Various Experimental Factors behind the Missing Elements in PIXE Spectrum of Charnockite Matrix

A.V.S. Satyanarayana1 and M. Jagannadha Rao2

1Department of Engineering Physics, Andhra University, Visakhapatnam, Andhra Pradesh-530003, India
2Department of Geology, Andhra University, Visakhapatnam, Andhra Pradesh-530003, India

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ABSTRACT

In the chemical characterization of Charnockite is well investigated by using PIXE and other analytical techniques, but PIXE not given information about some of elements completely, because every technique including PIXE has proper limitations. To obtain the complete geochemical explanation of the matrix Charnockite composition, the experimental factors behind missing elements in PIXE at 3 MeV techniques with comparison must know and hence are used for the analysis of matrix compositions related to the earth materials like Charnockite. In this study, the results obtained from PIXE compared with other analytical techniques and experimental factors of PIXE are discussed. By investigating the geochemical nature of complex Charnockite material, the experimental factors which are related to the missing elements in this study of a wide range of elements obtained from PIXE spectrum through the previous analytical techniques have been discussed.

1. Introduction

Geologically the Visakhapatnam city, AP, India is characterized by rocks termed as Eastern Ghats. The hill, a 136', located at the Visakhapatnam airport is a made up of pyroxene granulites, and Charnockite pegmatites are the first reported occurrence from Visakhapatnam in the Eastern Ghats. The rocks are Precambrian age and comprise mainly Khondalites, Lepitynites, Poryxene Granulites and Charnockites include Biotite, Hastingsite and Apeite mineral halogen rich compositions and all of them have undergone metamorphosis. Among these, Charnockites are termed as upper mantle basic igneous rocks and are emplaced into proto crustal rocks during Precambrian times. In this way the Charnockites sometimes may contain the relict bodies of earlier crustal rocks (proto crust).

Geological materials are normally complex matrix, heterogeneous structures with mineral grains and inclusions. Chemical characteristics of the various compartments have been important for understanding the basic process of formation and the characterization of minerals in terms of their chemical composition, morphology, other minerals and other physical attributes is important in studies of mineral deposits of all types. In many instances, these are significant advantages in trace elements instead of major elements while contracting models of the genesis of geological system, non-destructive techniques include atomic and nuclear processes. Since cross sections for inner shell ionization for nuclear reactions by several orders of magnitudes, the inner shell ionization process is preferably used for the analysis of trace elements. Some of the multi elements are present in minute amounts in geological minerals. In the geosciences like exploration studies and mineral processing of rocks, there is considerable need for elemental analysis.

Earlier, it was very difficult to measure their precise concentrations because of the non-availability of sophisticated analytical methods. With the invention of many modern analytical techniques like Atomic Absorption Spectrometry (AAS), Instrumental Neutron Activation Analysis (INAA), Rutherford Back Scattering (RBS), X-ray Fluorescence (XRF), Energy Dispersive X-ray Fluorescence (EDXRF), Auger Electron Spectroscopy (AES), Particle Induced Gamma ray Emission (PIGE), Particle Induced X-ray Emission (PIXE), Wavelength Dispersive X-ray Fluorescence (WDXRF) etc., it has become possible to estimate the concentrations of trace elements in parts per million (ppm) and parts per billion
(ppb) levels [1-3]. These analytical techniques have the capability to measure the entire major, minor and trace elements present even in the smallest sample with great precision and accuracy.

Among all the aforementioned techniques, PIXE technique [4] has its own advantages and drawbacks [5] over the other techniques. Using the data obtained from the previous investigation, it could be established the performance of PIXE [6] with respect to different elements of the Charnockite samples by comparing previous studies. An attempt also made to present the genetic aspect of the Charnockites studied by obtaining geo chemical data [7] through review of the PIXE spectrum of previous analytical [8] techniques. The present study is aimed at estimating the concentrations of a wide range of elements in matrix Precambrian Charnockite samples of a Charnockite hill near Visakhapatnam airport using particle induced X-ray emission (PIXE) technique.

But some of the elements present in complex geological material by using PIXE analysis are purely determined or could not be determined at all, due to various reasons including the matrix. It is felt that a systematic investigation needs to be designed and implemented to understand the limitation of PIXE in certain elements. The experimental factors behind the missing elements in the analysis of Charnockite composition by PIXE at 3 MeV are investigated.

