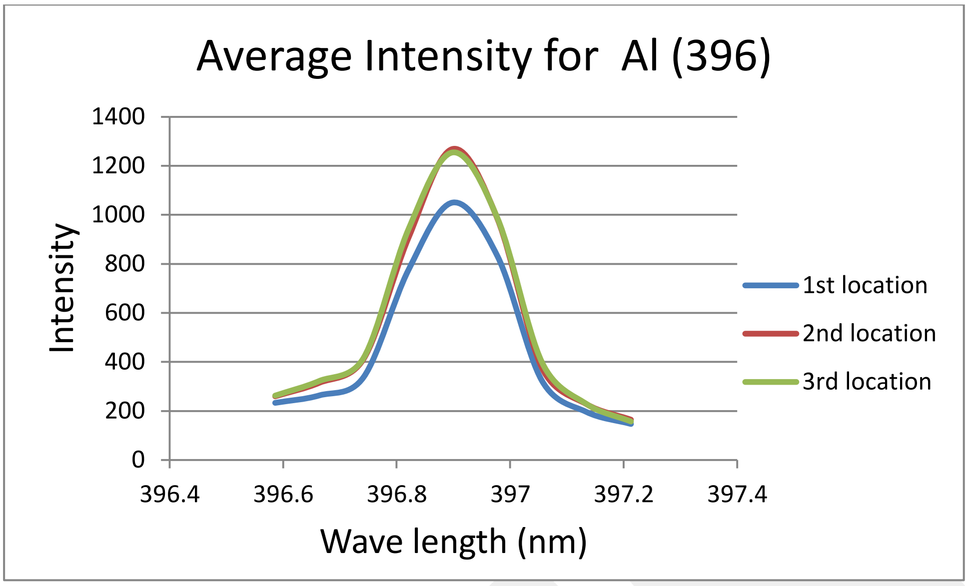

fig (4.144). Average Intensity for Al (396)

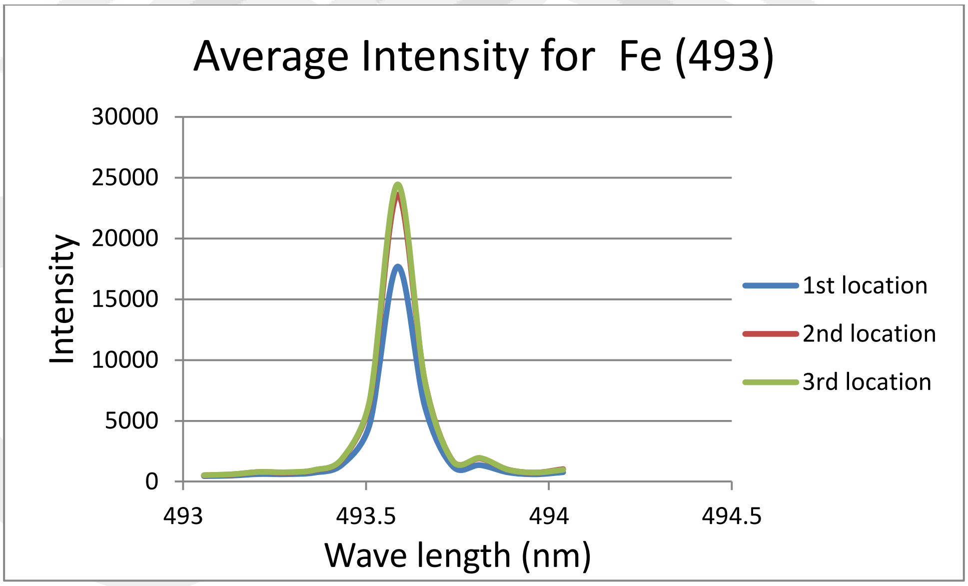

fig (4.145). Average Intensity for Fe (493)

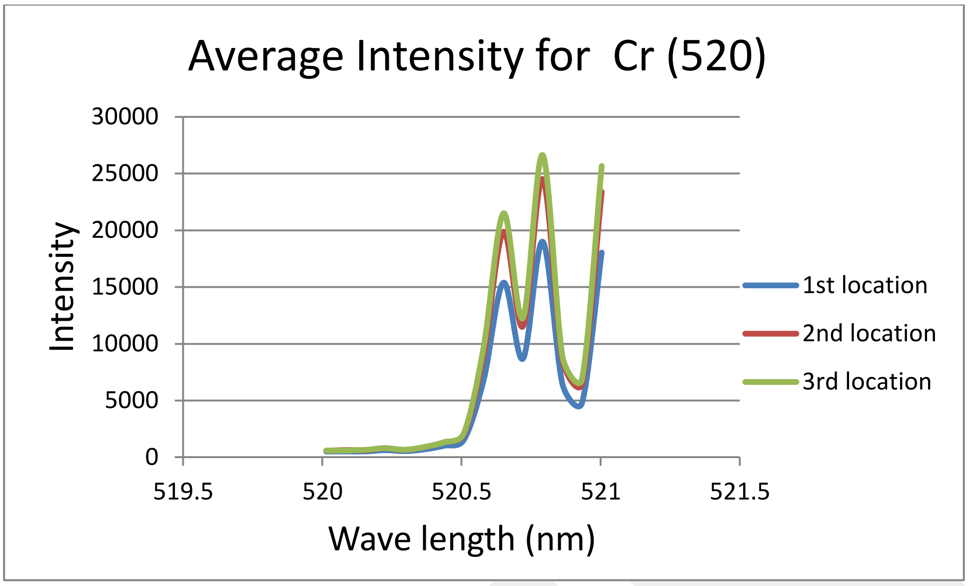

fig (4.146). Average Intensity for Cr (520)

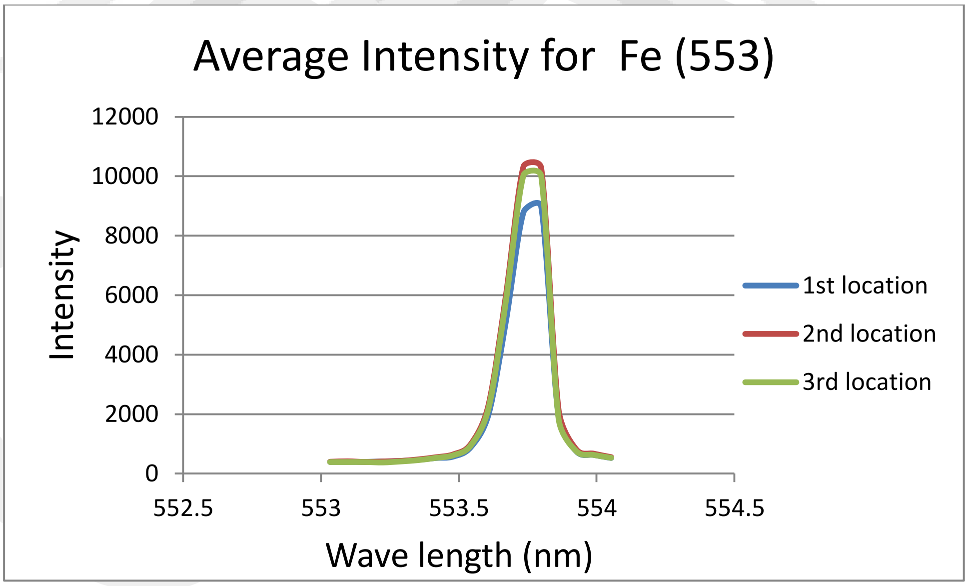

fig (4.147). Average Intensity for Fe (553)

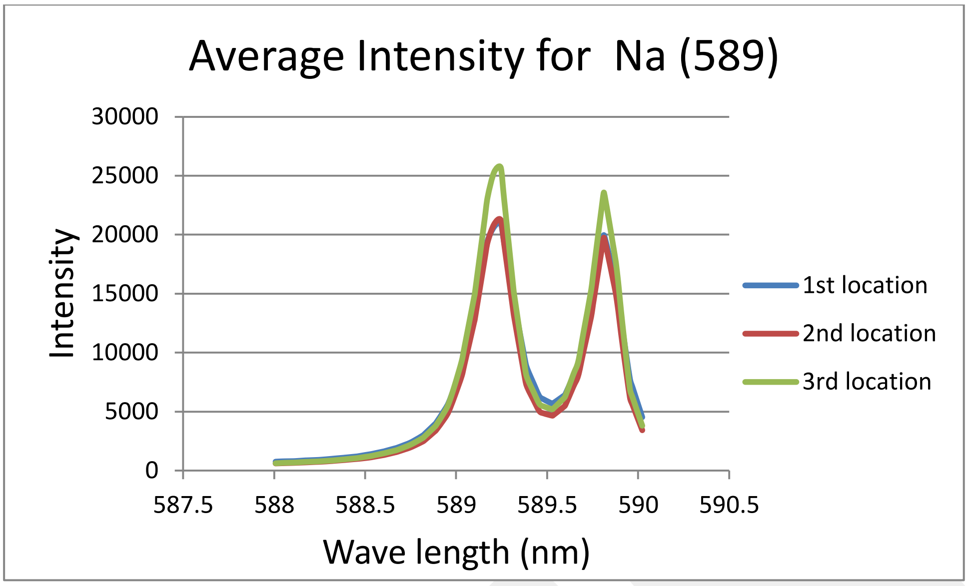

fig (4.148). Average Intensity for Na (589)

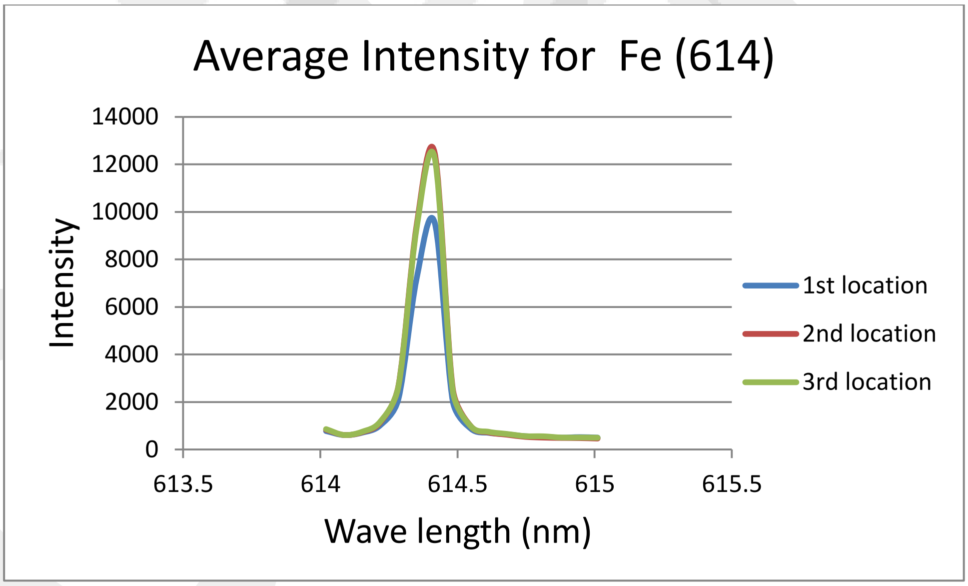

fig (4.149). Average Intensity for Fe (614)

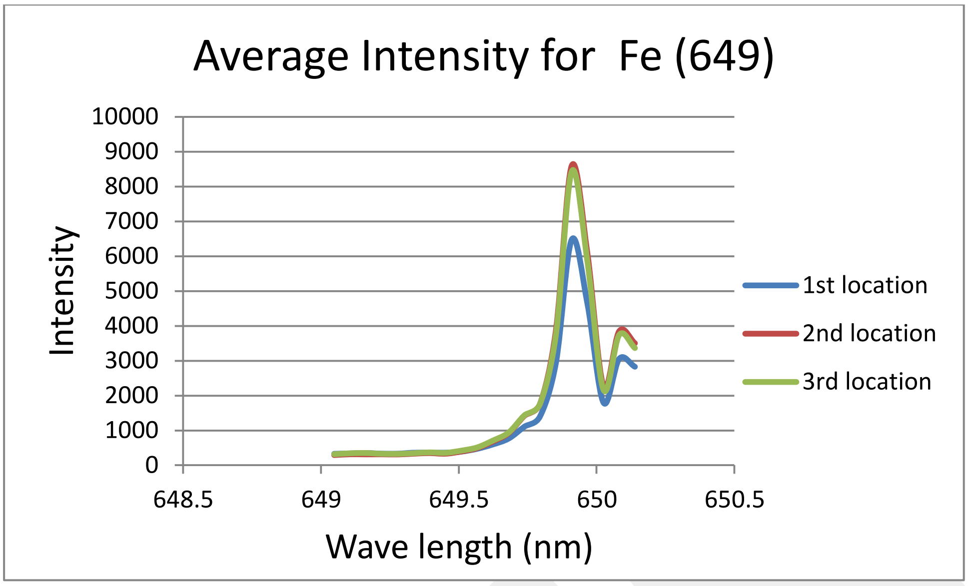

fig (4.150). Average Intensity for Fe (649)

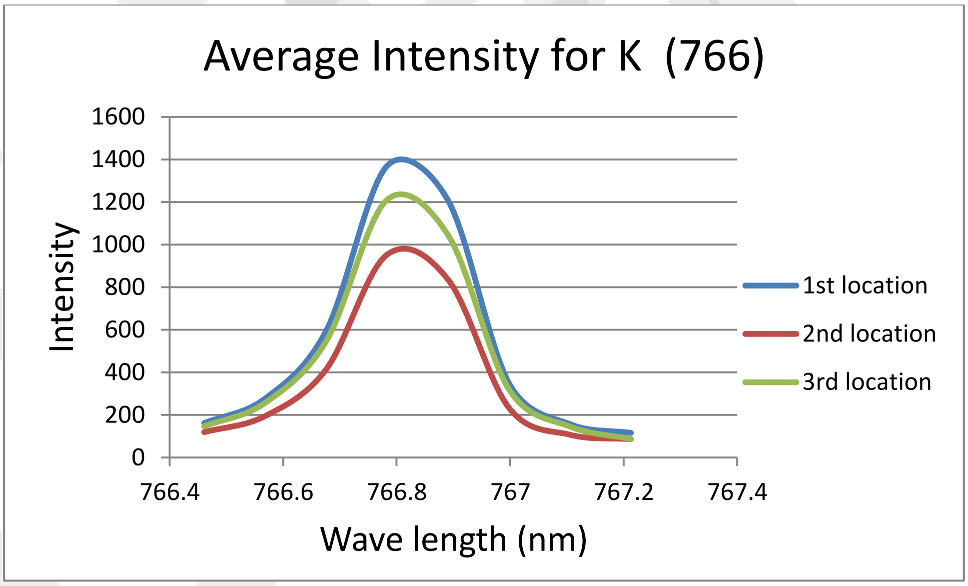

fig (4.151). Average Intensity for K (766)

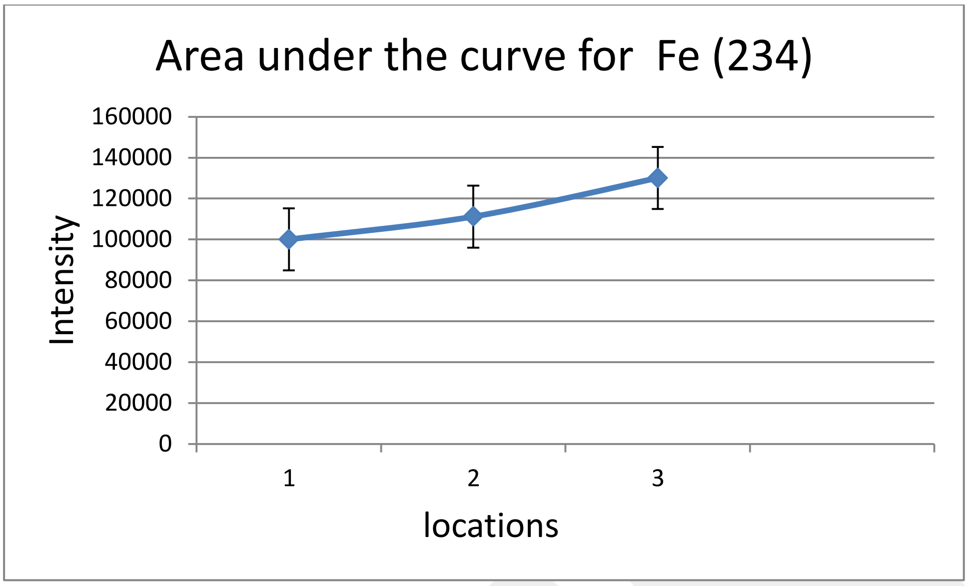

fig (4.152). Area under the curve for Fe (234)

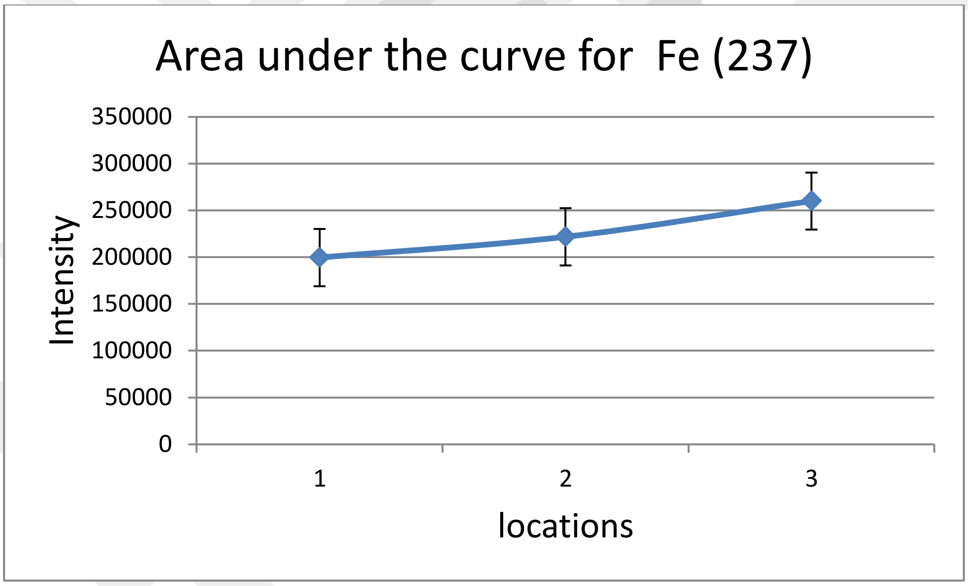

fig (4.153). Area under the curve for Fe (237)

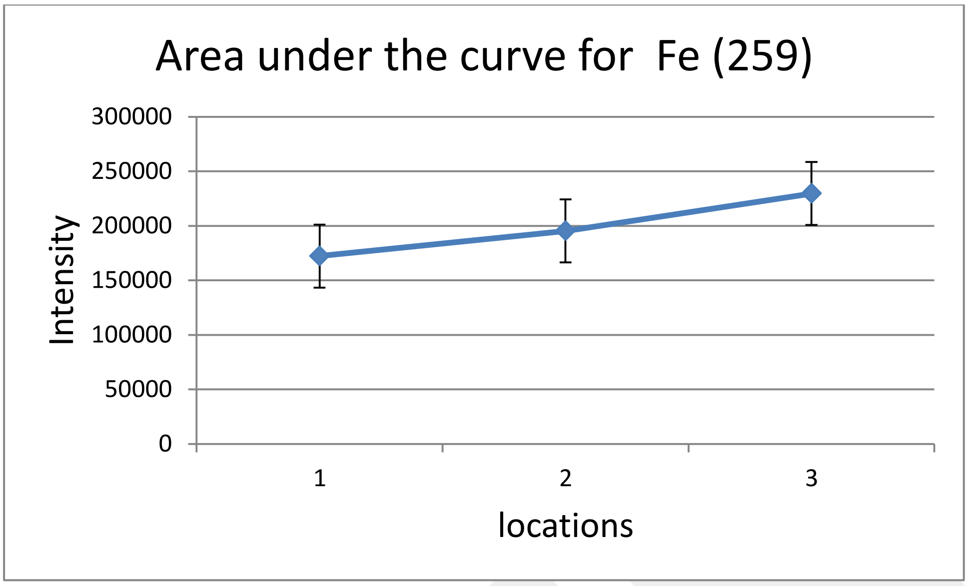

fig (4.154). Area under the curve for Fe (259)

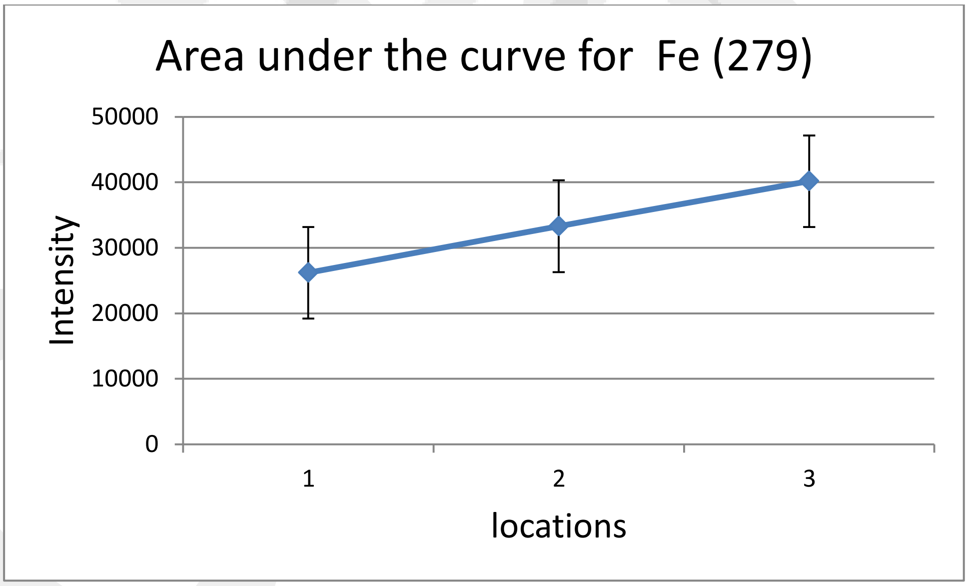

fig (4.155). Area under the curve for Fe (279)

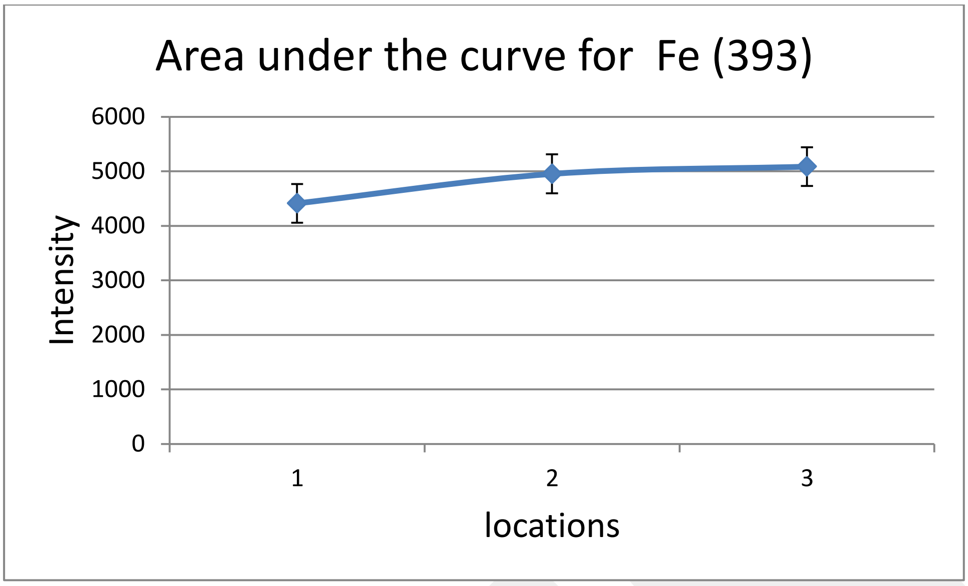

fig (4.156). Area under the curve for Fe (393)

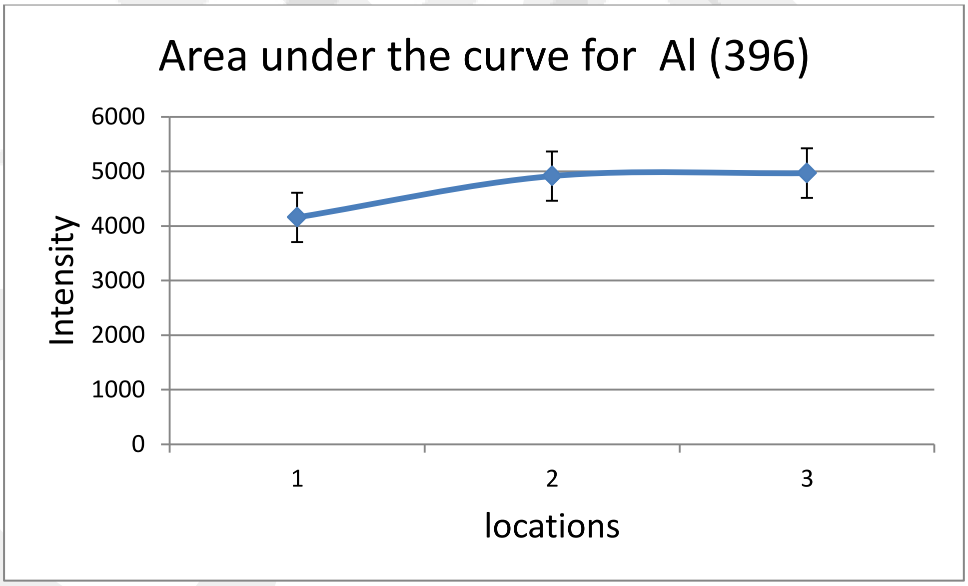

fig (4.157). Area under the curve for Al (396)

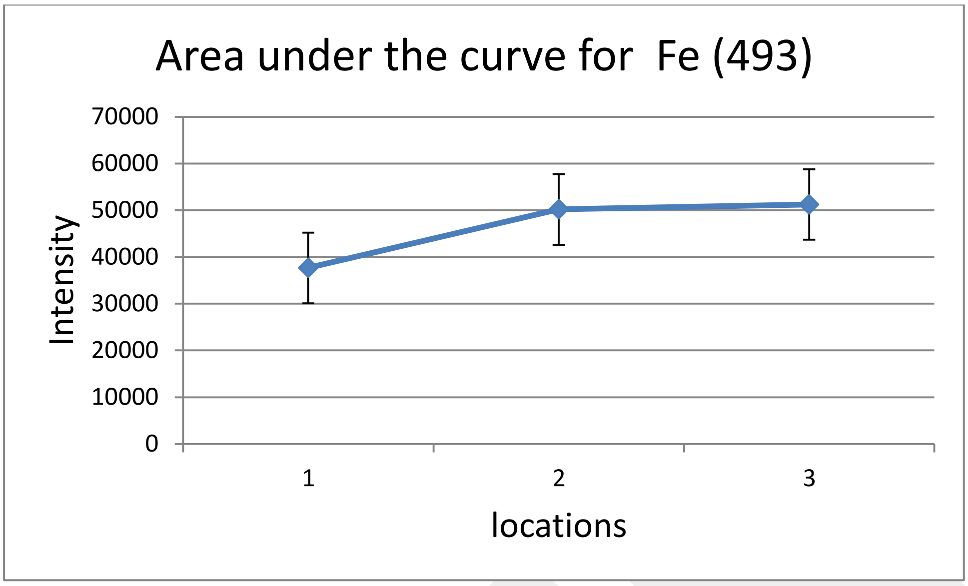

fig (4.158). Area under the curve for Fe (493)

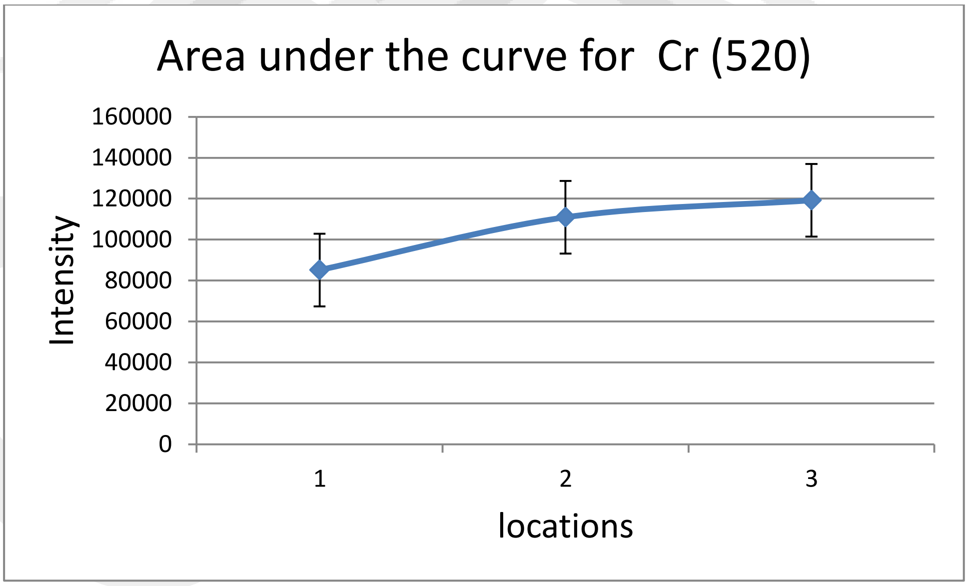

fig (4.159). Area under the curve for Cr (520)

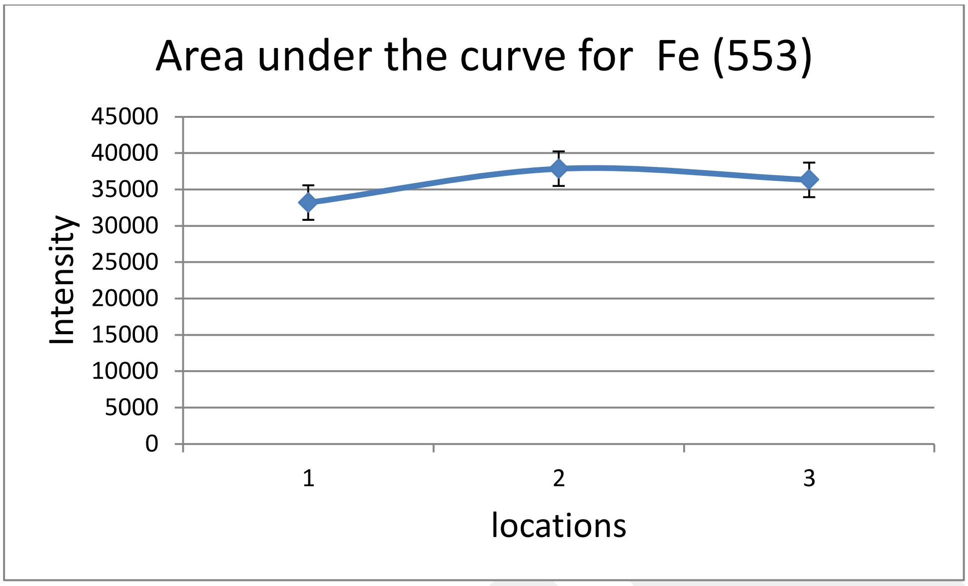

fig (4.160). Area under the curve for Fe (553)

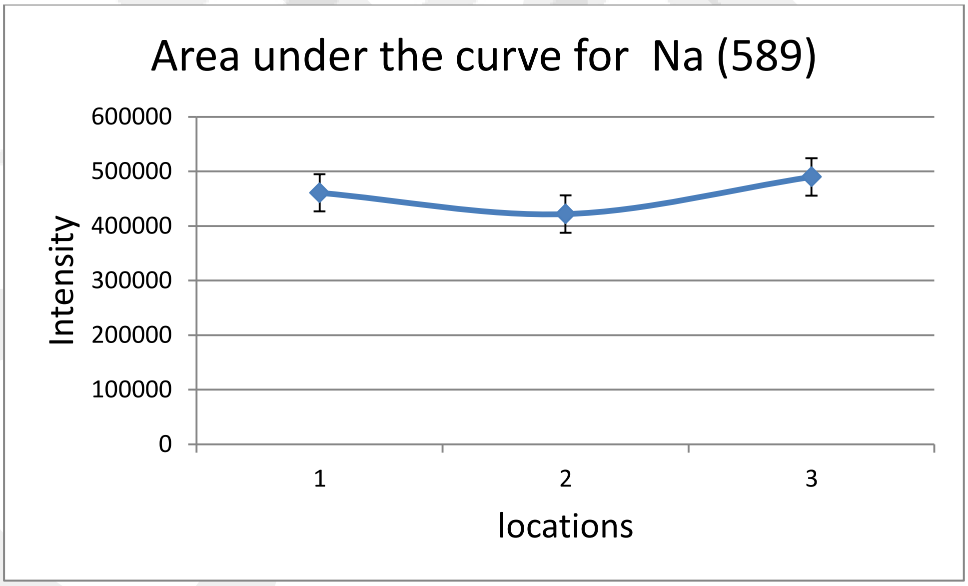

fig (4.161). Area under the curve for Na (589)

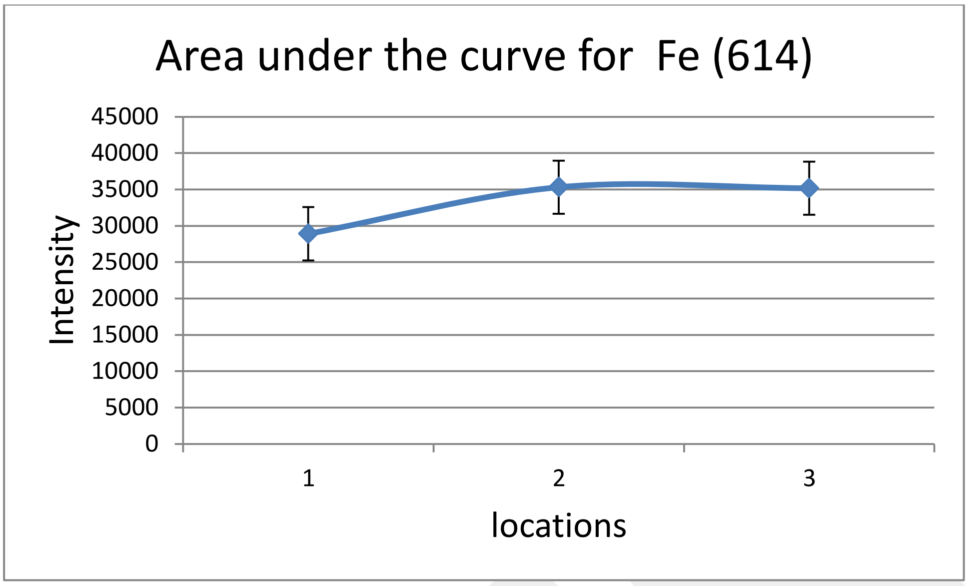

fig (4.162). Area under the curve for Fe (614)

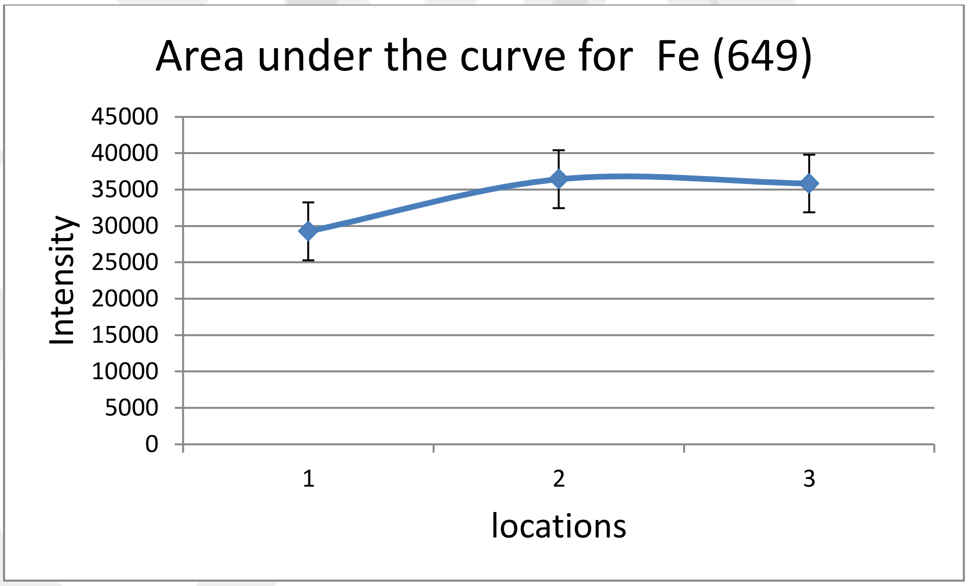

fig (4.163). Area under the curve for Fe (649)

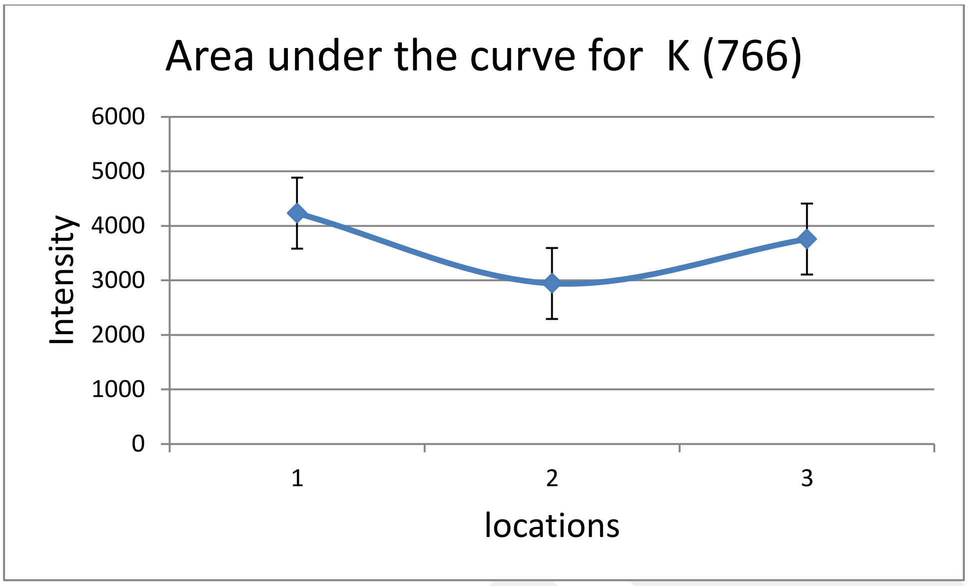

fig (4.164). Area under the curve for K (766)

After that we used 0,5 μs time delay, and power of 20 mJ, with frequency 10 hz, and 50 shots, then we have moved the alloy in three locations, and we got the following results as shown in the following figures.

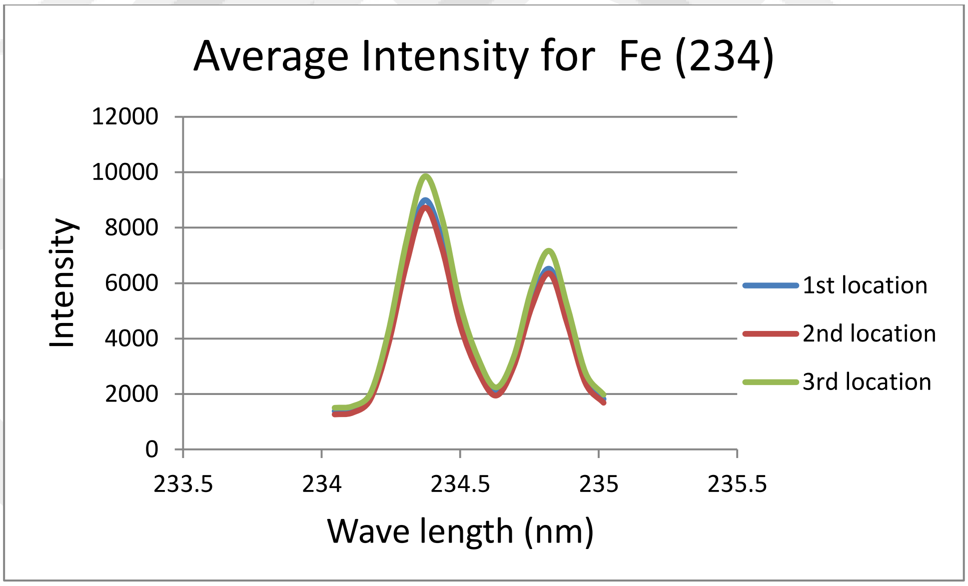

fig (4.165). Average Intensity for Fe (234)

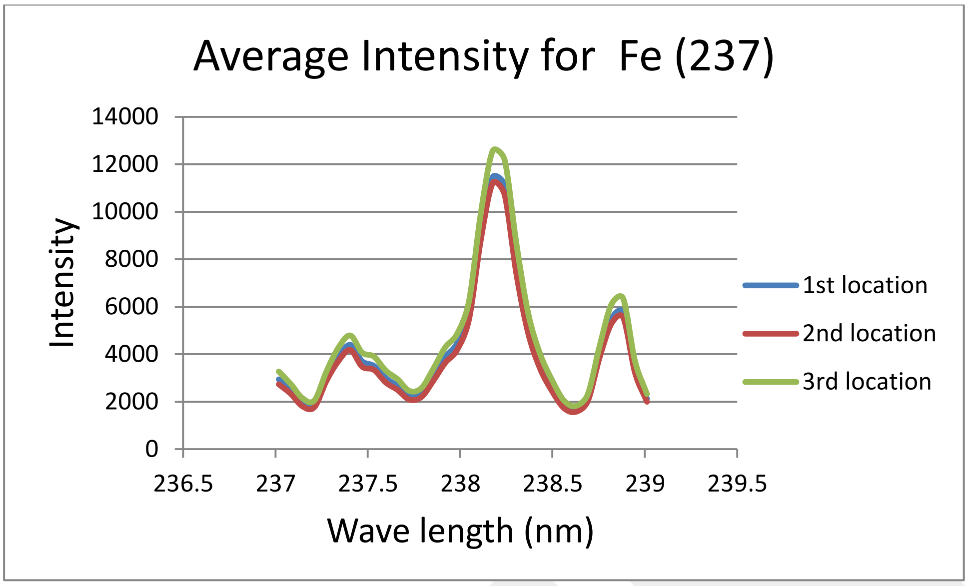

fig (4.166). Average Intensity for Fe (237)

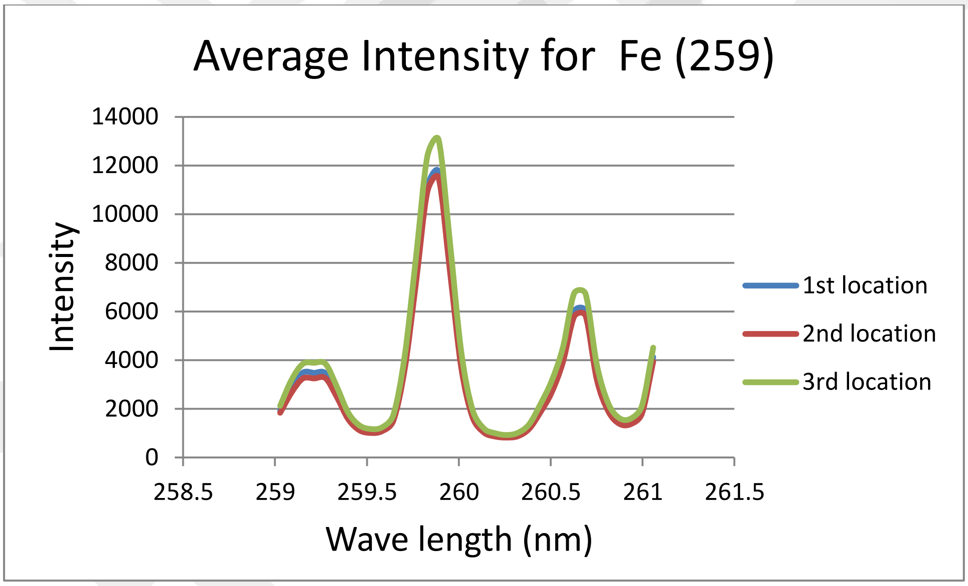

fig (4.167). Average Intensity for Fe (259)

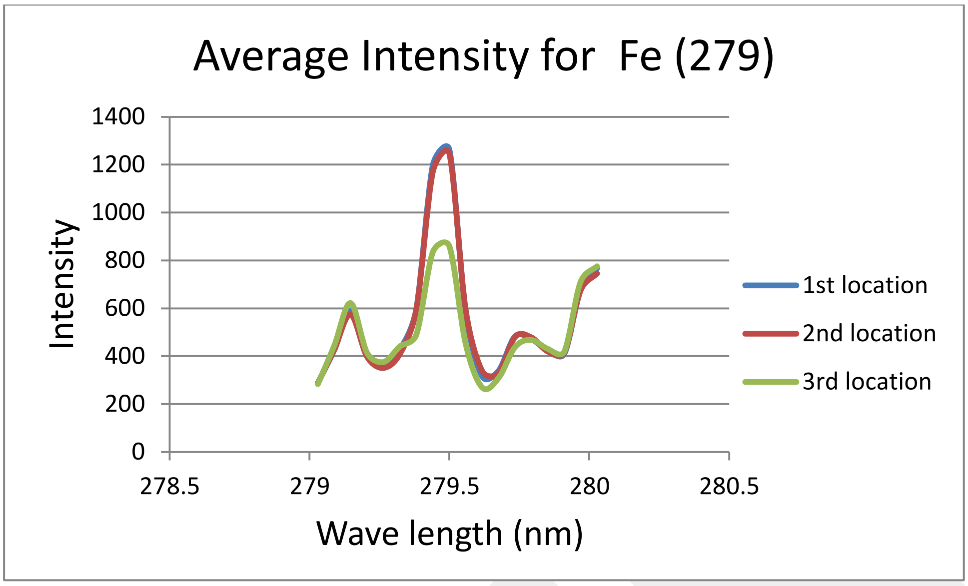

fig (4.168). Average Intensity for Fe (279)

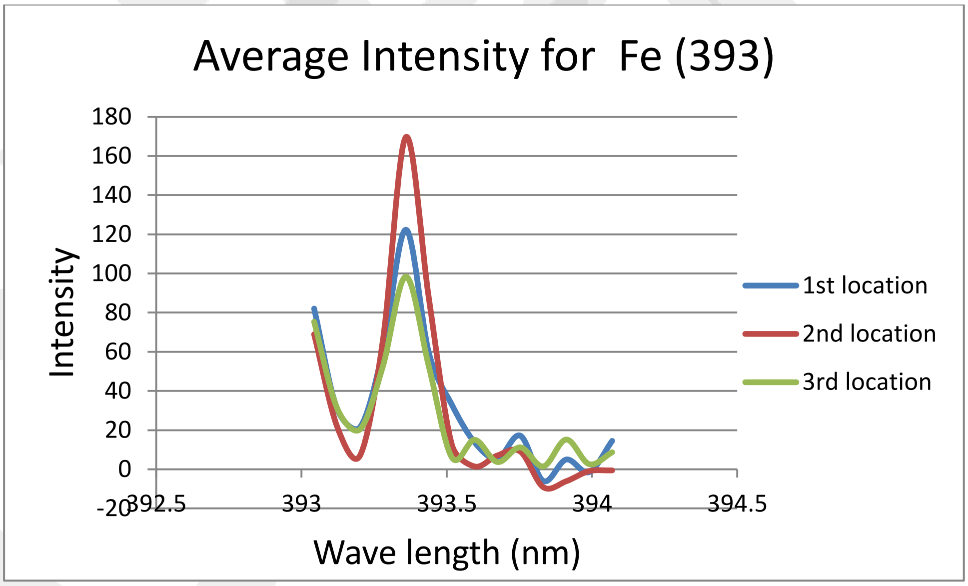

fig (4.169). Average Intensity for Fe (393)

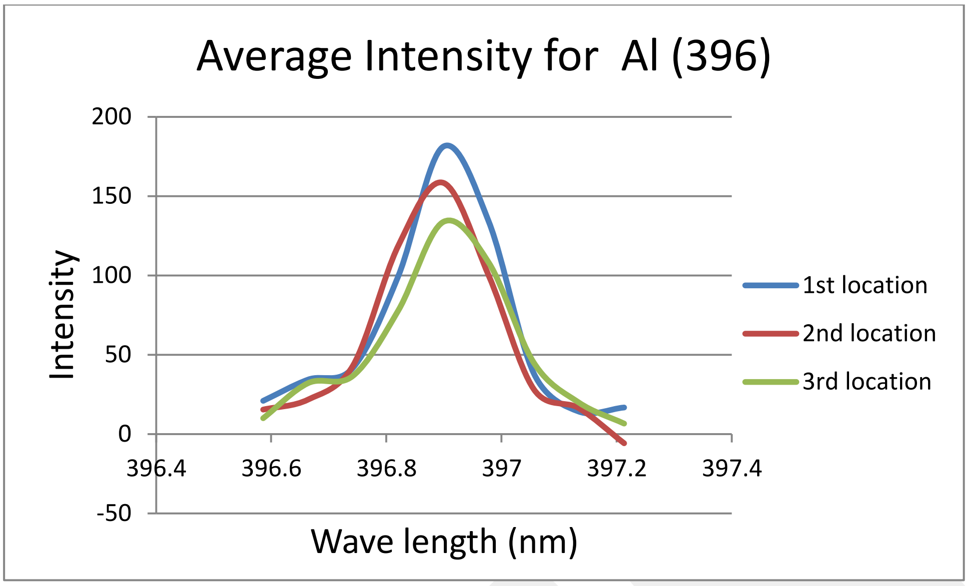

fig (4.170). Average Intensity for Al (396)

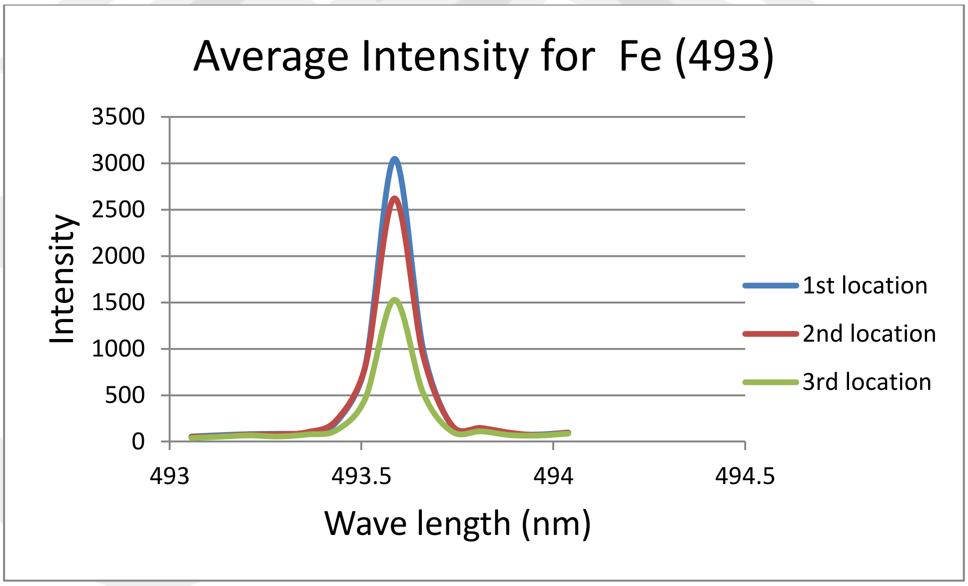

fig (4.171). Average Intensity for Fe (493)

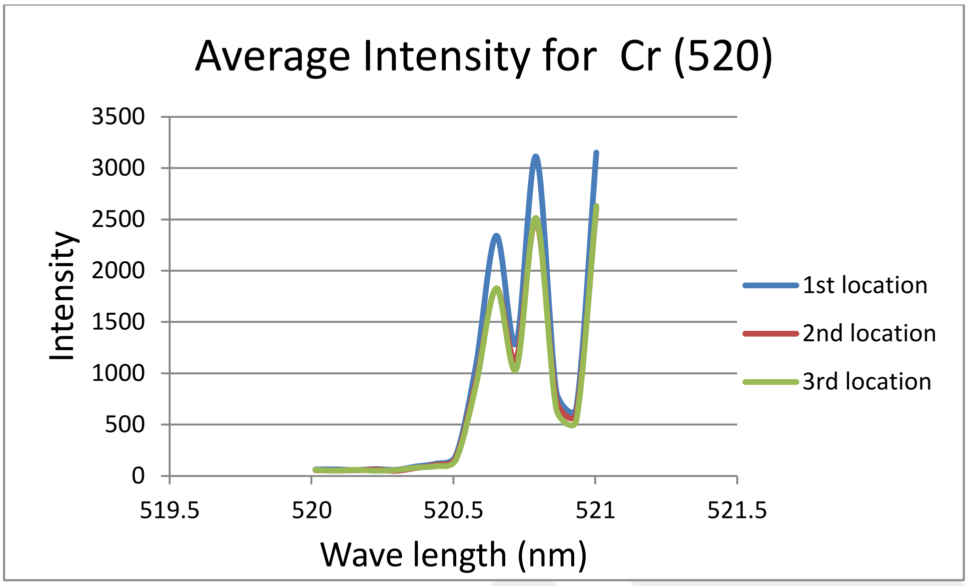

fig (4.172). Average Intensity for Cr (520)

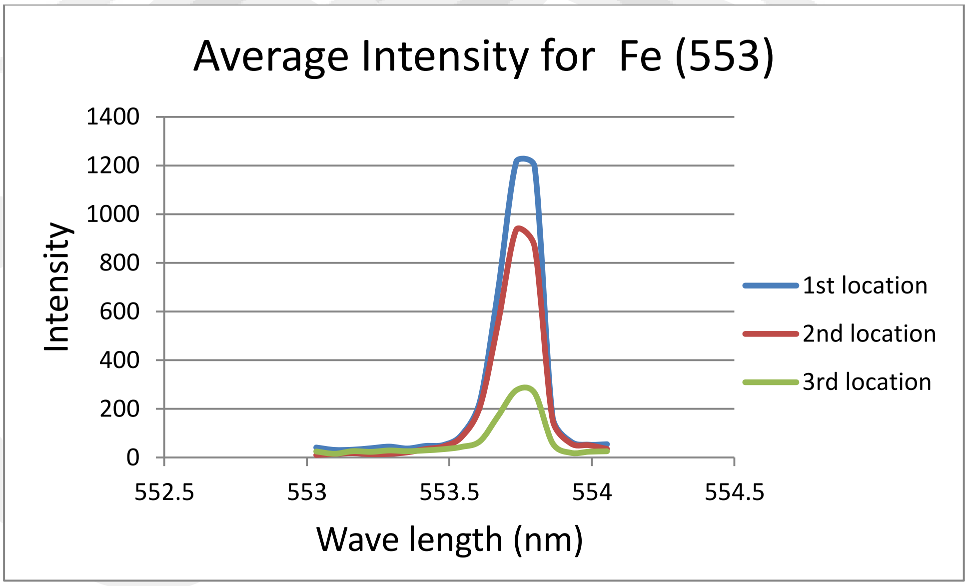

fig (4.173). Average Intensity for Fe (553)

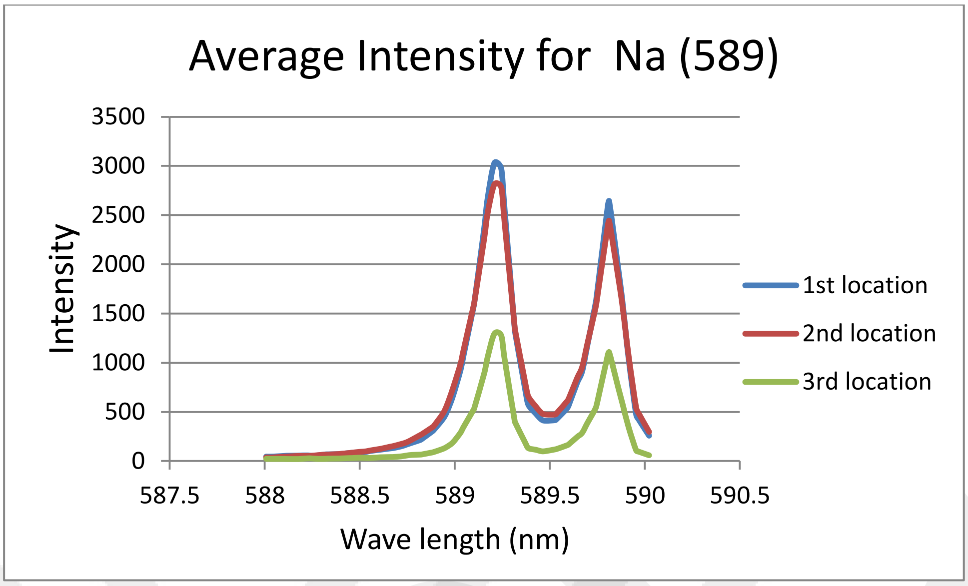

fig (4.174). Average Intensity for Na (589)

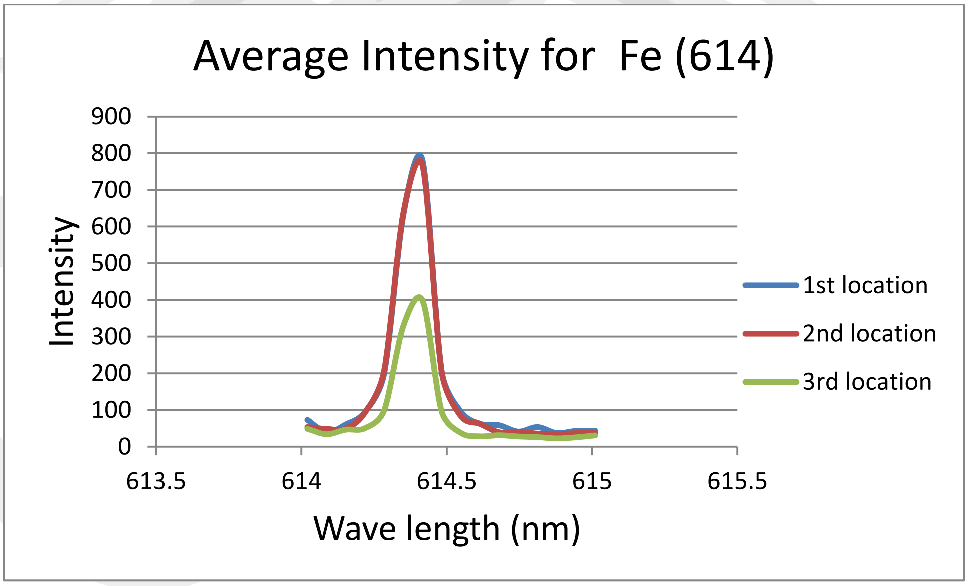

fig (4.175). Average Intensity for Fe (614)

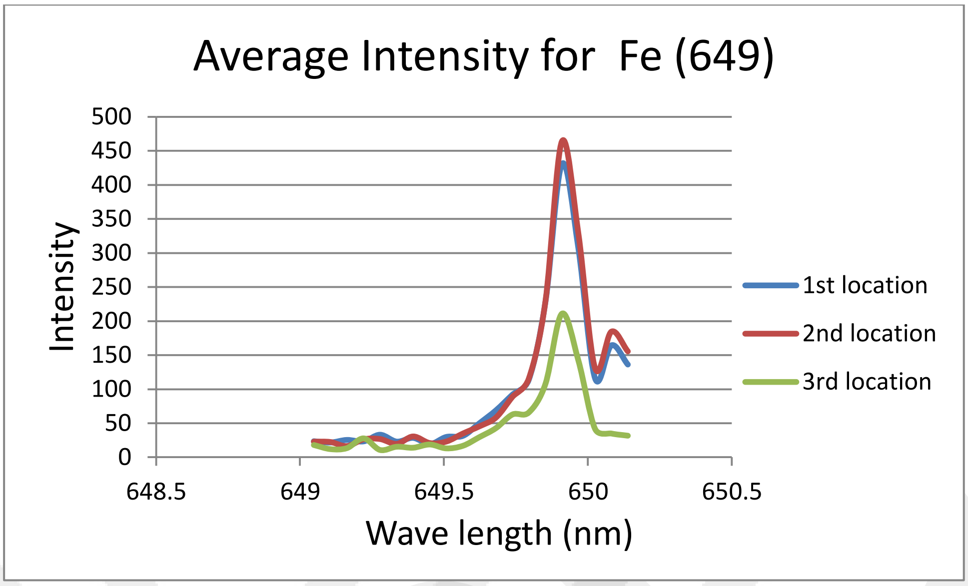

fig (4.176). Average Intensity for Fe (649)

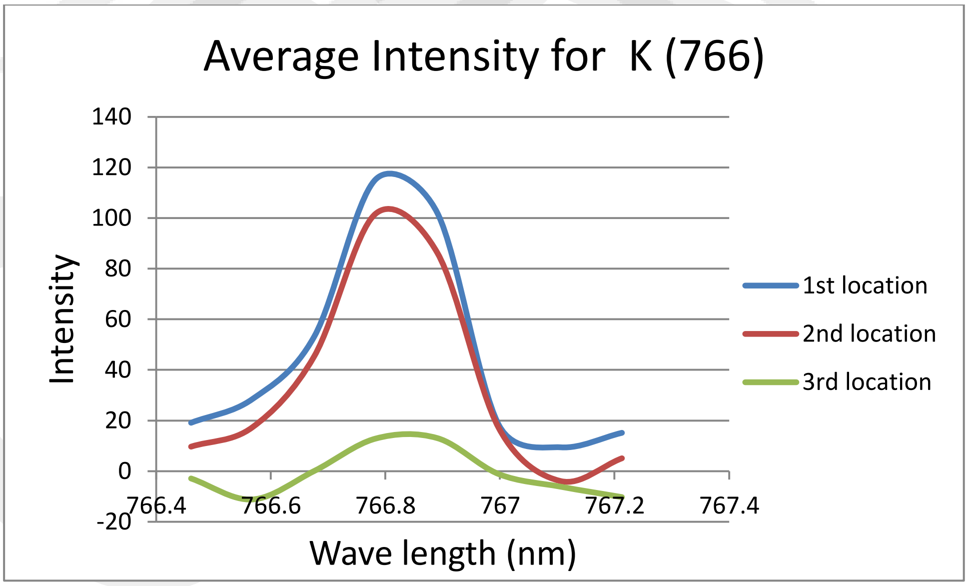

fig (4.177). Average Intensity for K (766)

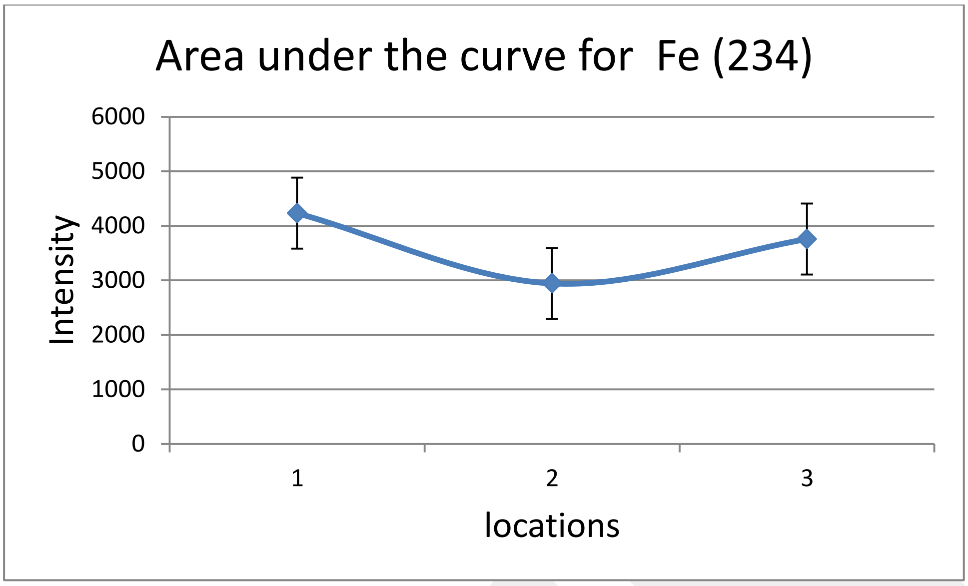

fig (4.178). Area under the curve for Fe (234)

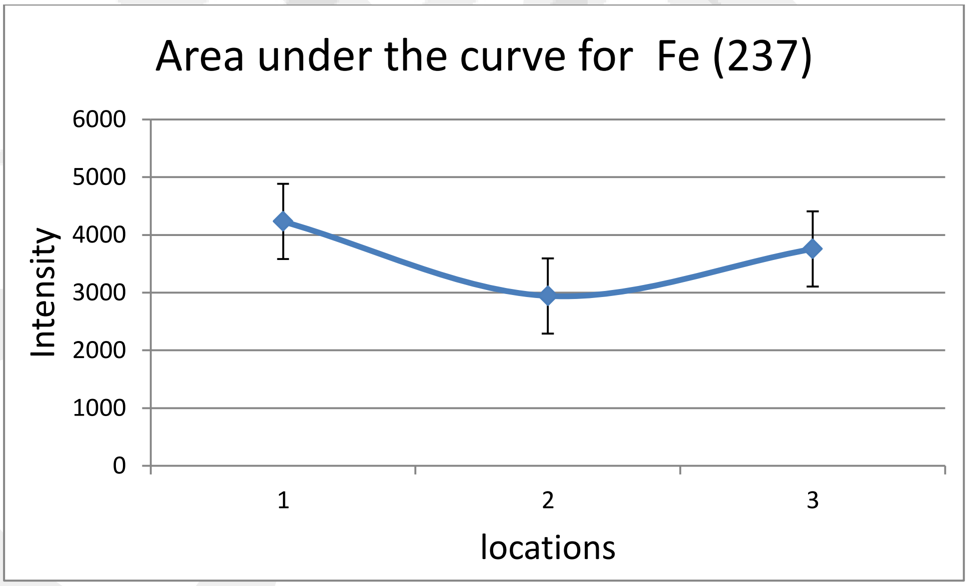

fig (4.179). Area under the curve for Fe (237)

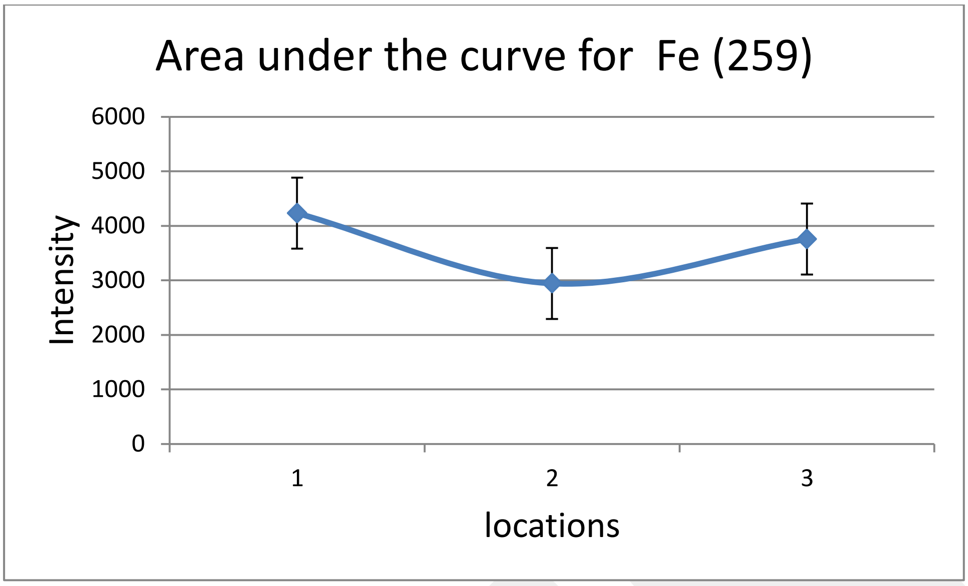

fig (4.180). Area under the curve for Fe (259)

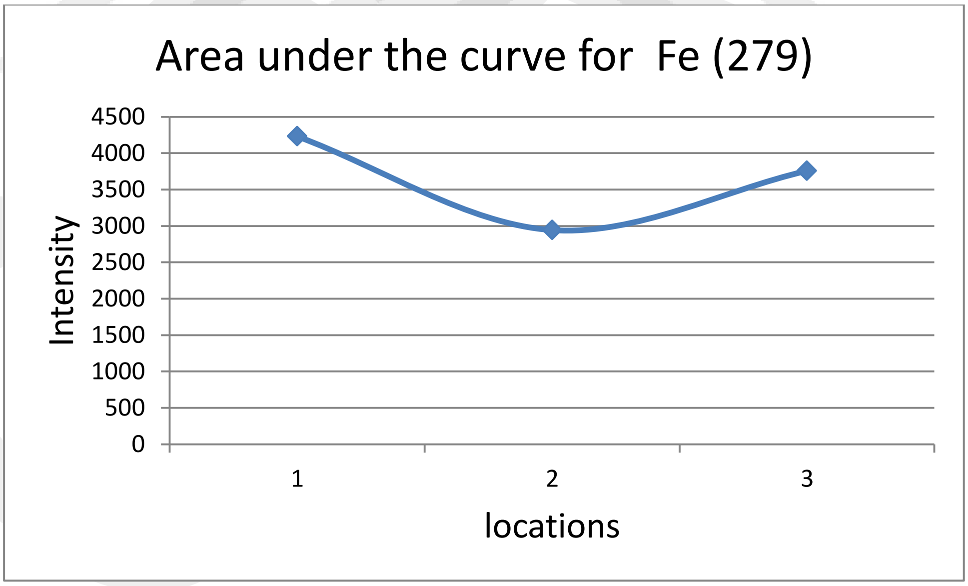

fig (4.181). Area under the curve for Fe (279)

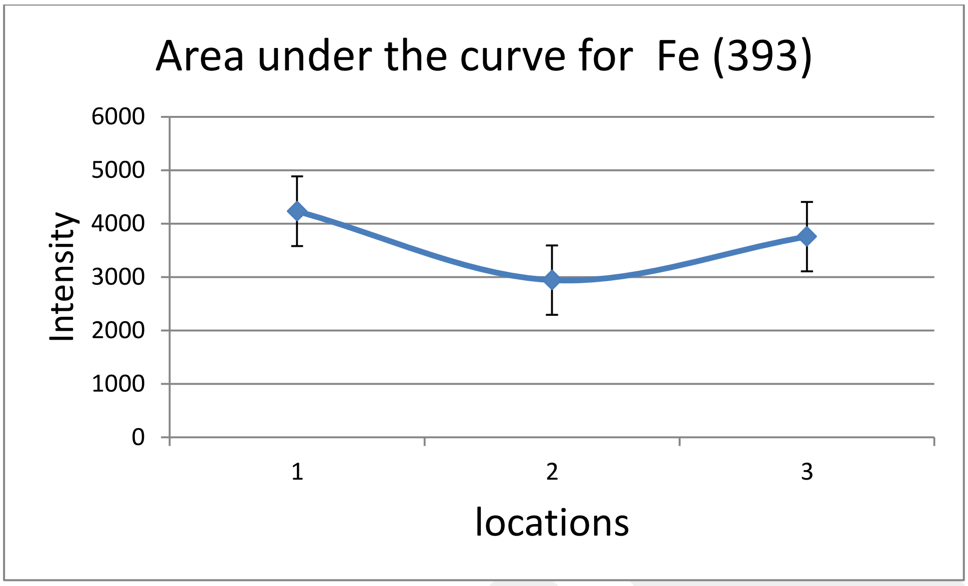

fig (4.182). Area under the curve for Fe (393)

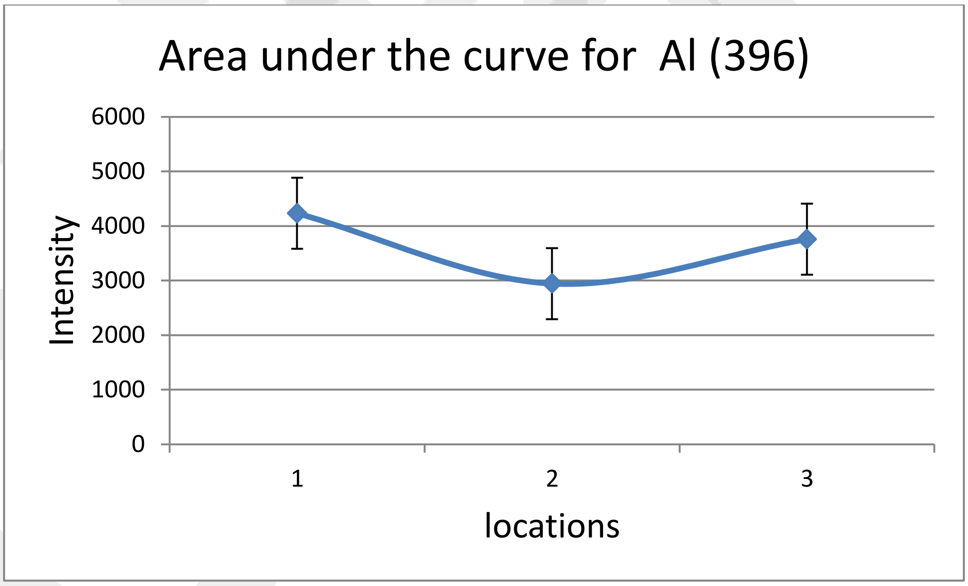

fig (4.183). Area under the curve for Al (396)

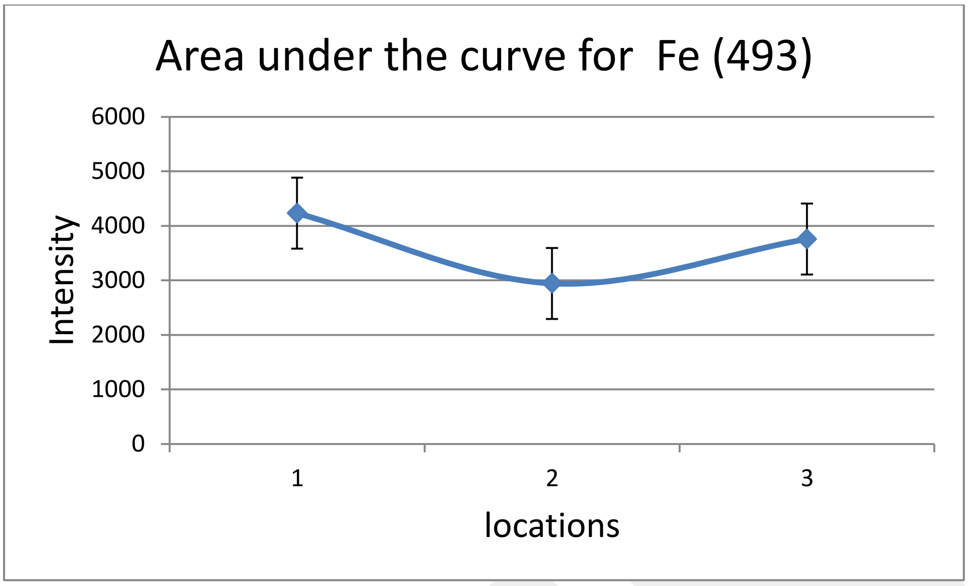

fig (4.184). Area under the curve for Fe (493)

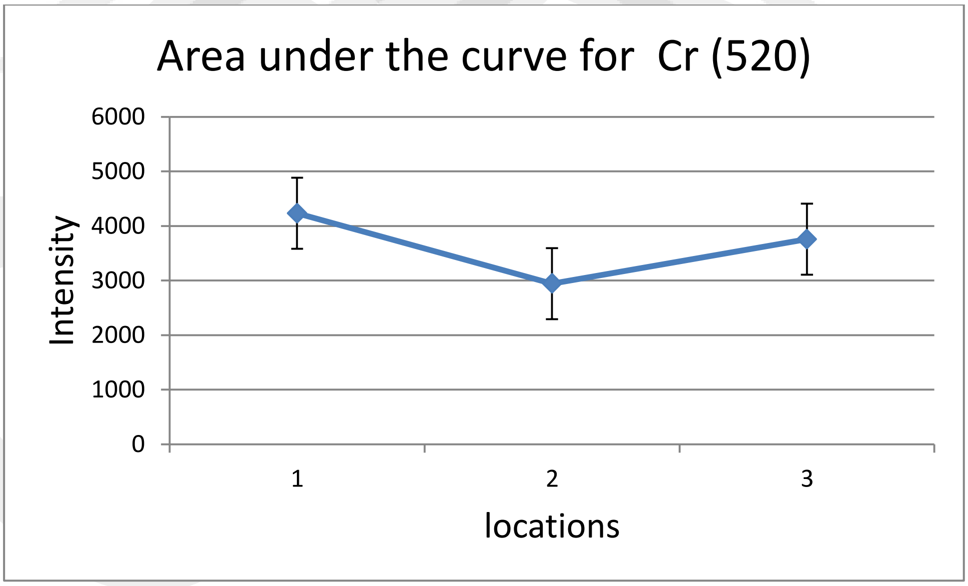

fig (4.185). Area under the curve for Cr (520)

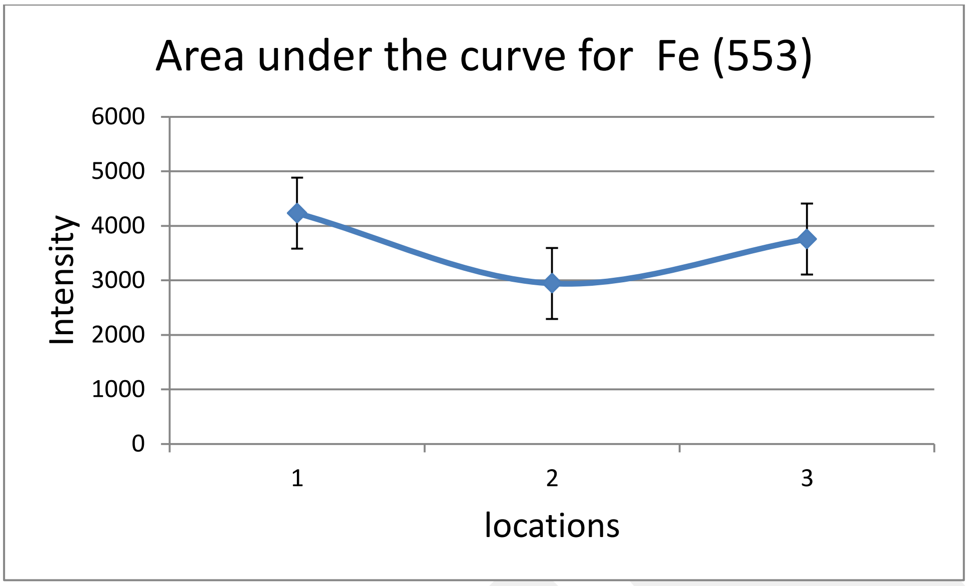

fig (4.186). Area under the curve for Fe (553)

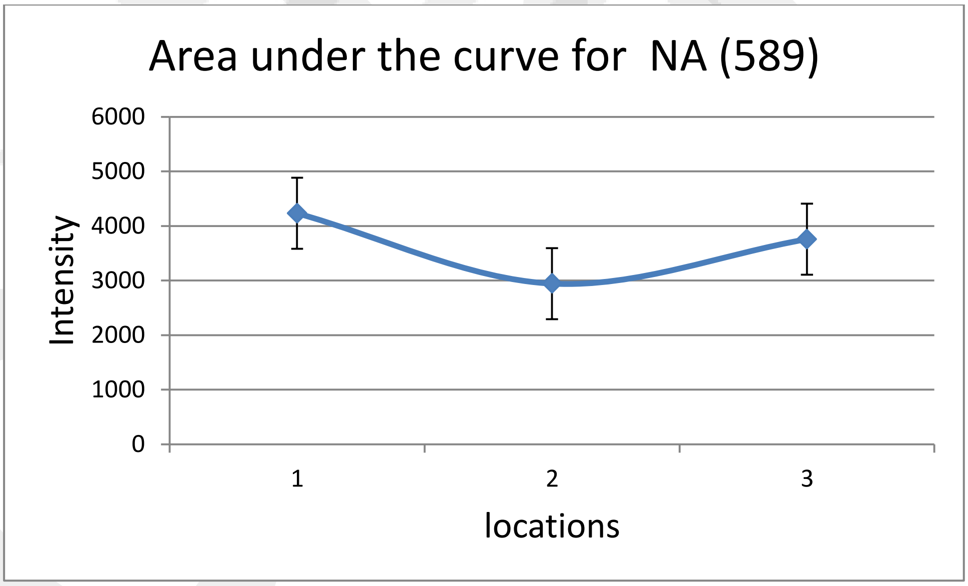

fig (4.187). Area under the curve for NA (589)

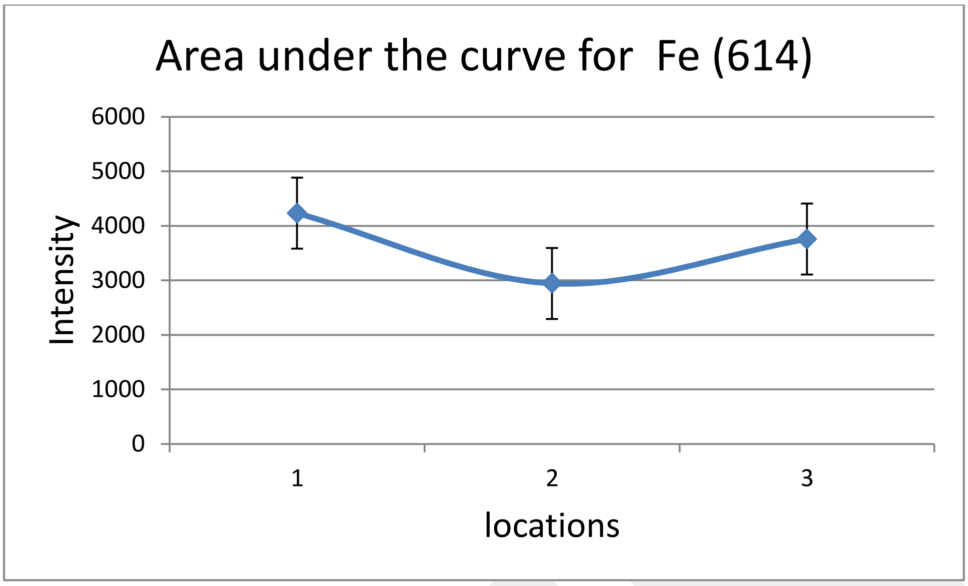

fig (4.188). Area under the curve for Fe (614)

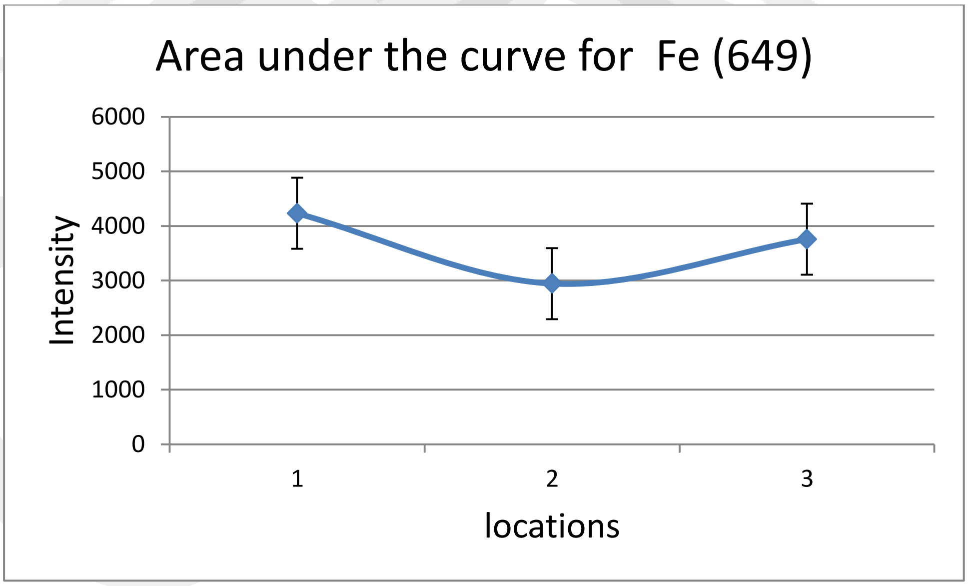

fig (4.189). Area under the curve for Fe (649)

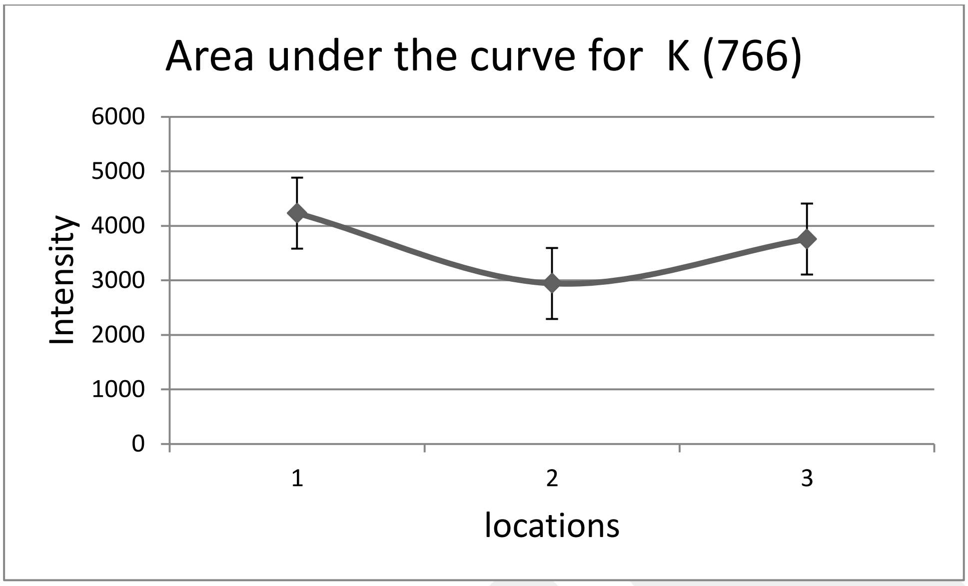

fig (4.190). Area under the curve for K (766)

After that we used 0,5 µs time delay, and power of 30 mJ, with frequency 10 hz, and 50 shots, then we have moved the alloy in three locations, and we got the following results as shown in the following figures.

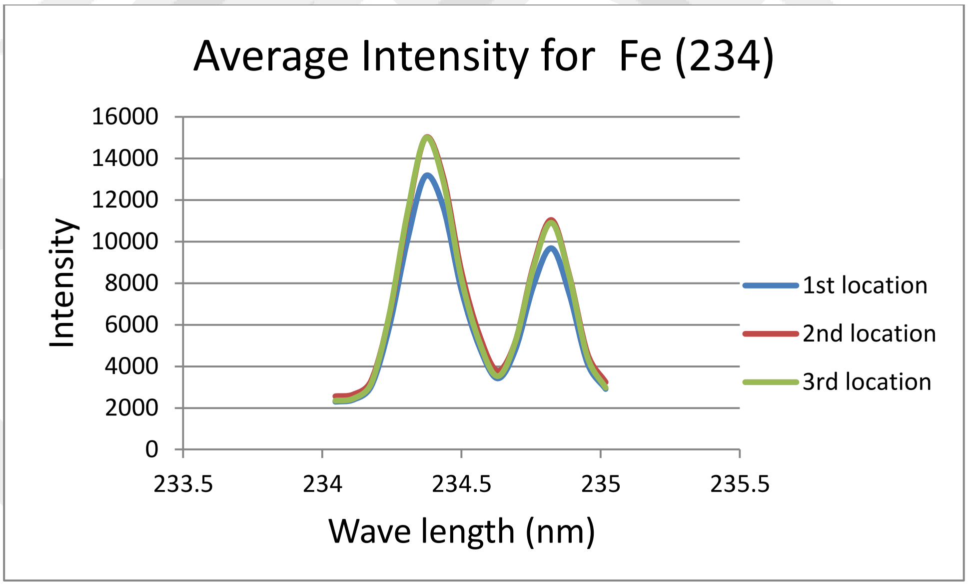

fig (4.191). Average Intensity for Fe (234)

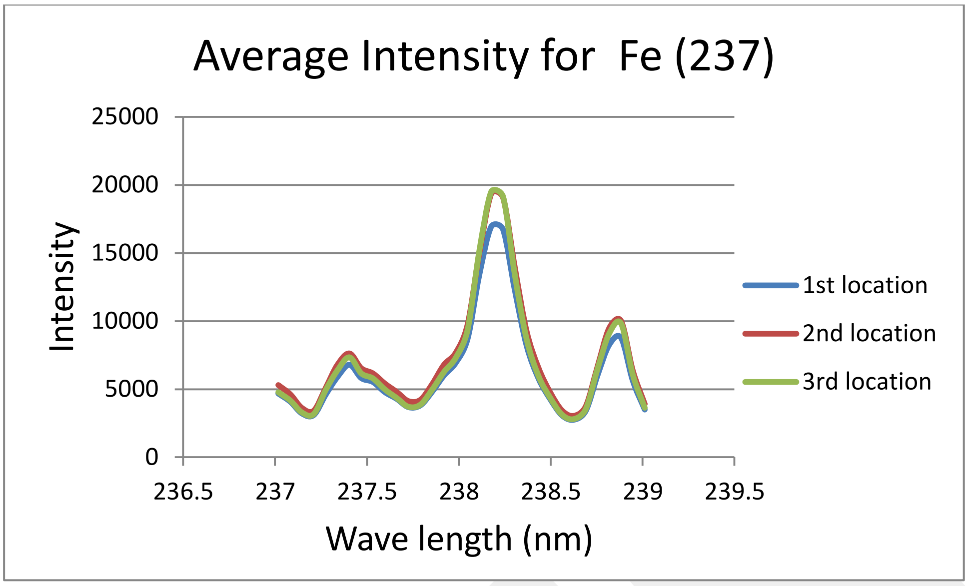

fig (4.192). Average Intensity for Fe (237)

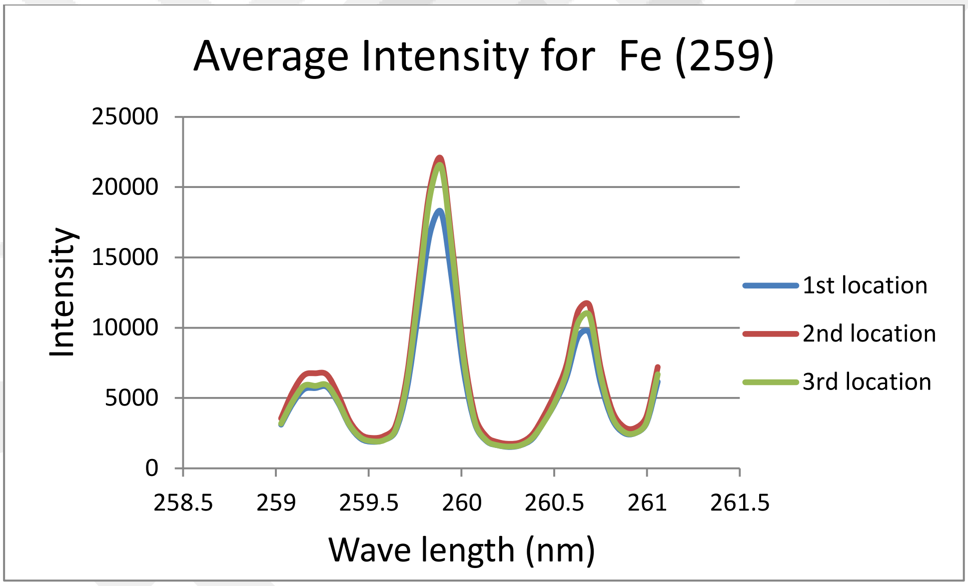

fig (4.193). Average Intensity for Fe (259)

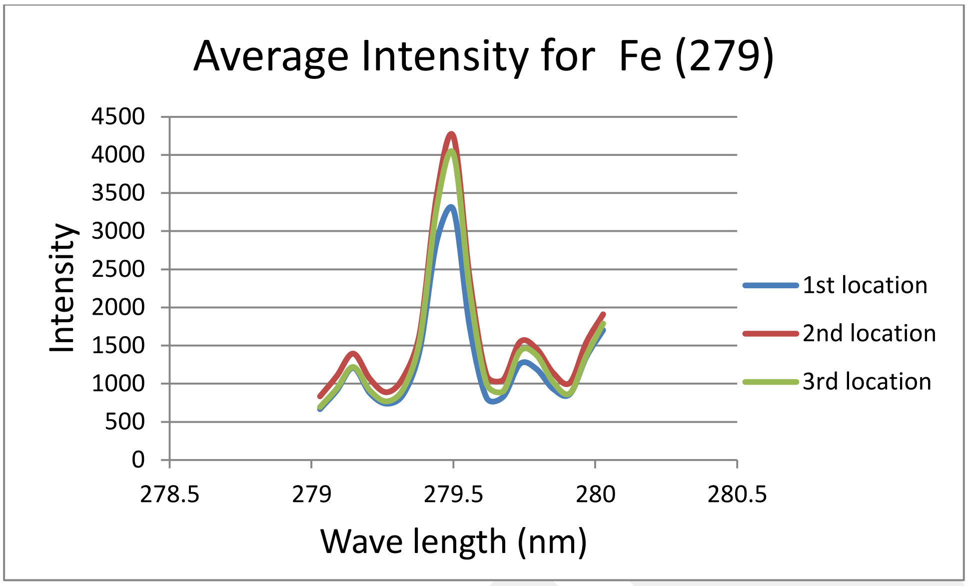

fig (4.194). Average Intensity for Fe (279)

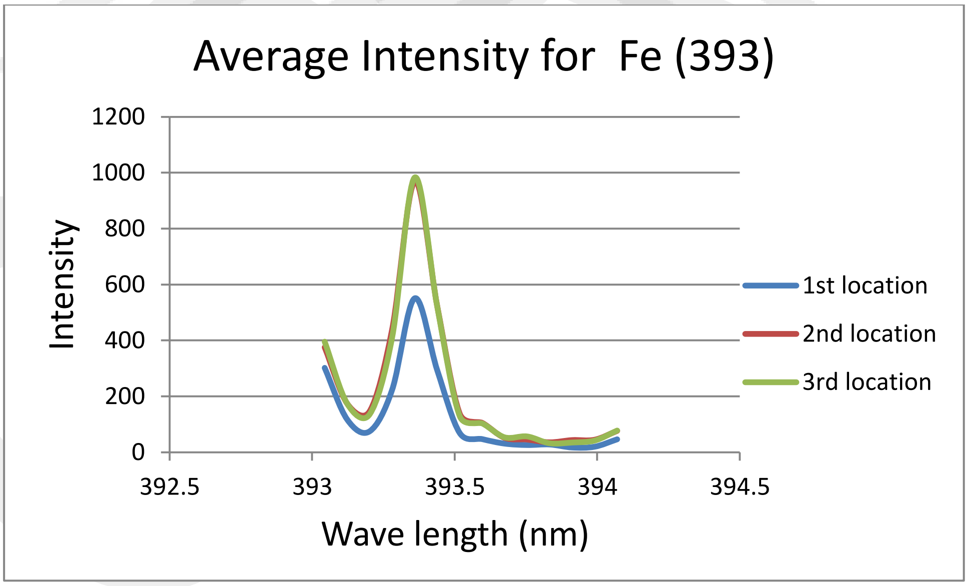

fig (4.195). Average Intensity for Fe (393)

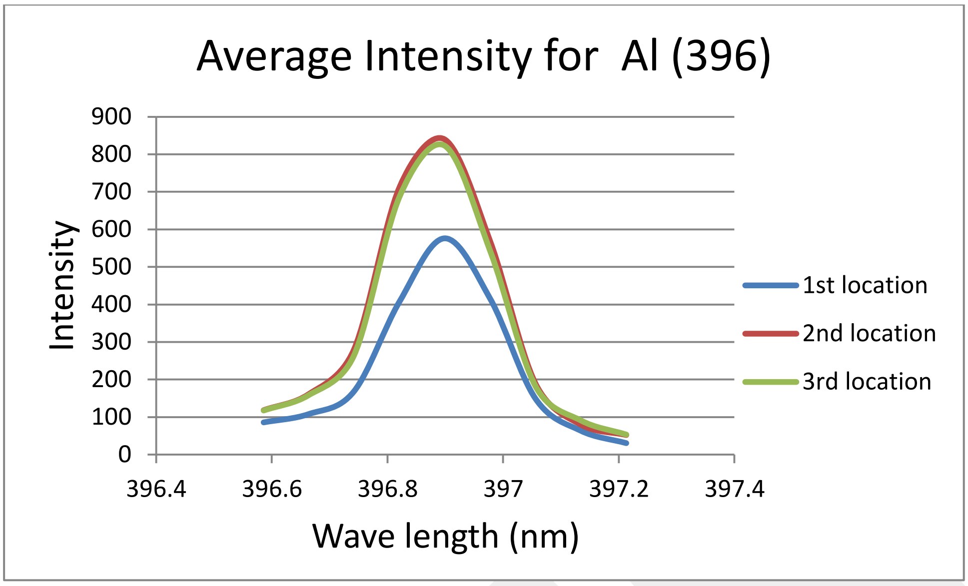

fig (4.196). Average Intensity for Al (396)

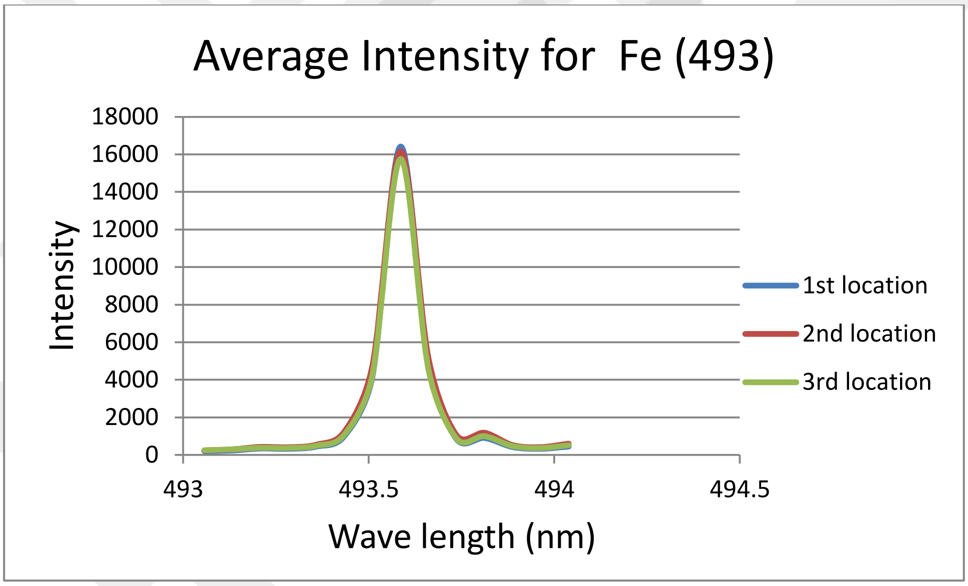

fig (4.197). Average Intensity for Fe (493)

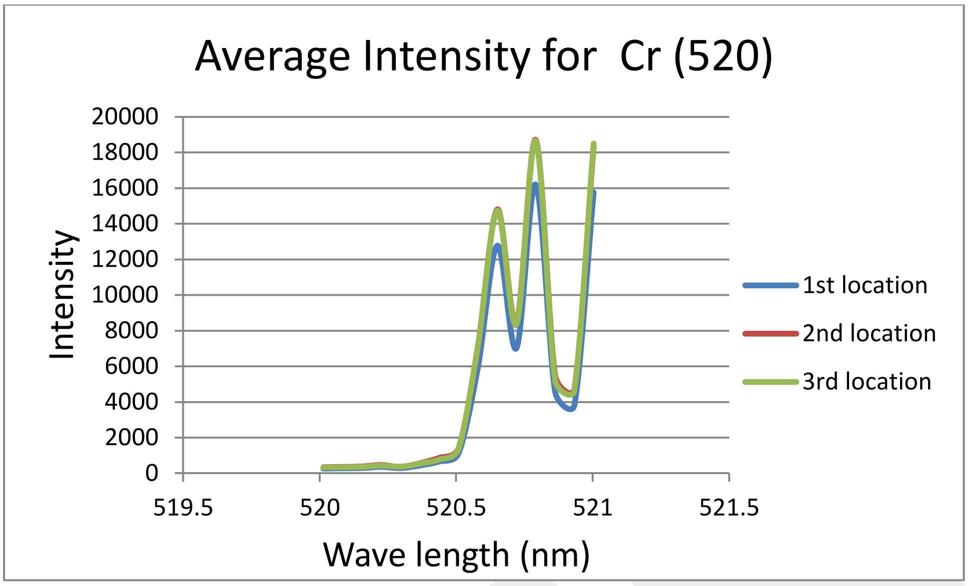

fig (4.198). Average Intensity for Cr (520)

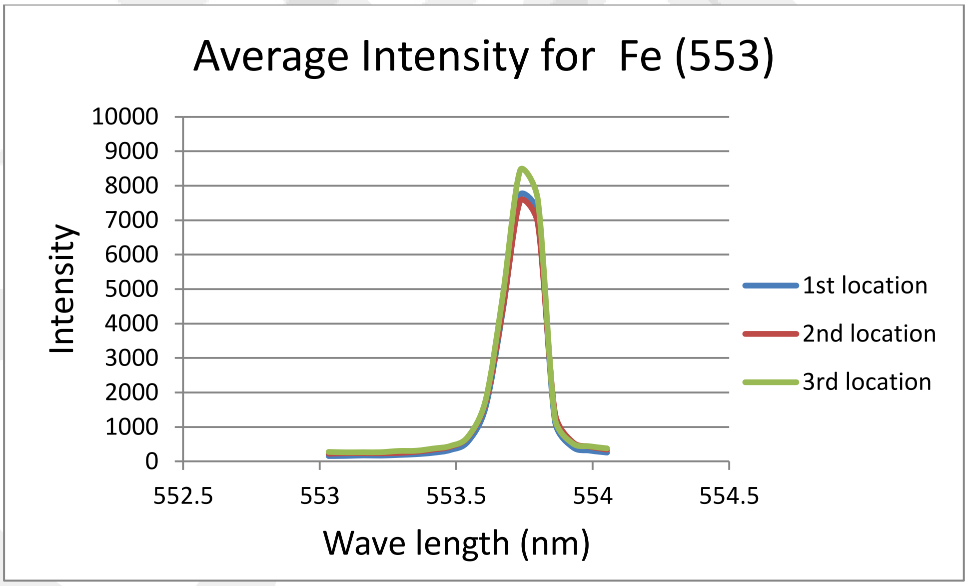

fig (4.199). Average Intensity for Fe (553)

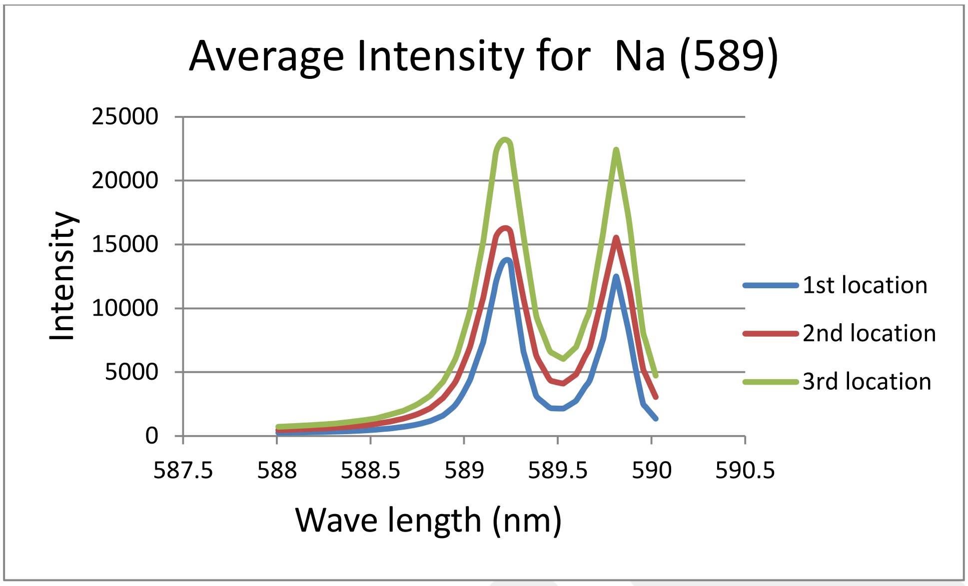

fig (4.200). Average Intensity for Na (589)

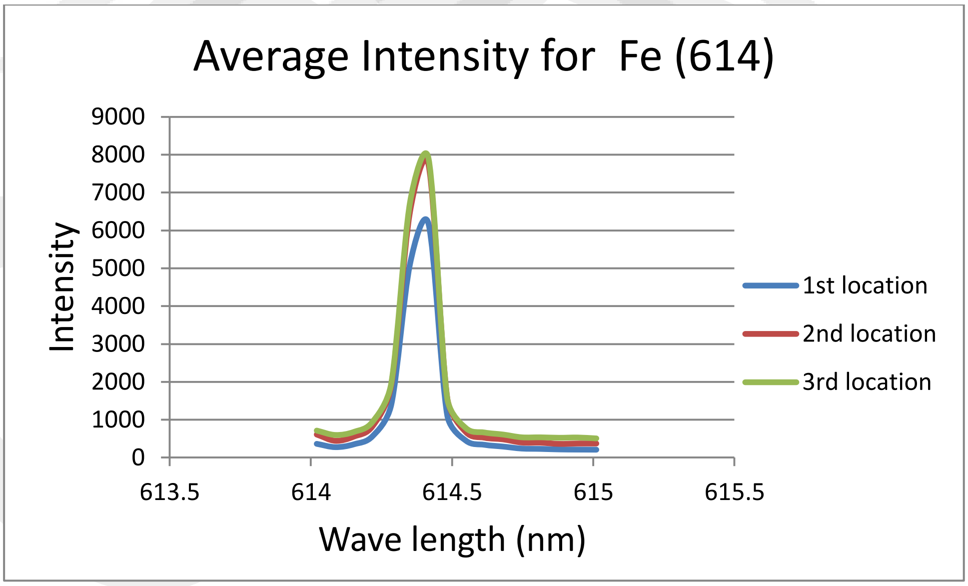

fig (4.201). Average Intensity for Fe (614)

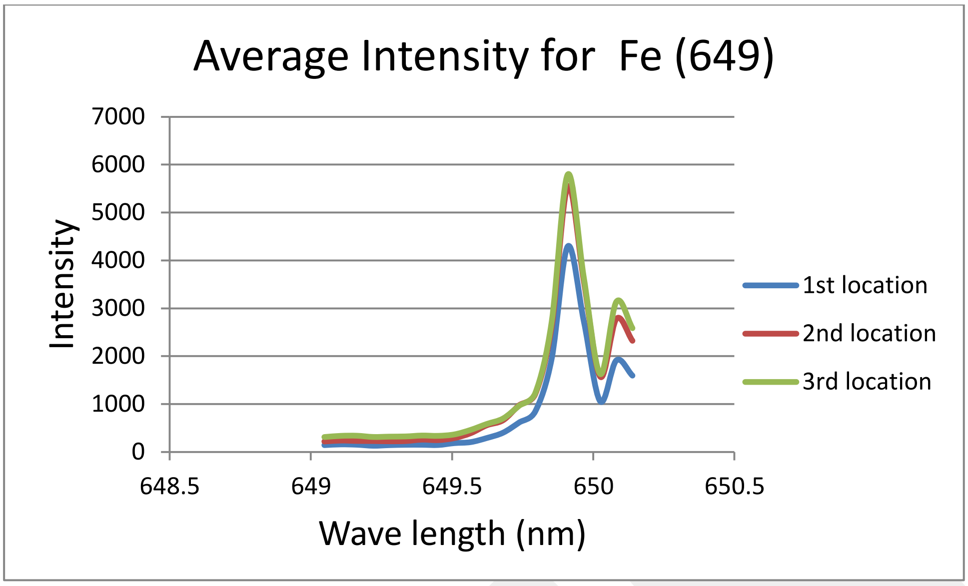

fig (4.202). Average Intensity for Fe (649)

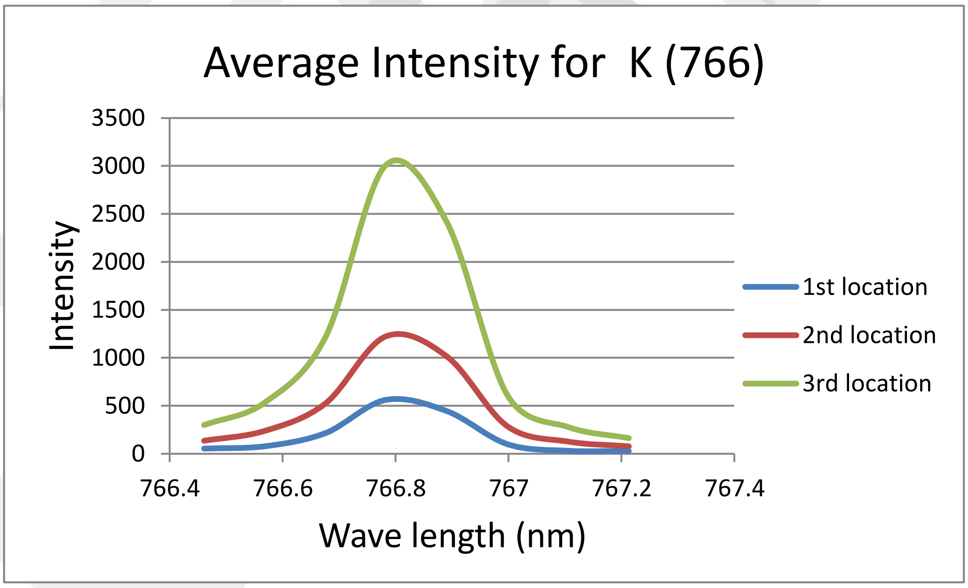

fig (4.203). Average Intensity for K (766)

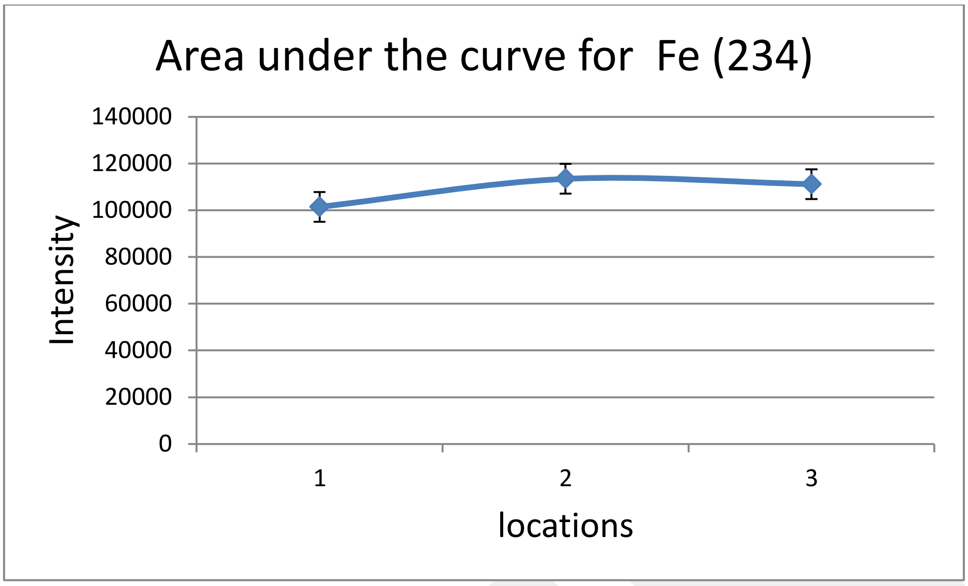

fig (4.204). Area under the curve for Fe (234)

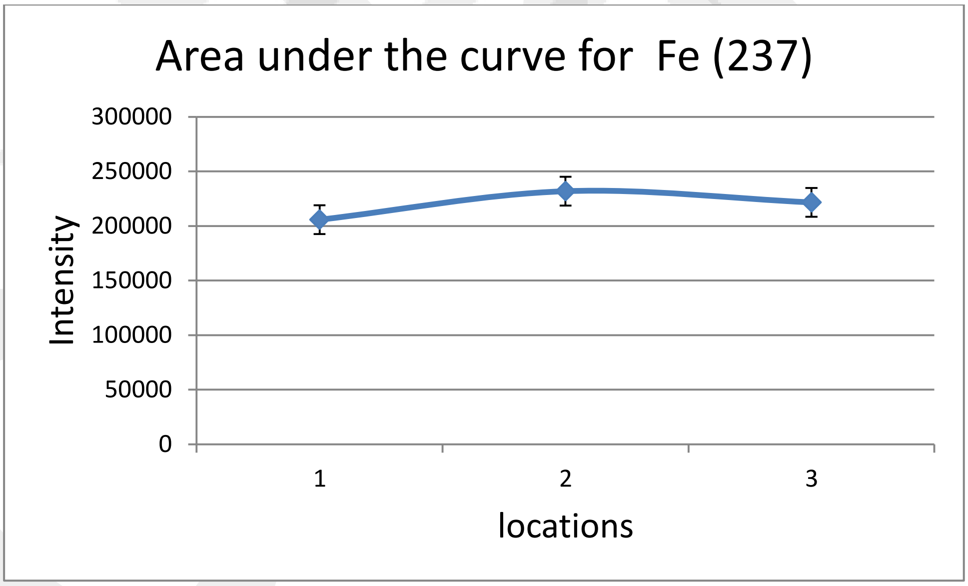

fig (4.205). Area under the curve for Fe (237)

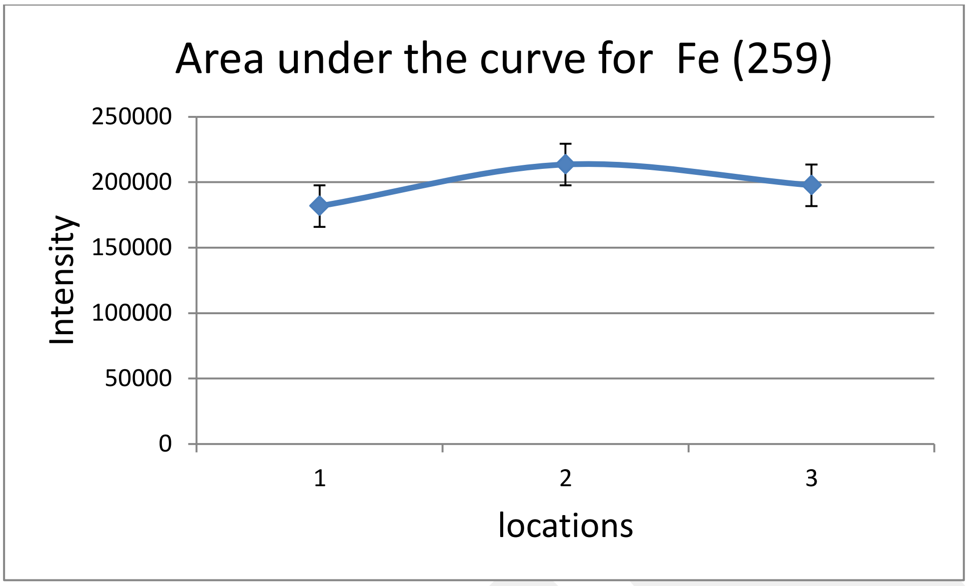

fig (4.206). Area under the curve for Fe (259)

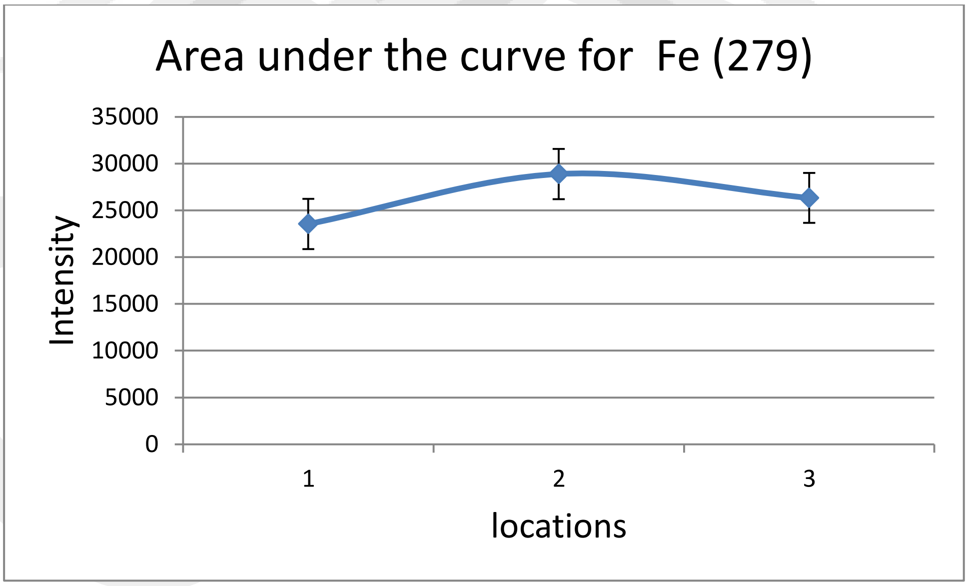

fig (4.207). Area under the curve for Fe (279)

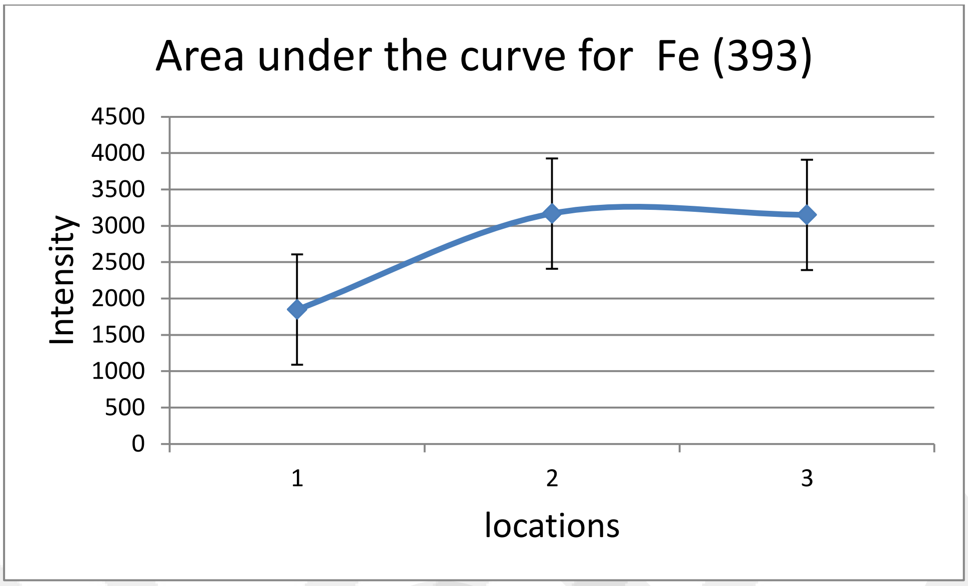

fig (4.208). Area under the curve for Fe (393)

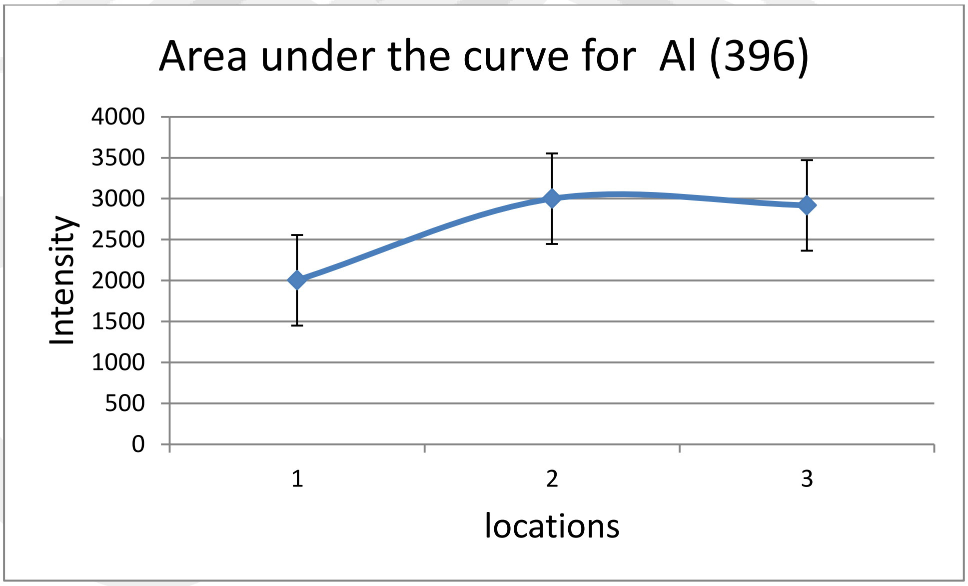

fig (4.209). Area under the curve for Al (396)

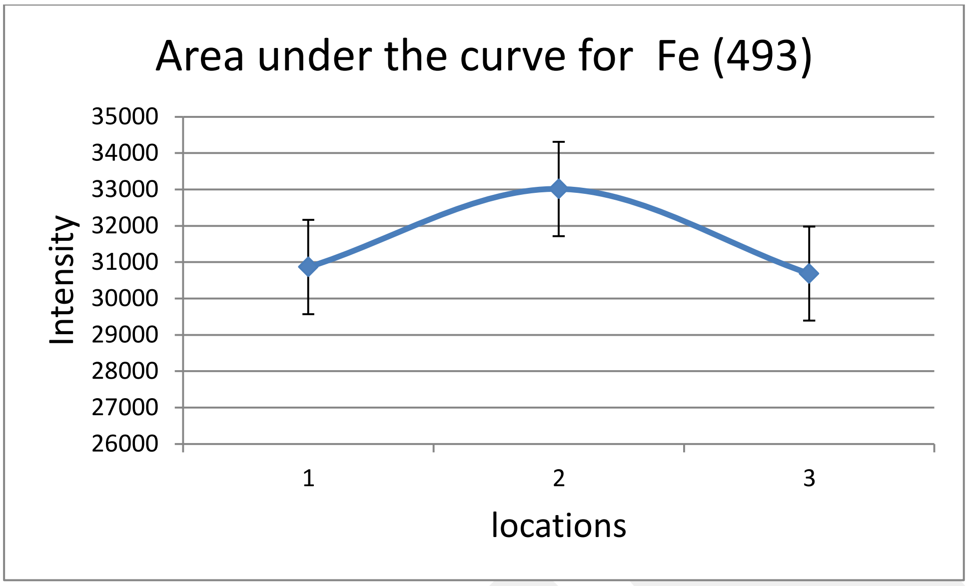

fig (4.210). Area under the curve for Fe (493)

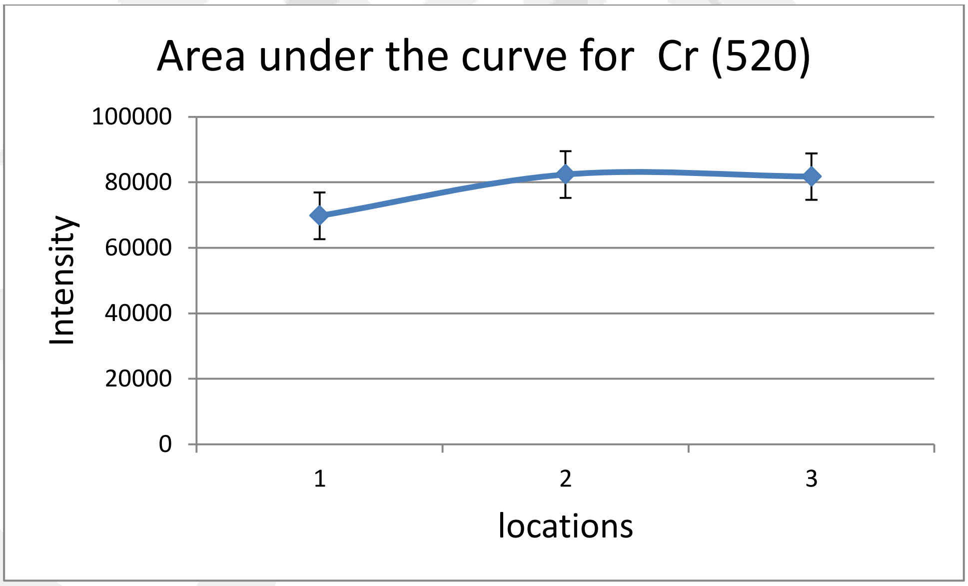

fig (4.211). Area under the curve for Cr (520)

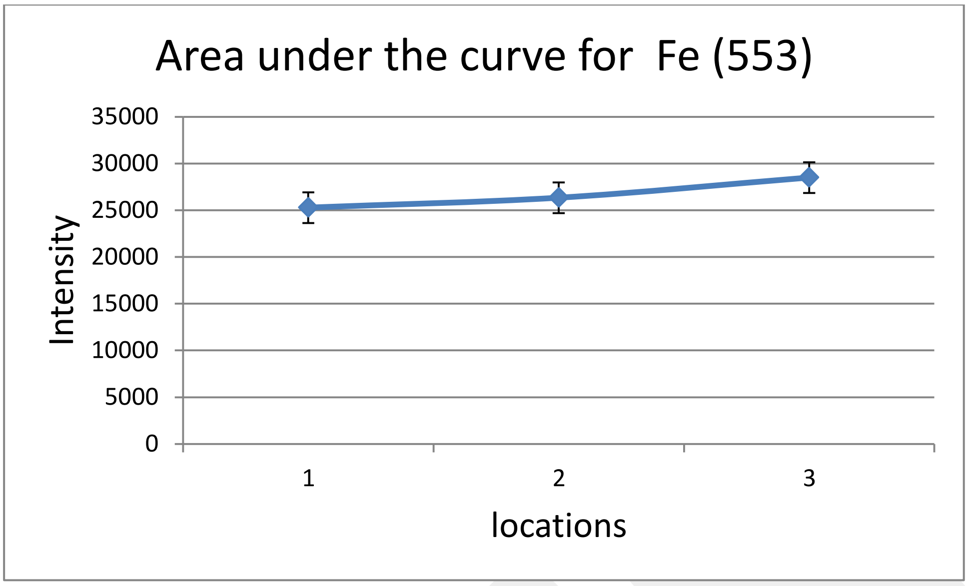

fig (4.212). Area under the curve for Fe (553)

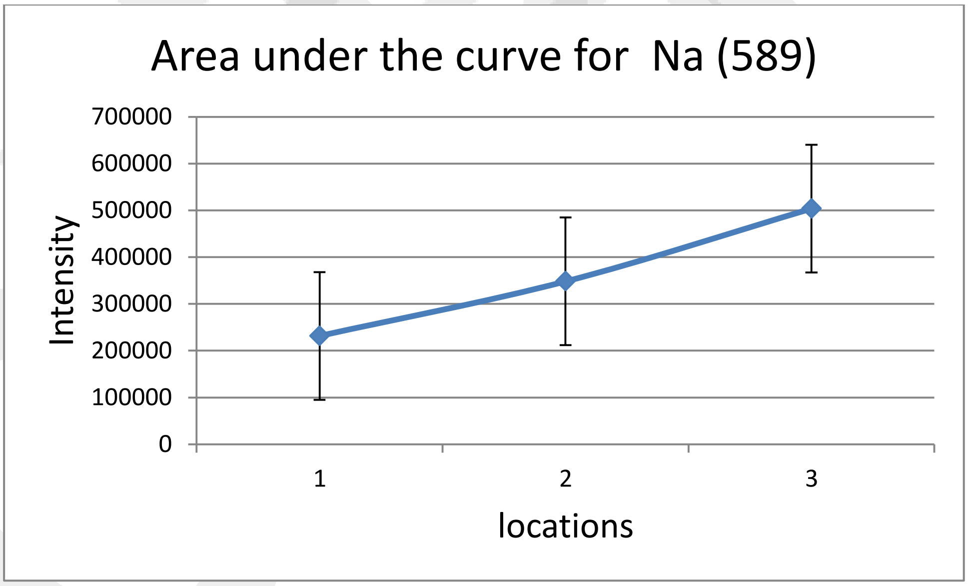

fig (4.213). Area under the curve for Na (589)

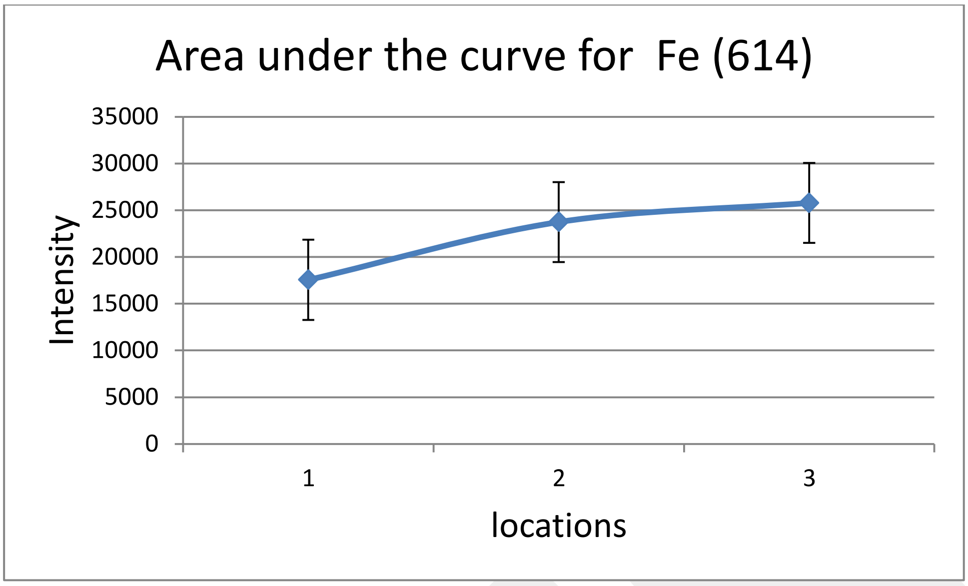

fig (4.214). Area under the curve for Fe (614)

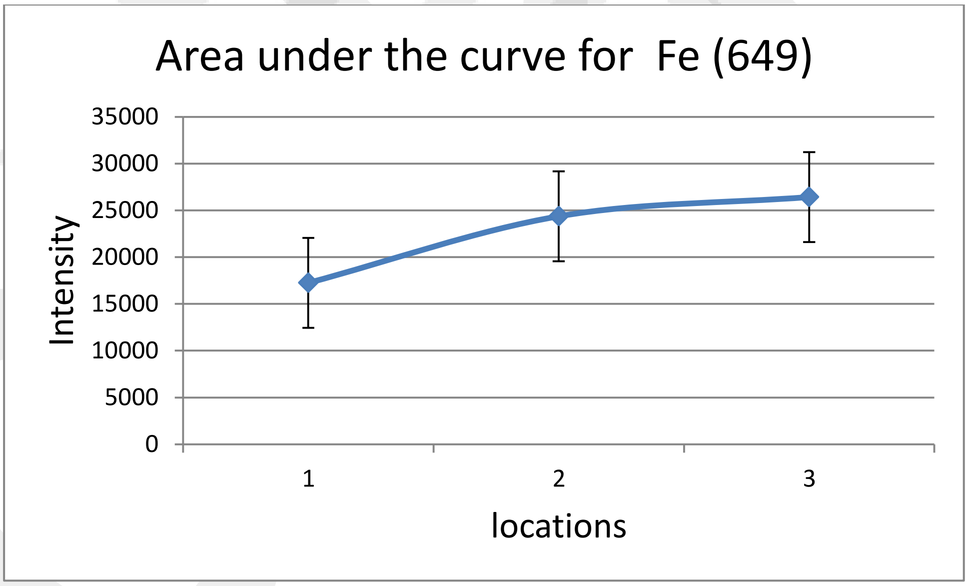

fig (4.215). Area under the curve for Fe (649)

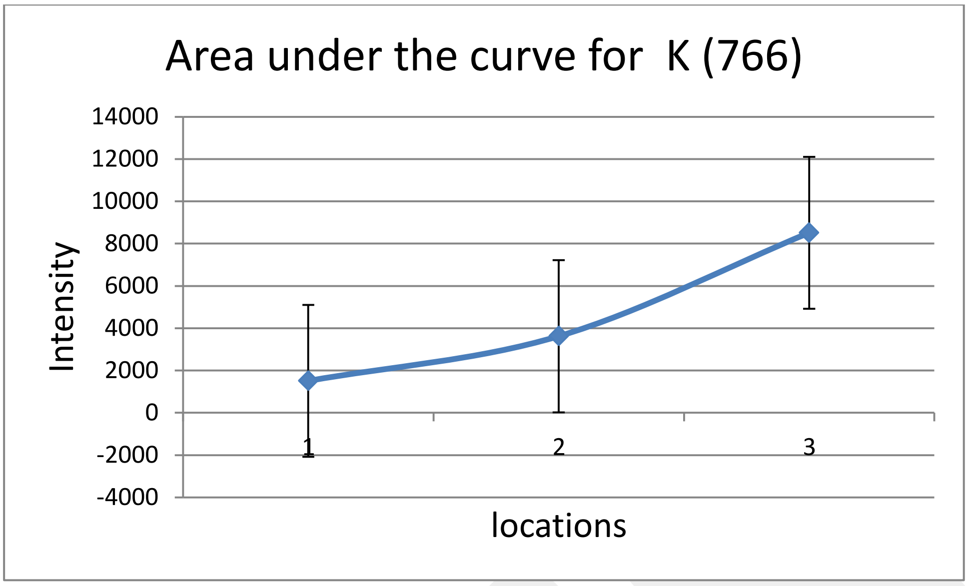

fig (4.216). Area under the curve for K (766)

Finally we used 0,5 µs time delay, and power of 40 mJ, with frequency 10 hz, and 50 shots, then we have moved the alloy in three locations, and we got the following results as shown in the following figures.

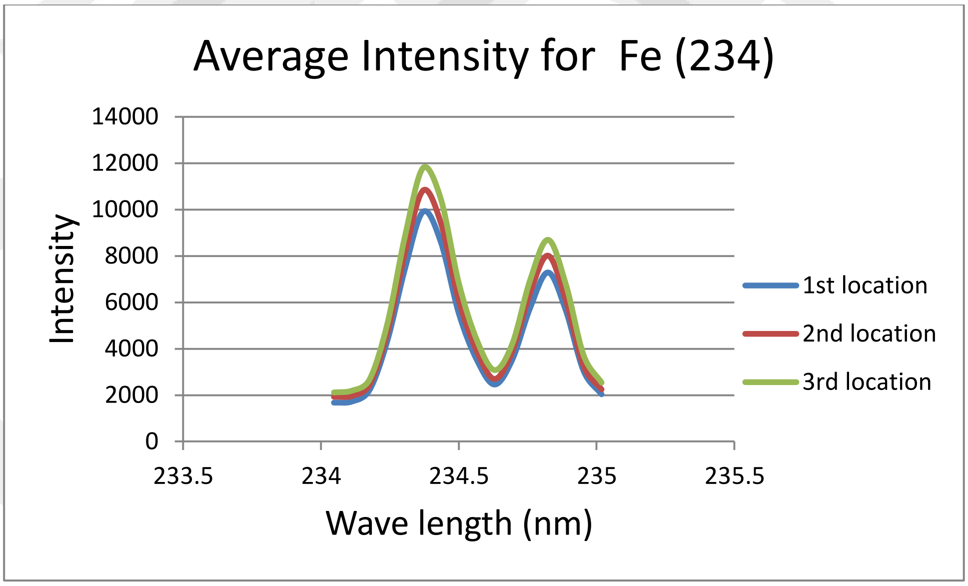

fig (4.217). Average Intensity for Fe (234)

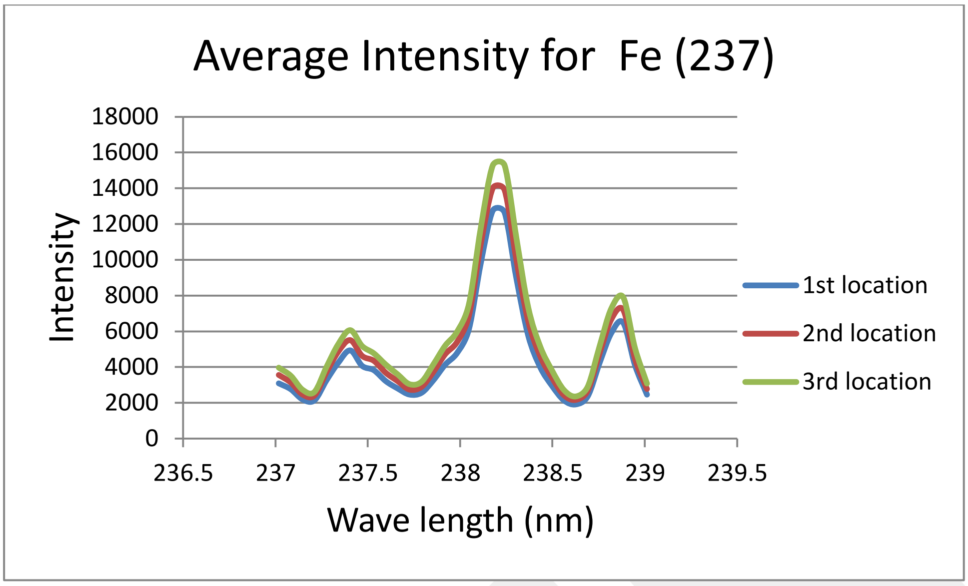

fig (4.218). Average Intensity for Fe (237)

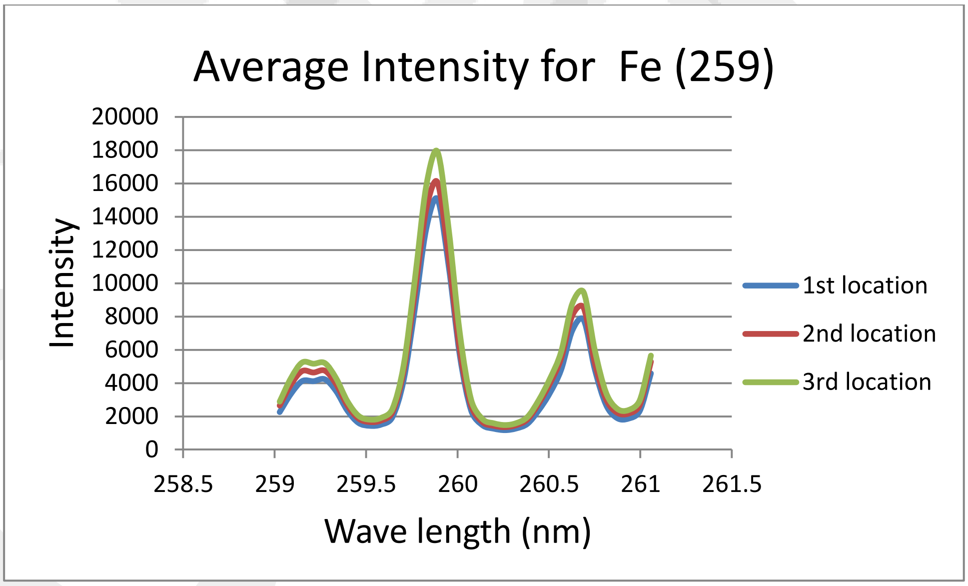

fig (4.219). Average Intensity for Fe (259)

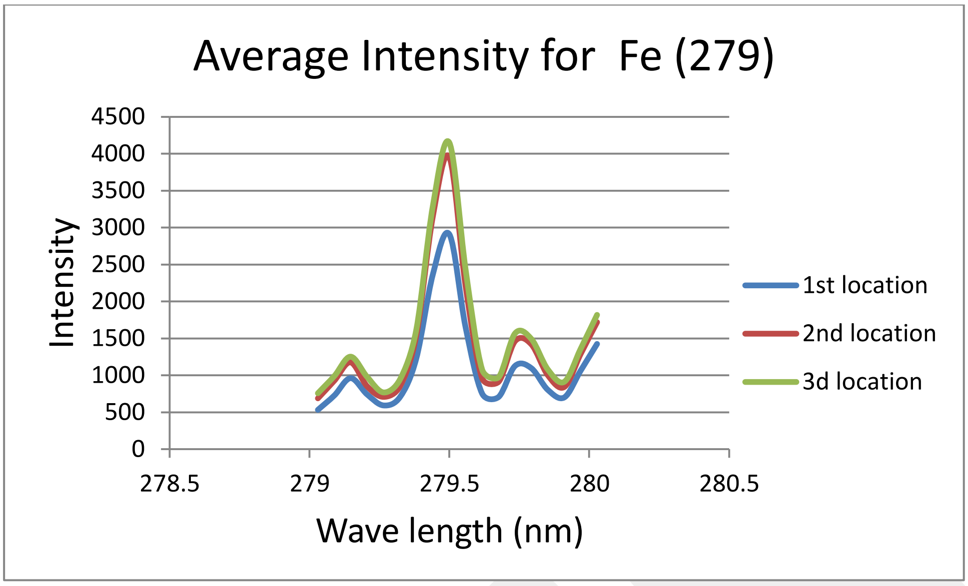

fig (4.220). Average Intensity for Fe (279)

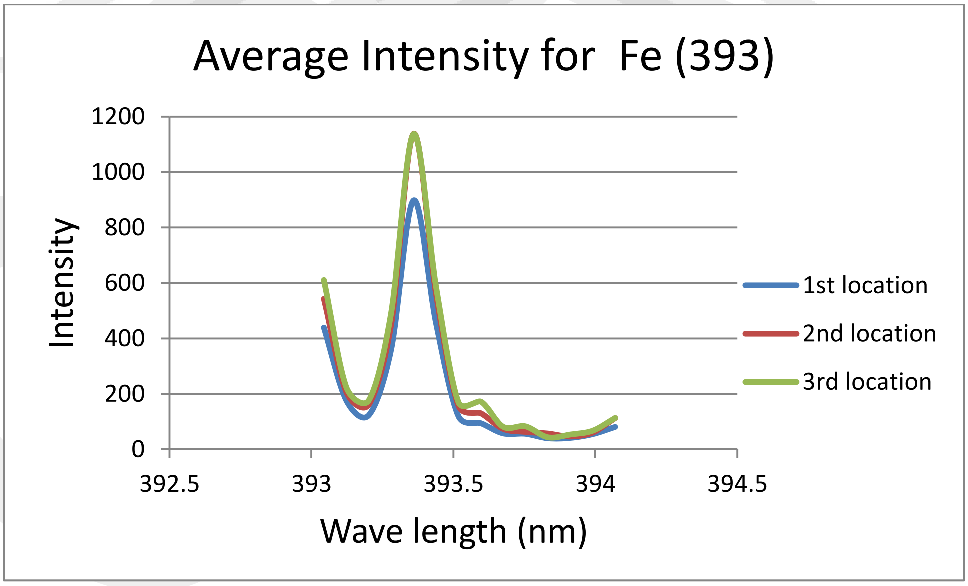

fig (4.221). Average Intensity for Fe (393)

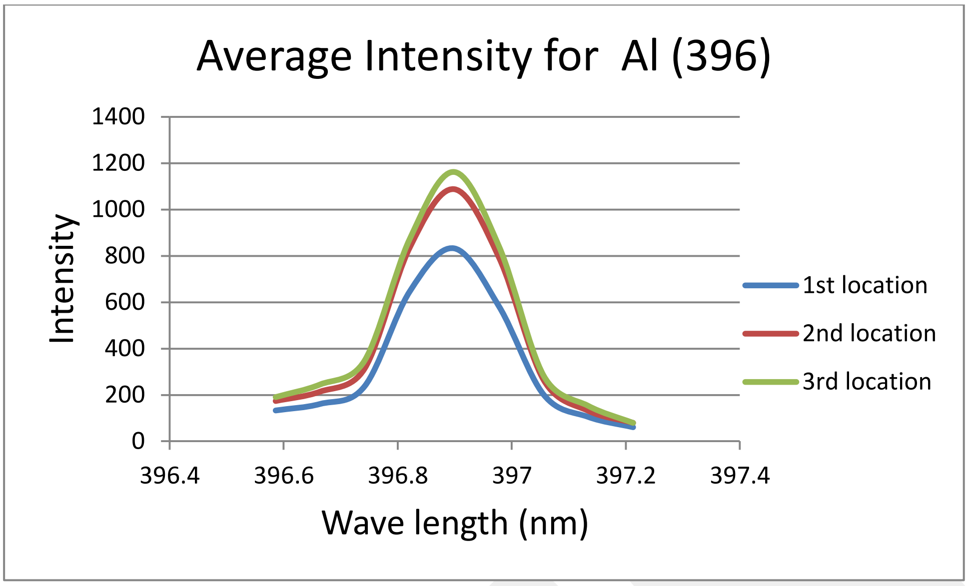

fig (4.222). Average Intensity for Al (396)

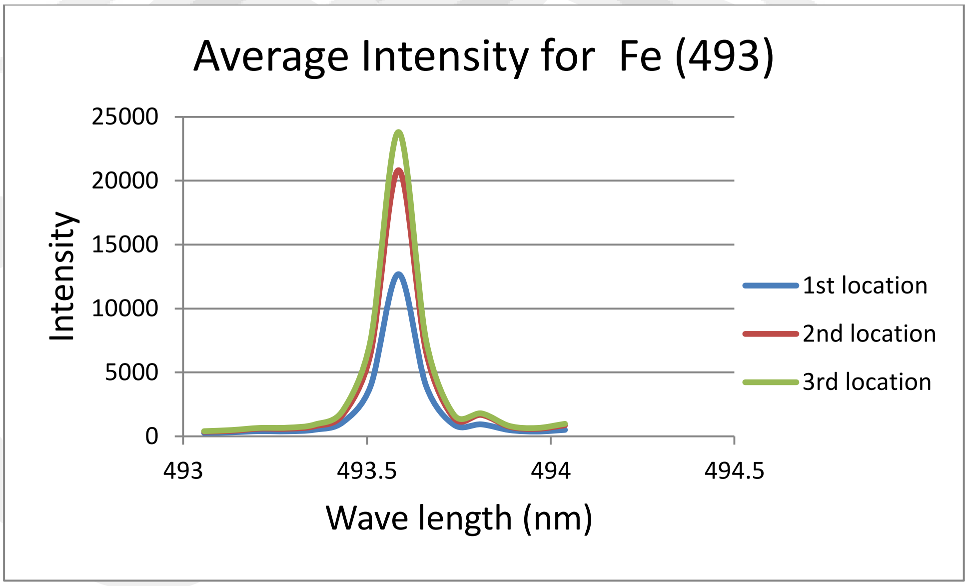

fig (4.223). Average Intensity for Fe (493)

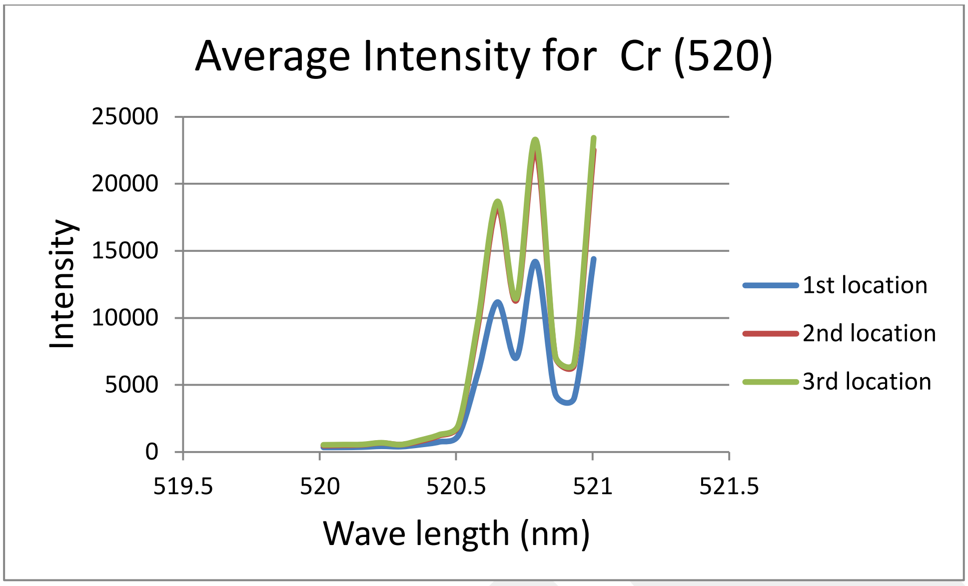

fig (4.224). Average Intensity for Cr (520)

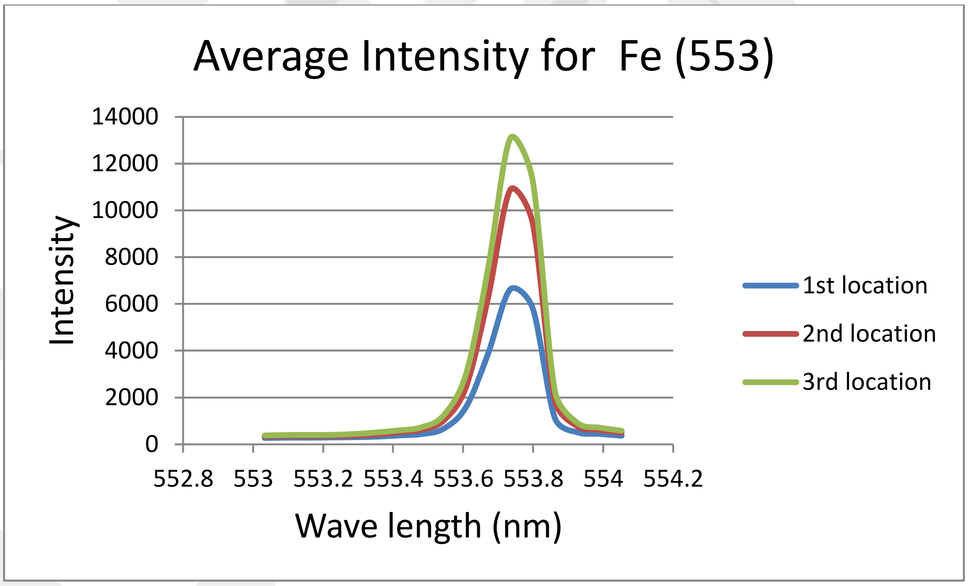

fig (4.225). Average Intensity for Cr (553)

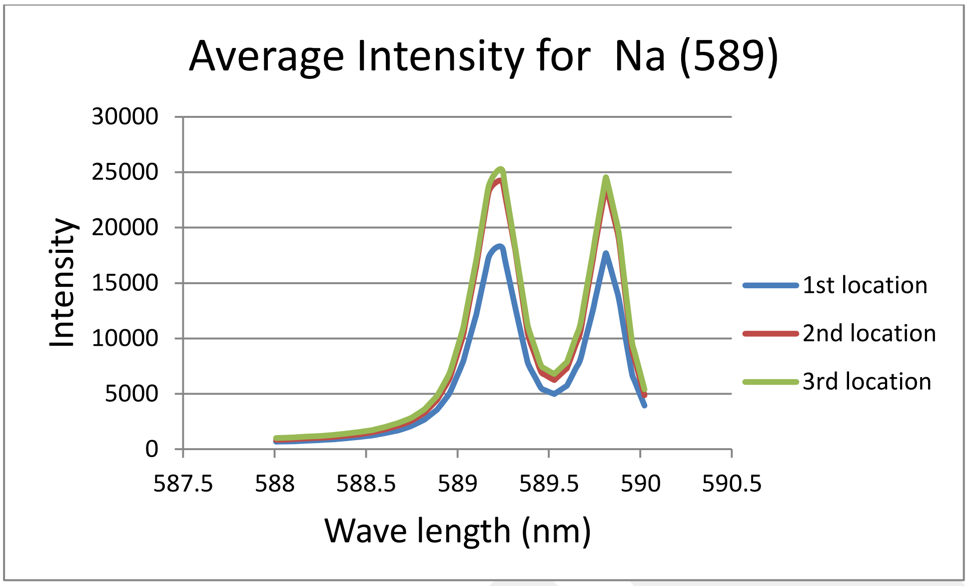

fig (4.226). Average Intensity for Na (589)

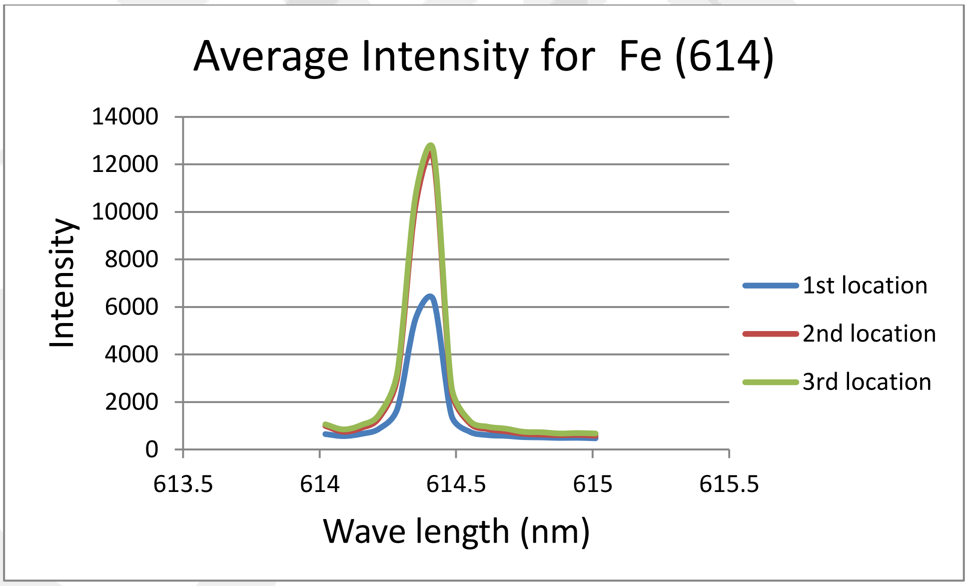

fig (4.227). Average Intensity for Fe (614)

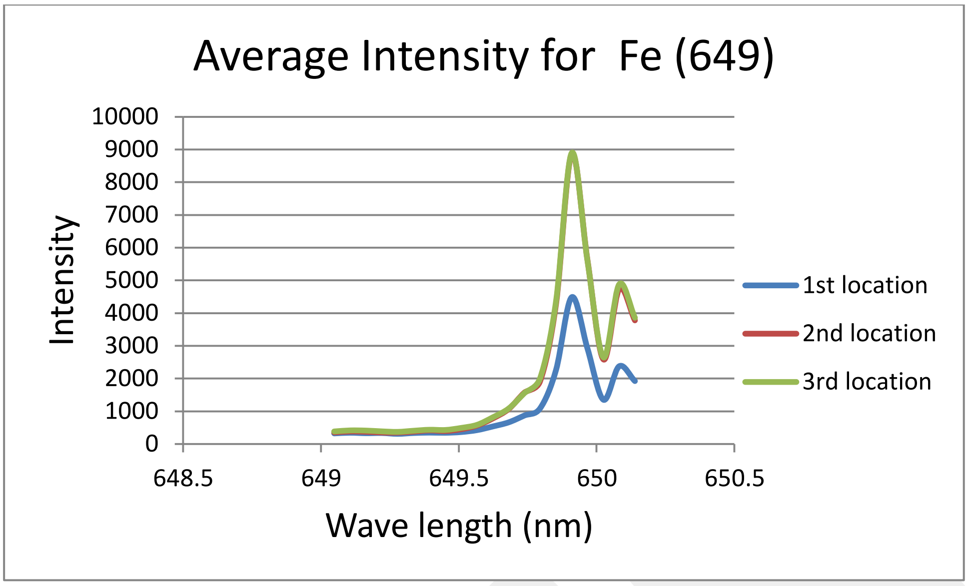

fig (4.228). Average Intensity for Fe (649)

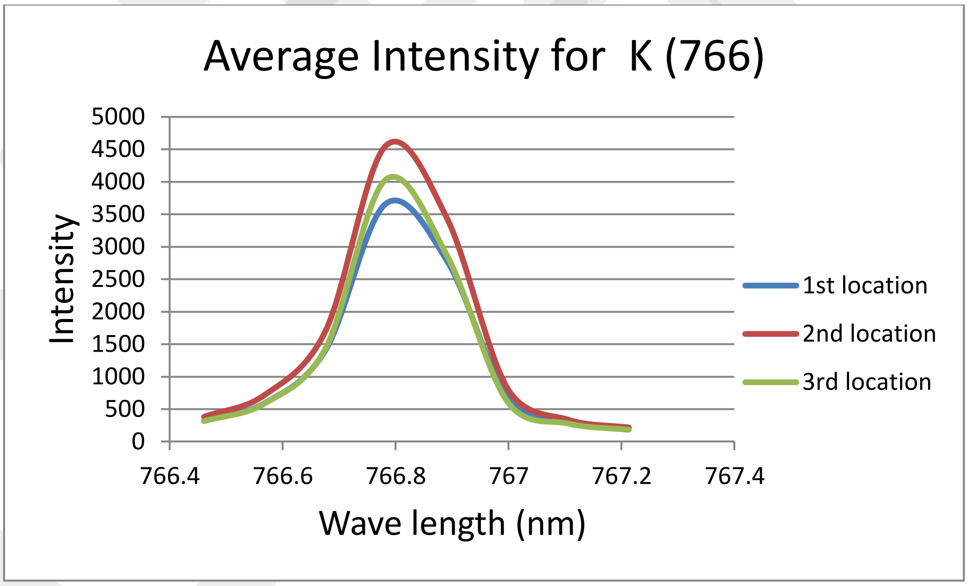

fig (4.229). Average Intensity for K (766)

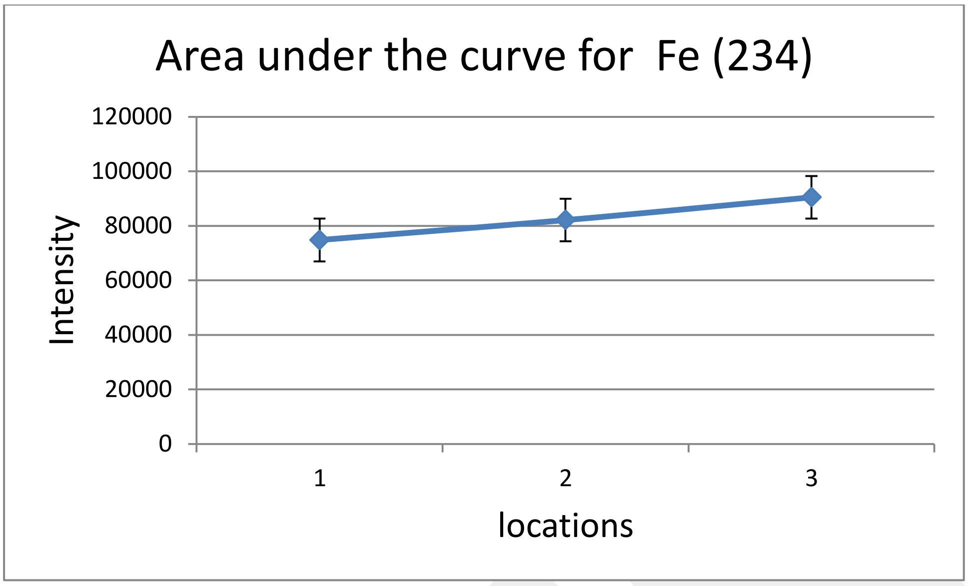

fig (4.230). Area under the curve for Fe (234)

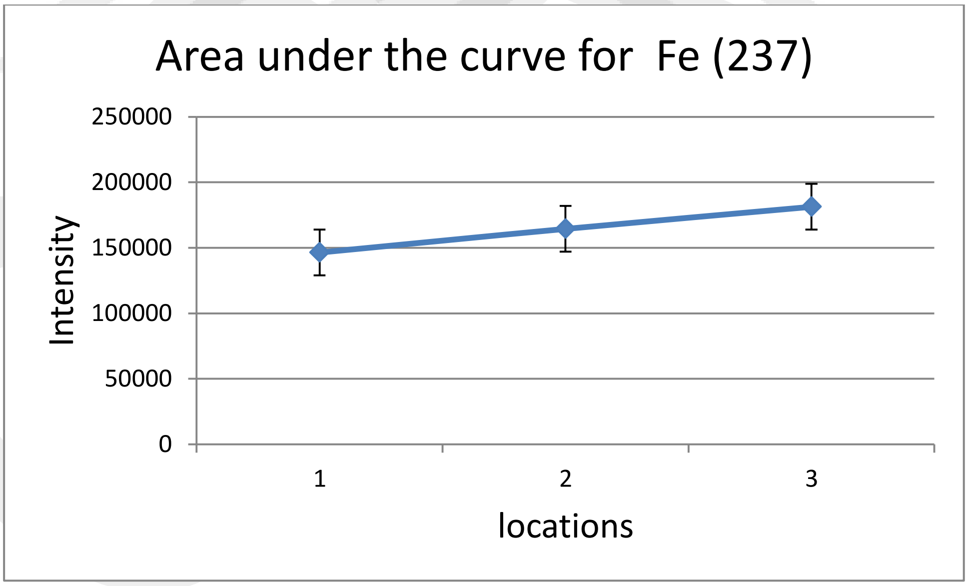

fig (4.231). Area under the curve for Fe (237)

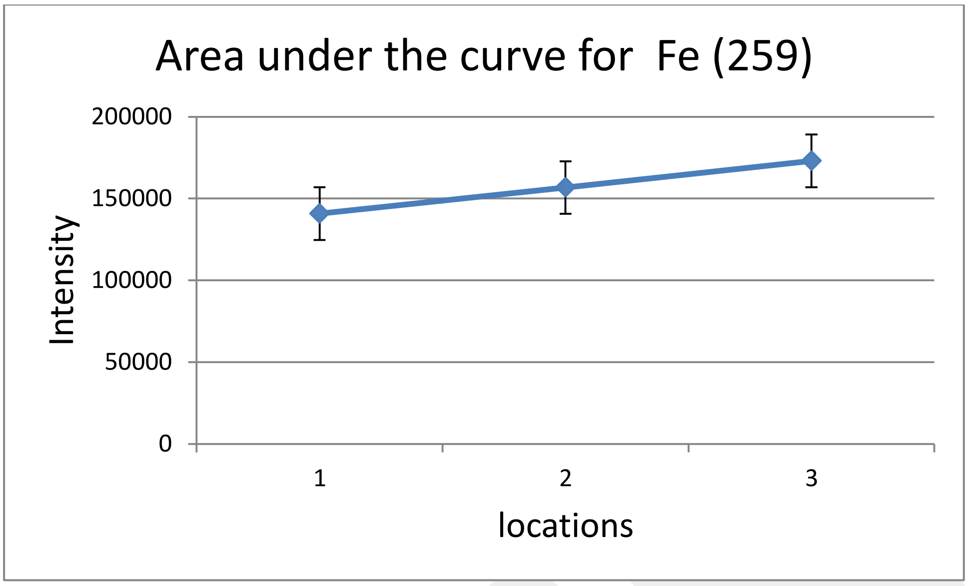

fig (4.232). Area under the curve for Fe (259)

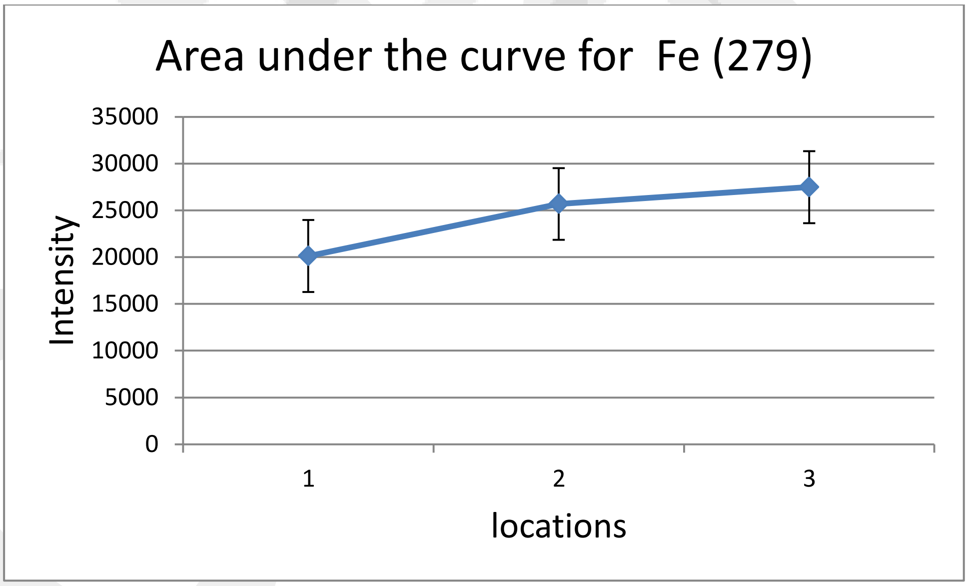

fig (4.233). Area under the curve for Fe (279)

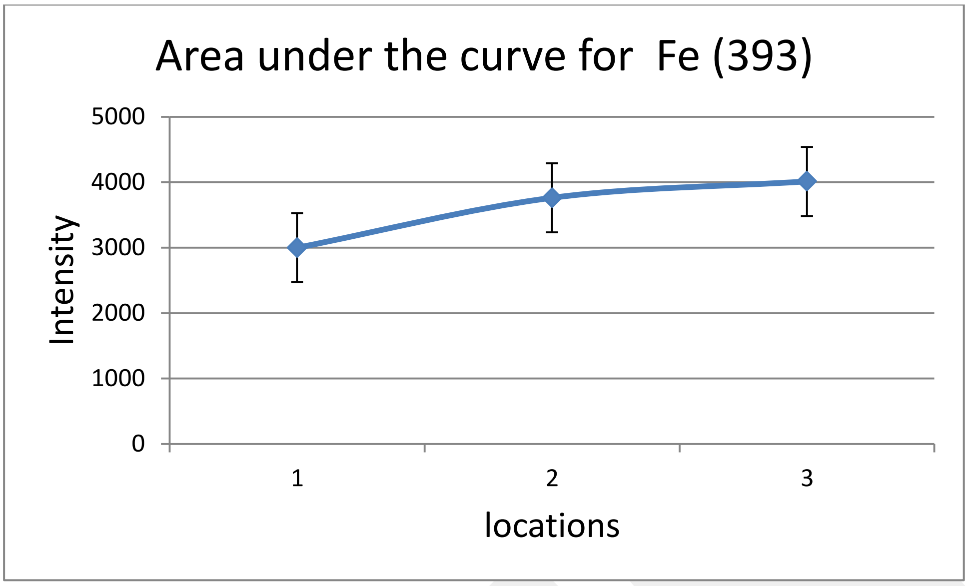

fig (4.234). Area under the curve for Fe (393)

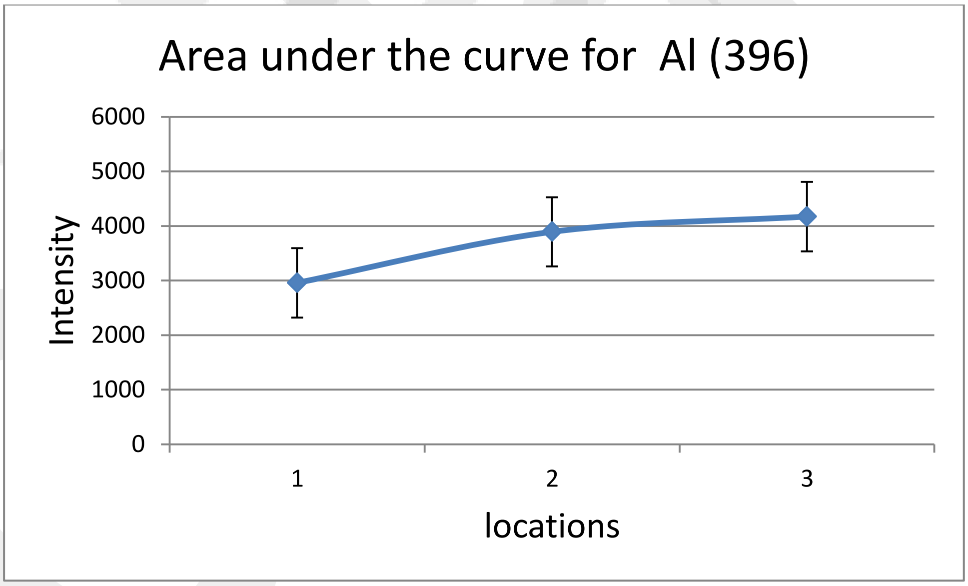

fig (4.235). Area under the curve for Al (396)

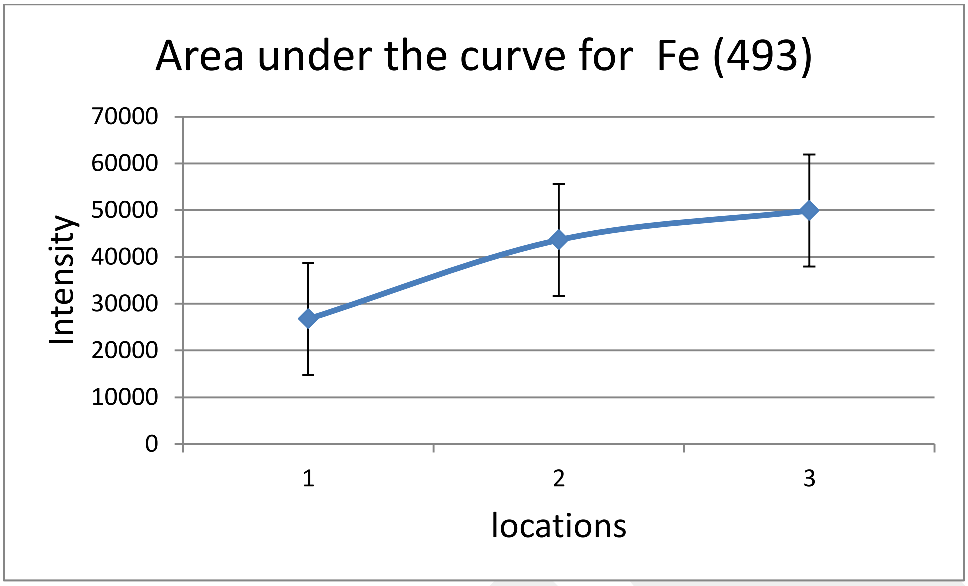

fig (4.236). Area under the curve for Fe (493)

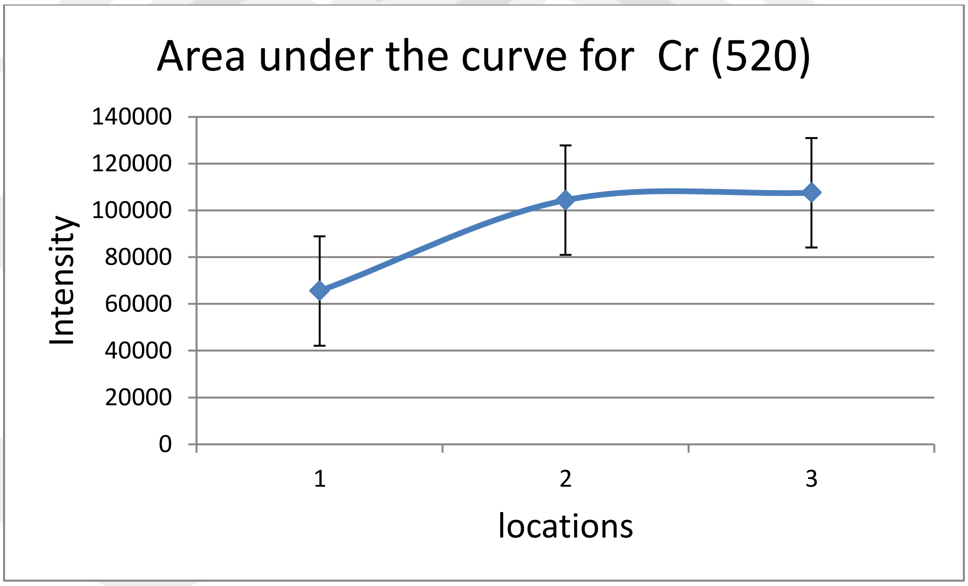

fig (4.237). Area under the curve for Cr (520)

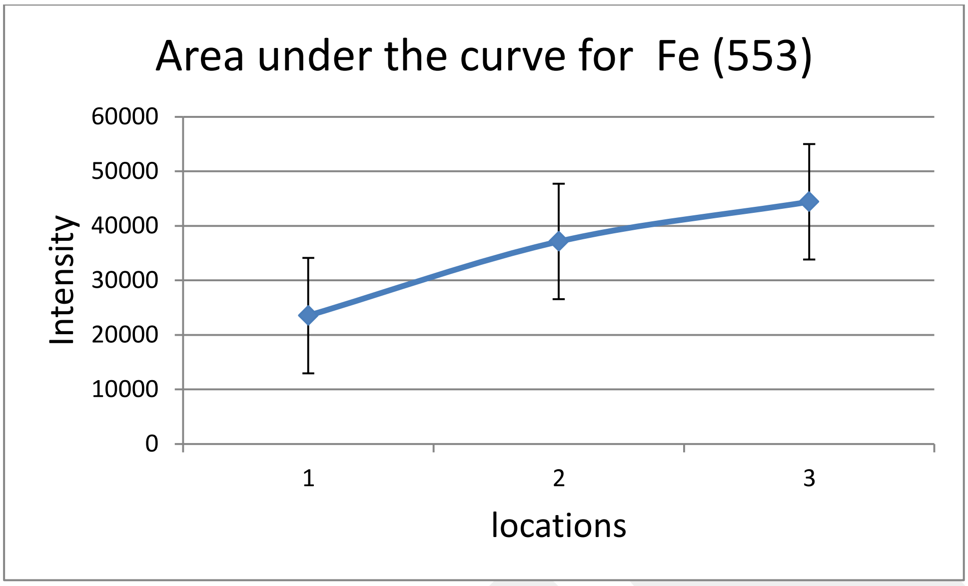

fig (4.238). Area under the curve for Fe (553)

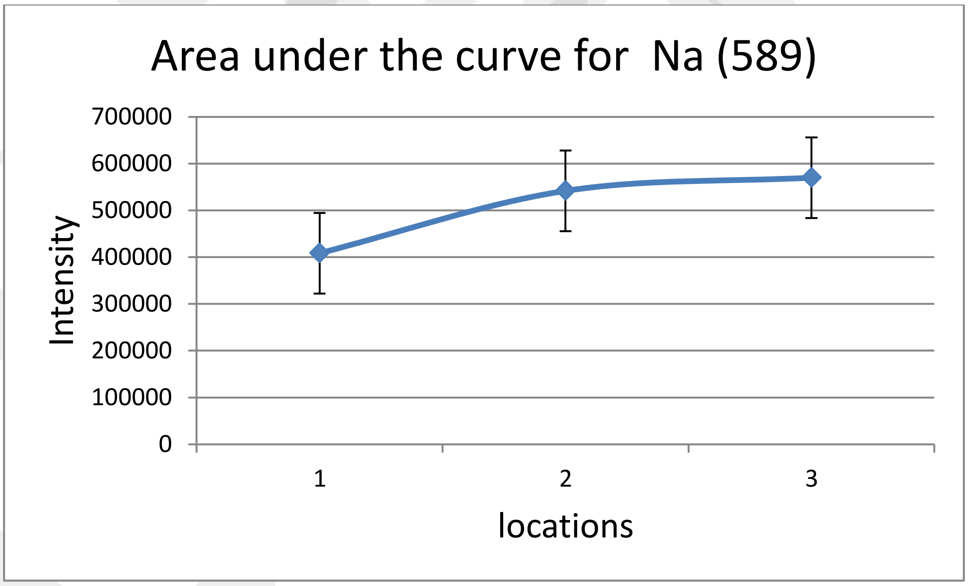

fig (4.239). Area under the curve for Na (589)

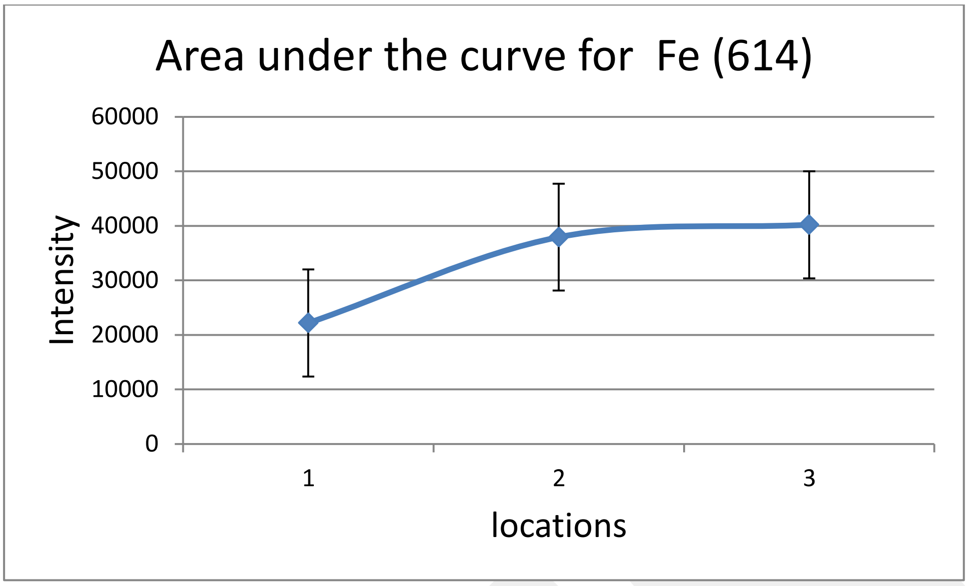

fig (4.240). Area under the curve for Fe (614)

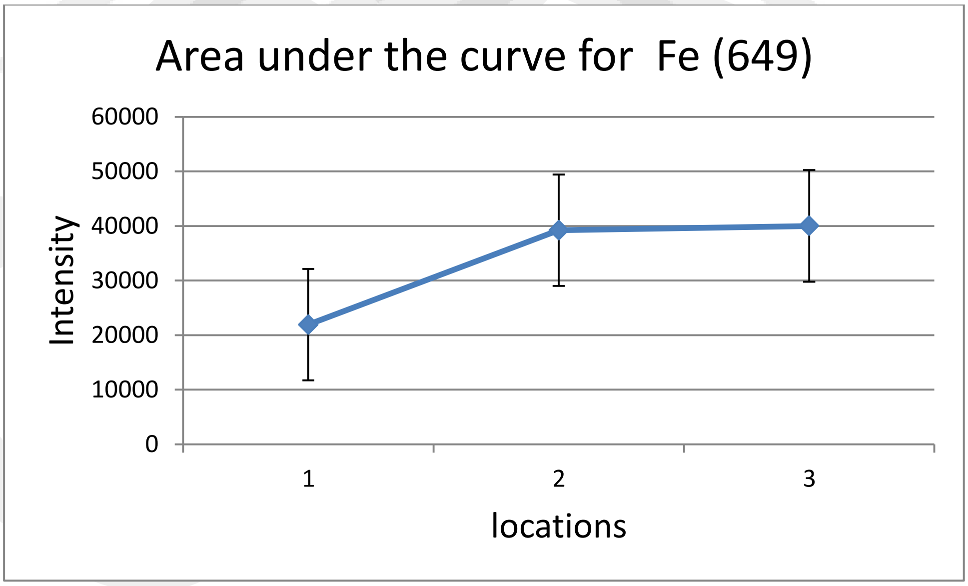

fig (4.241). Area under the curve for Fe (649)

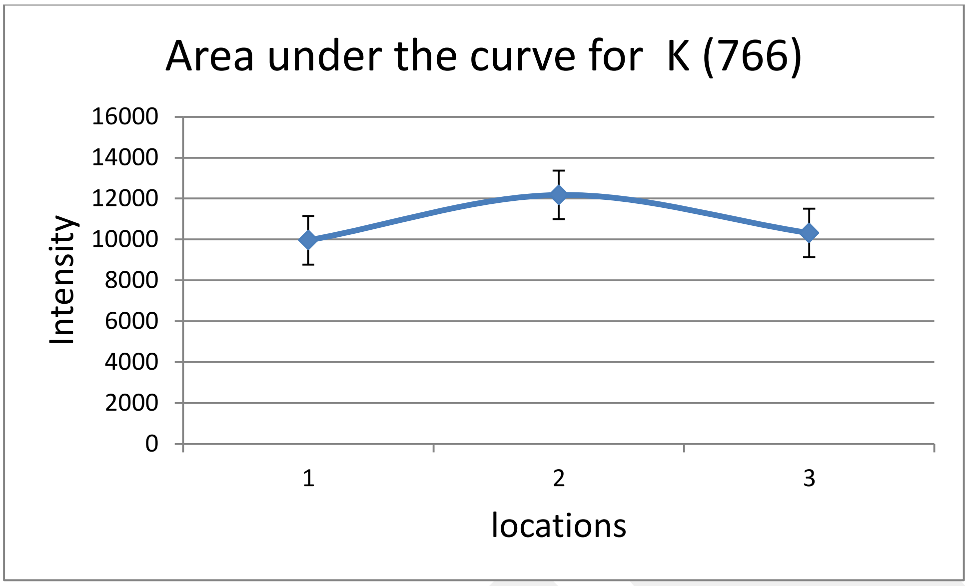

fig (4.242). Area under the curve for K (766)

We did a comparative with Average of different locations in different energies and we got the following results as shown in the following figures.

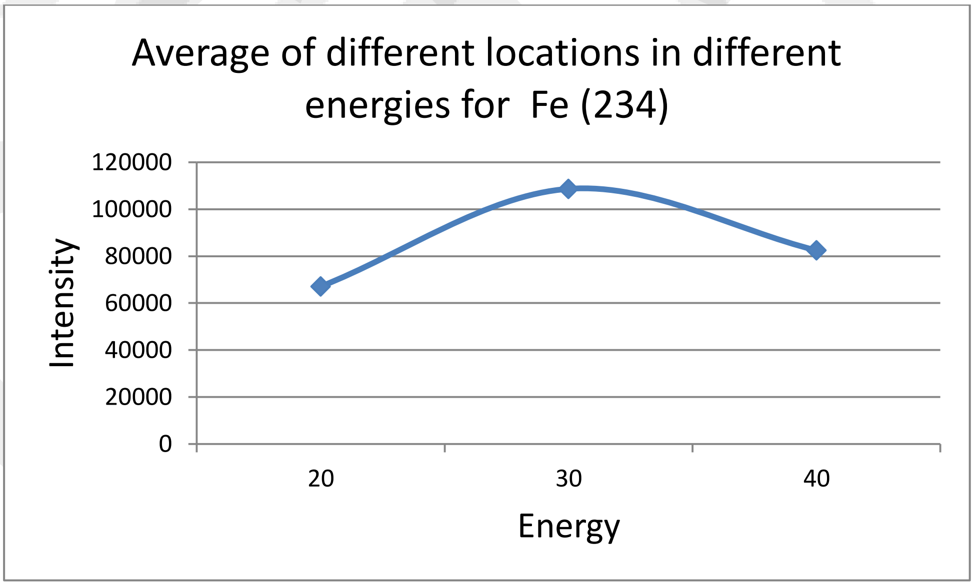

fig (4.243). Average of different locations in different energies for Fe (234)

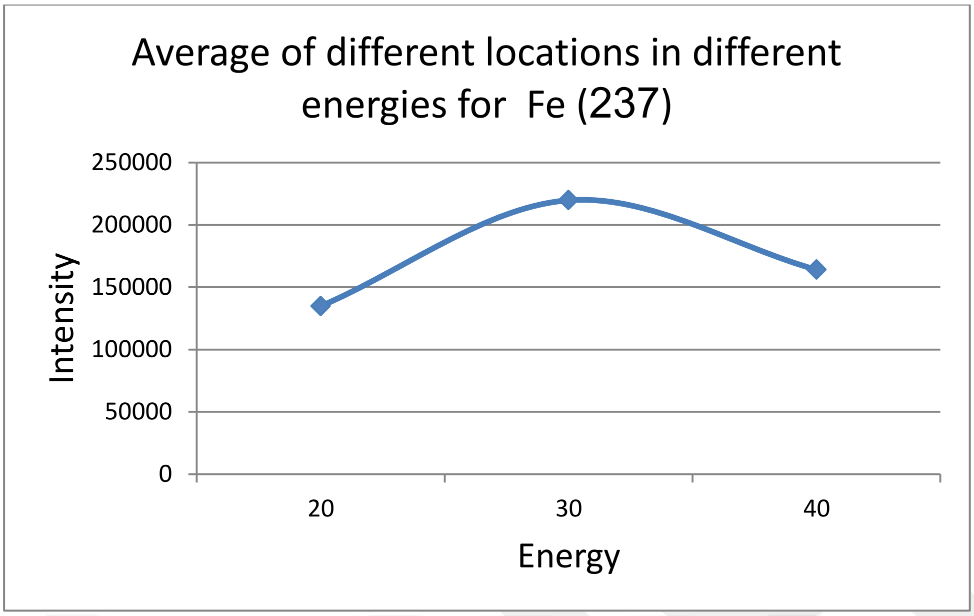

fig (4.244). Average of different locations in different energies for Fe (237)

We took the summation of averages in three locations for the power of 20 mJ, and the same for 30 and 40 mJ, finally we used ‘‘line chart’’ to get average of different locations in different energies as shown in the following figures.

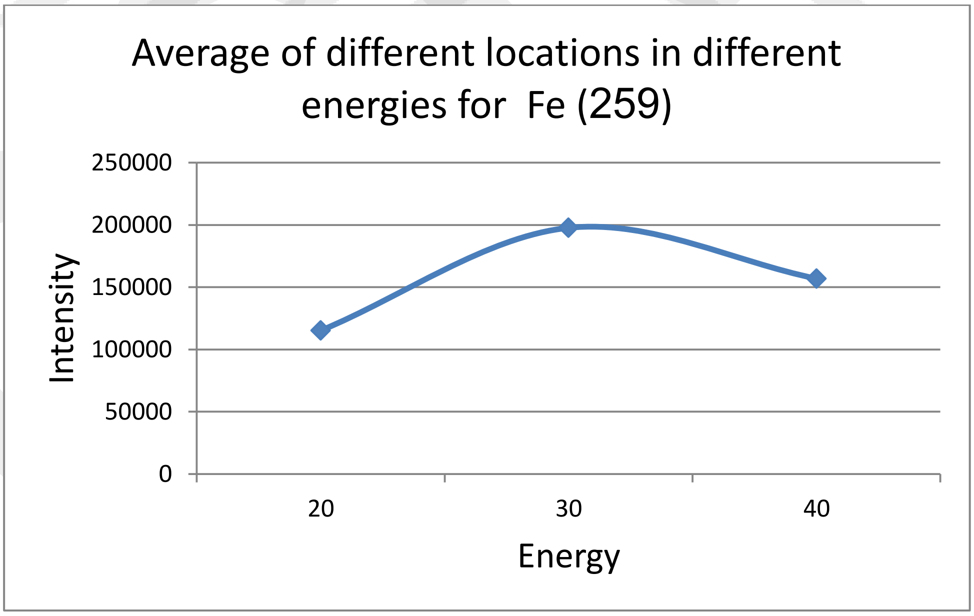

fig (4.245). Average of different locations in different energies for Fe (259)

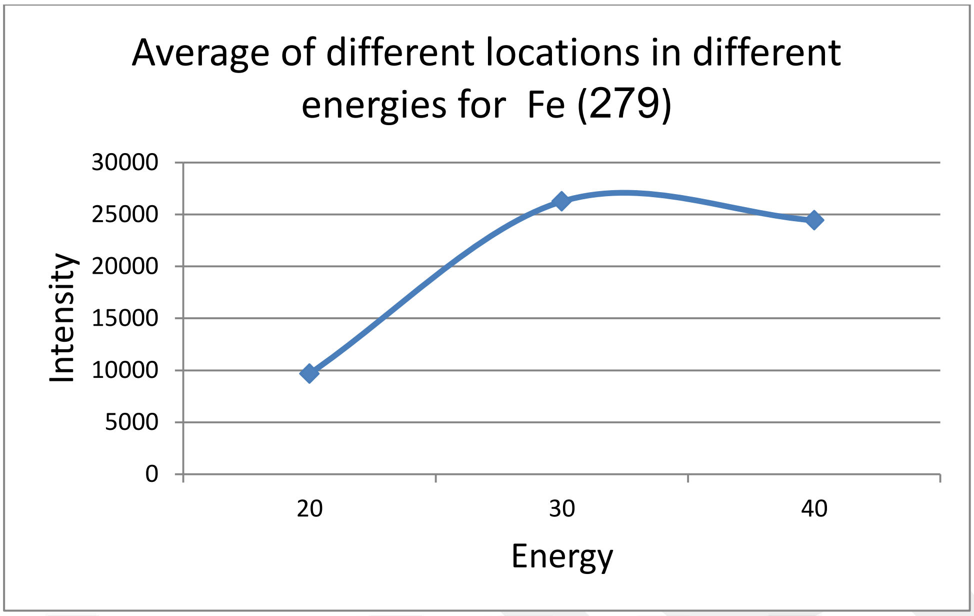

fig (4.246). Average of different locations in different energies for Fe (279)

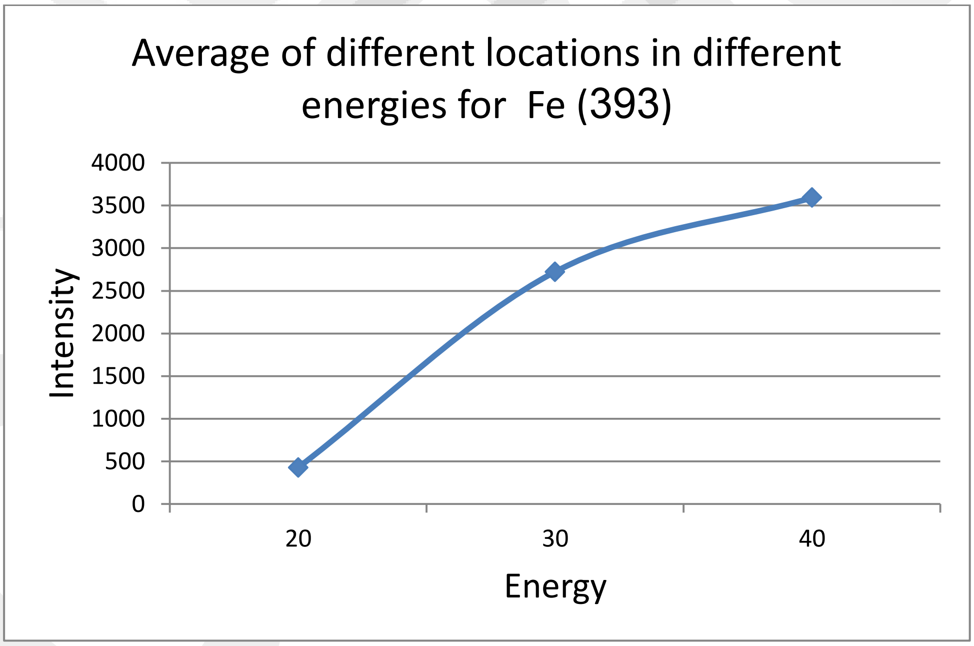

fig (4.247). Average of different locations in different energies for Fe (393)

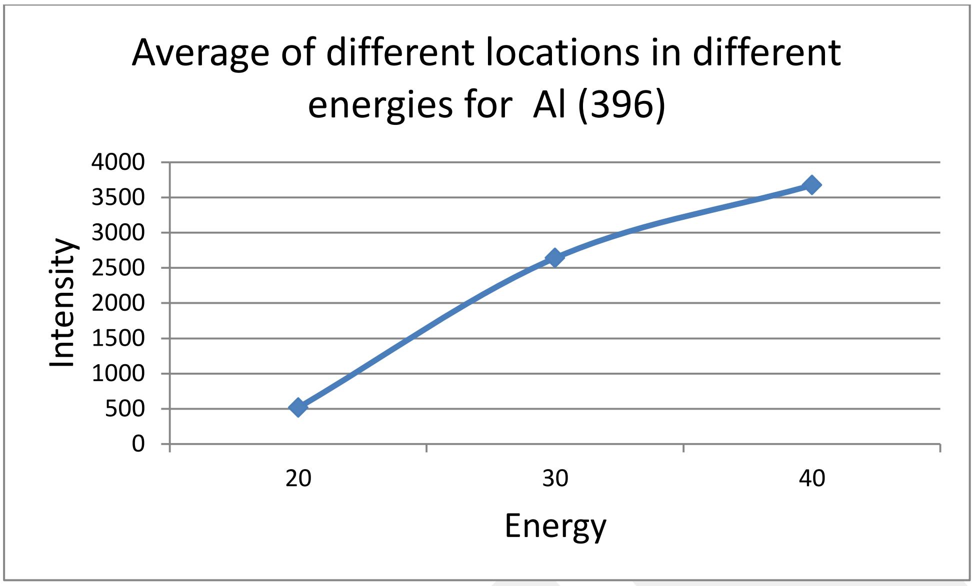

fig (4.248). Average of different locations in different energies for Al (396)

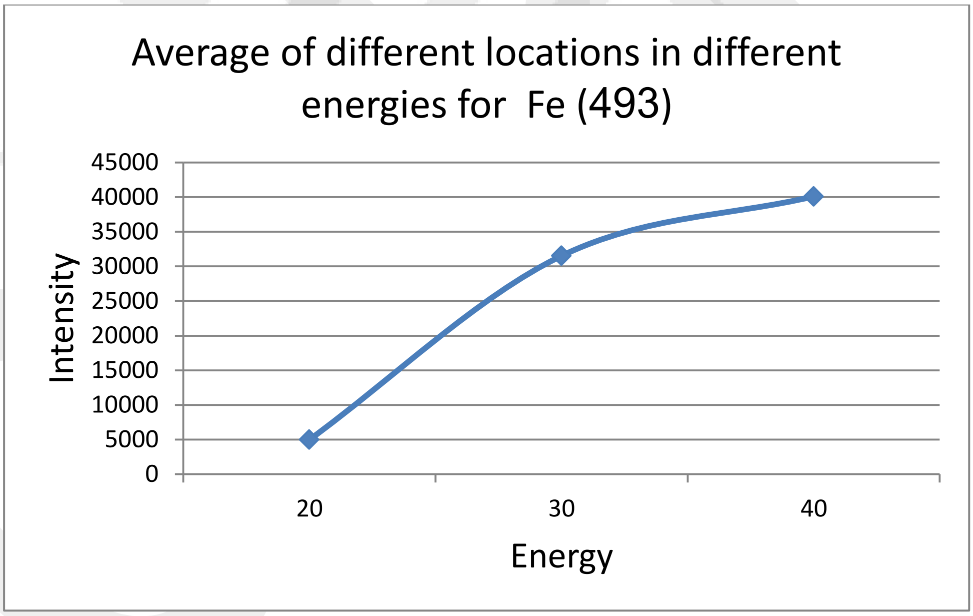

fig (4.249). Average of different locations in different energies for Fe (493)

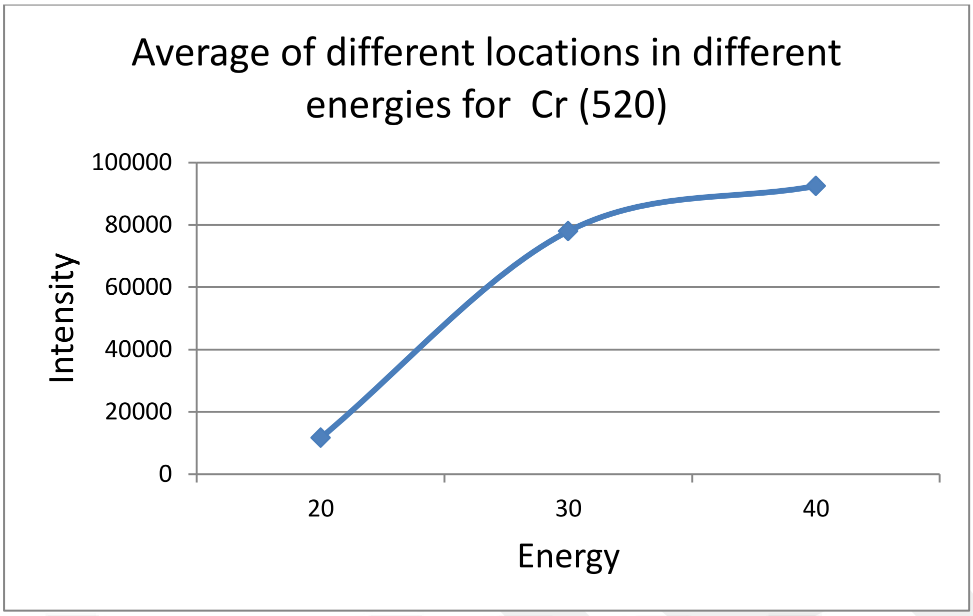

fig (4.250). Average of different locations in different energies for Cr (520)

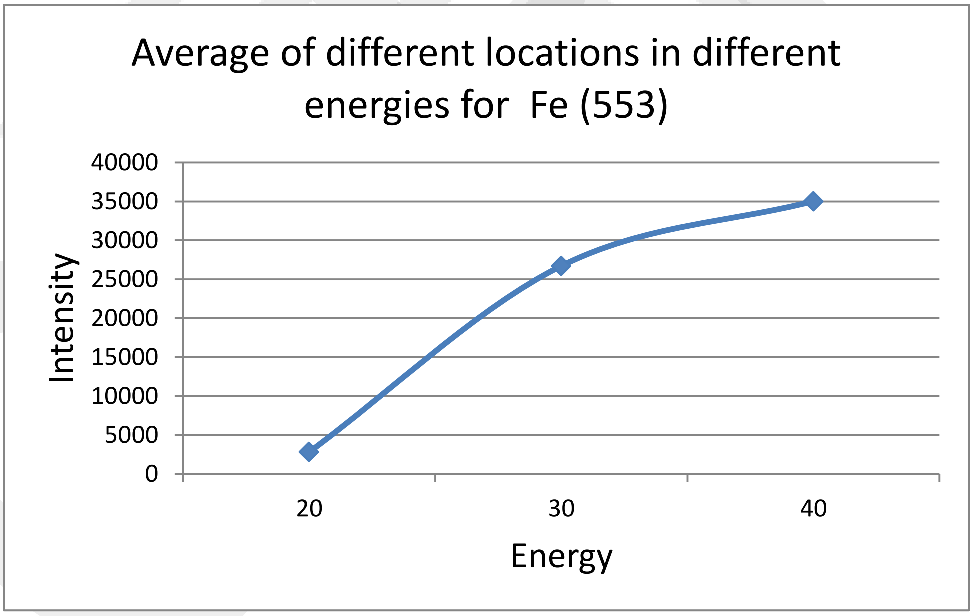

fig (4.251). Average of different locations in different energies for Fe (553)

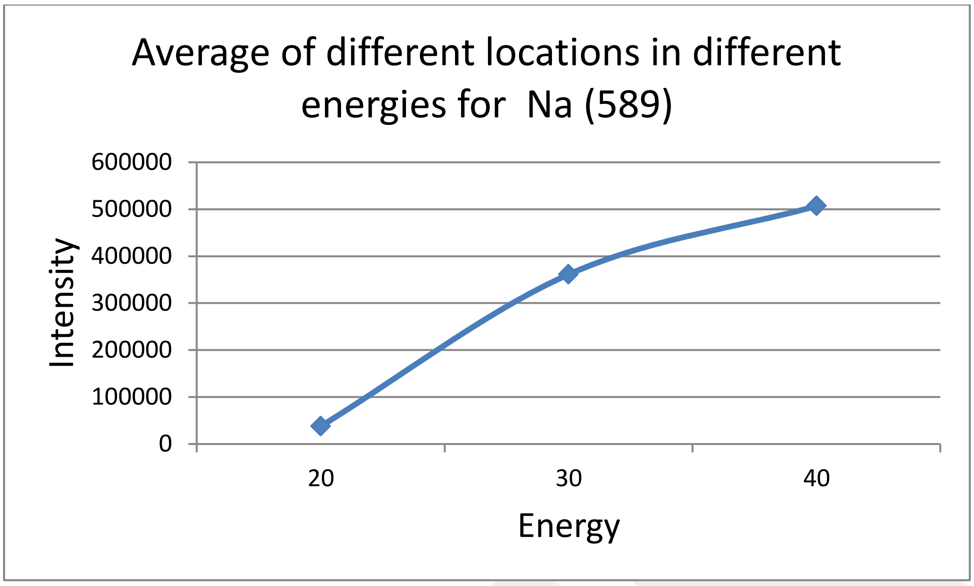

fig (4.252). Average of different locations in different energies for Na (589)

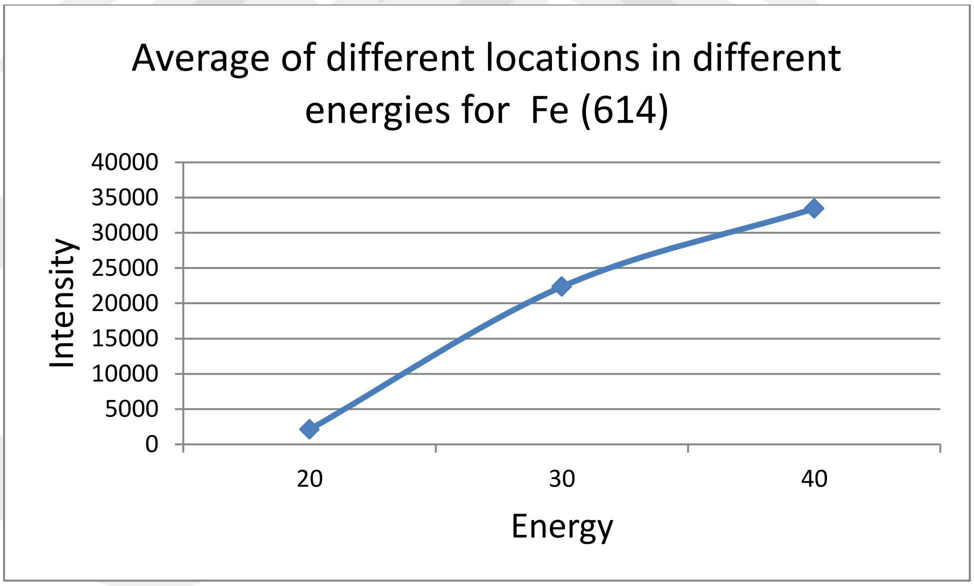

fig (4.253). Average of different locations in different energies for Fe (614)

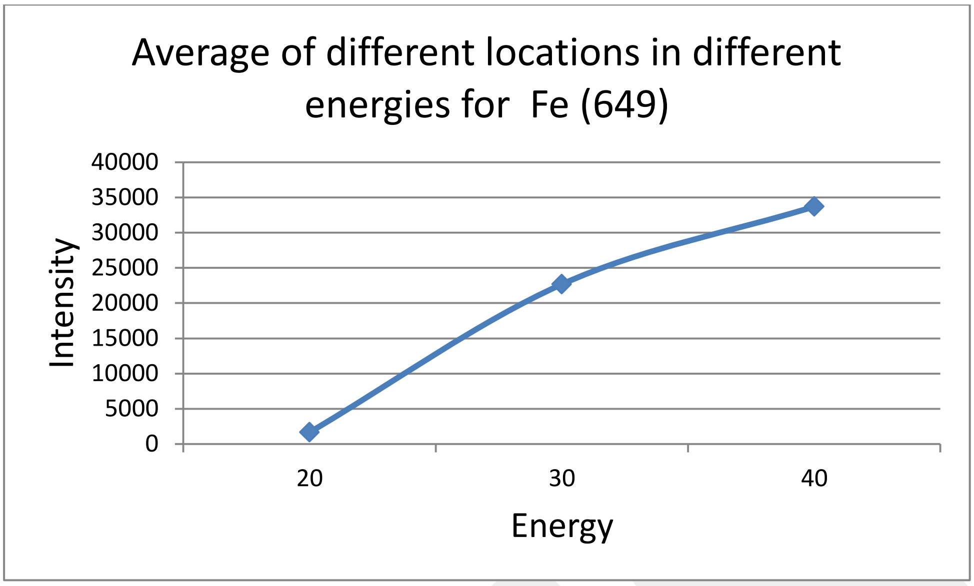

fig (4.254). Average of different locations in different energies for Fe (649)

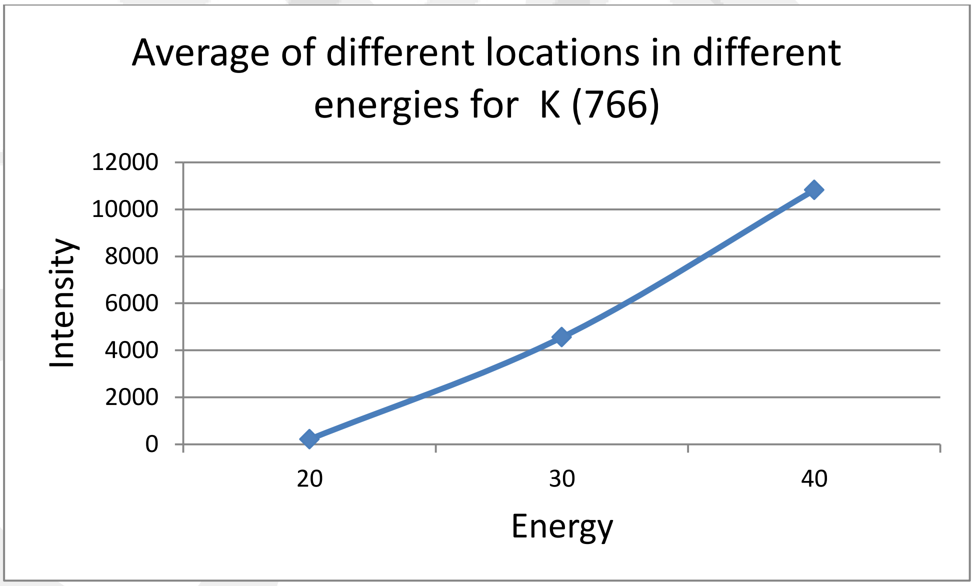

fig (4.255). Average of different locations in different energies for K (766)

To get the signal to noise ratio, first we choose the lowest values for the spectrum, next take the function ‘‘STDEV’’ of them which is the noise, after that we do the function S/N ratio, where S is the peak of the spectrum.
For example we take the signal to noise ratio for the elements Fe (234) and Al (396).

S/N for Fe (234) = 11959.06 / 1622.85401498923 = 7.36915328769073.

S/N for Al (396) = 487.64 / 55.166005232691 = 8.83950175371814.

CHAPTER FIVE

CONCLUSION

LIBS was used in this research for Detection of Trace Elements in Metals**.** The set up for LIBS was done by analyzing an alloy to do the experiment. After we have done all the steps, we got the results and detect the elements in this alloy. The elements were Iron (Fe) (about 70 % of this alloy), and the other elements were Chromium (Cr), Sodium (Na), Aluminum (Al), and Potassium (K).

We noticed that we got different data when we used three delay times 0.1, 0.3 and 0.5 µs, also 20, 30 and 40 mJ of power. Finally we moved the alloy in to three locations; the frequency was 10 hz and 50 shots every time. We did all that to see and to get best data and compare the results for each case. We did the calculation by means of average intensity and area under the curve. We found some curves abnormal as result of some elements are not homogeneous. Also we calculated the standard error deviations for area under the curve for all of them. The standard error deviation was different in these curves, it depends on the dispersed of values from the average value or the mean. These calculations were important to analyze the data which we took from data analysis software and calculated by excel to detect the elements.

We took signal to noise ratio for two elements Iron (Fe 234) which was 7.369153, and Aluminum (Al 396) was 8.839502.

In this experiment we found on two elements potassium and sodium which are strange to find in this alloy with iron and aluminum and chromium, we referred that they came by hand touching.

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