### 2. Experimental Details

Since PIXE analysis is at Bhubaneswar energy proton created by using 3 MV pelletron accelerator facilities at an institute of physics, Bhubaneswar, India. A 3 MeV, X-ray proton beam magnetically focuses the proton beam of 2 mm diameter beam current to as proton the line surface of the specimen. The samples are mounted on an aluminium target holder (a ladder arrangement). Then the target holder is inserted into the scattering chamber and the irradiation is carried out in vacuum conditions. The beam current is kept at 20 nA and this spot which may be treated under box magnification is ultimately viewed optically with a 300 x microscope equipped with a change completed-device television camera. A large area 80 mm² X-ray detecting nominal resolution 160 eV at 15.9 keV is placed as close as possible to the specimen (25 mm). The specimen stage insulated to permit integration of the incident beam charge.

The samples on the target holder which are to be excited (or) positioned in this scattering chamber at an angle of 45° with respect to the direction of the proton beam. The position of the sample relative to the beam direction is adjusted properly by viewing through a window beryllium of 0.1 mm thickness provided in the scattering chamber. The direction is placed 90° with respect to the beam direction also placed in the chamber at 135° with respect to the beam direction. The output of the Si (Li) detection is completed with data acquisition system which records the X-ray spectrum. The spectrum of each sample is recorded for a sufficiently long time, so as to ensure good statistics.

The Guelph PIXE (GUPIX) software package [9] is used to analyze the spectra utilizing a standard Marquardt non-linear least square fitting procedure. This package provision is to identify different elements present in the sample and to estimate their relative intensities. Using this GUPIX software package the X-ray intensities of different elements is converted into the respective concentrations using a standardized technique involving fundamental parameters, pre-determined instrument constants and input parameters such as solid angle, charge collected etc. Comparing the concentrations of Yttrium obtained in the present work with the known concentration of Yttrium added to the sample, the reliability of the input parameters is checked.

To assure the reliability of experimental system and other parameters, in the same experimental conditions, the PIXE spectrum is recorded with NIST certified reference material and the relative concentrations of different elements are estimated using GUPIX software package. The relative concentrations of different elements, thus obtained in the present experiment for the above standard samples are compared with the certified concentrations supplied by NIST. Good agreement [10] within experimental uncertainties is observed and this shows the reliability of the present experimental system and use of GUPIX software package in the data analysis [11].

### 3. Results

The PIXE spectrum of the matrix Charnockite seven samples collected from the interior of the hill. The concentrations in ppm of these continuous ranges of elements in each sample were determined using the GUPIX software with Si (Li) detector. These concentrations are presented with errors in Table-1 by using the analysis of PIXE spectrum Figure-1-7. The missing elements in the PIXE spectrum of Charnockite samples with respect to previous analytical techniques and hence the experimental factors behind the analysis of charnockite matrix samples by PIXE at 3 MeV are discussed.
Figure 1

Figure 2

Figure 3

Figure 4
Table 1: Analytical results of all matrix charnockite samples (PIXE).

<table>
<thead>
<tr>
<th>S.NO</th>
<th>Element</th>
<th>G1</th>
<th>G2</th>
<th>G3</th>
<th>G4</th>
<th>G5</th>
<th>G6</th>
<th>G7</th>
</tr>
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<tr>
<td>1</td>
<td>Cl</td>
<td>394.1±16.5</td>
<td>399.6±17.1</td>
<td>379±16.5</td>
<td>403.5±19.5</td>
<td>546.9±23.5</td>
<td>383.7±16.7</td>
<td>462.6±20.6</td>
</tr>
<tr>
<td>2</td>
<td>K</td>
<td>4080±28.2</td>
<td>4187±25.1</td>
<td>4148±26.1</td>
<td>4246±29.3</td>
<td>6699±40.2</td>
<td>5458±27.1</td>
<td>5393±33.4</td>
</tr>
<tr>
<td>3</td>
<td>Ca</td>
<td>2229±25.9</td>
<td>2281±22.8</td>
<td>2637±25.6</td>
<td>2754±28.1</td>
<td>4120±23.5</td>
<td>2544±26.2</td>
<td>3091±32.1</td>
</tr>
<tr>
<td>4</td>
<td>Ti</td>
<td>1394±11.6</td>
<td>1271±9.3</td>
<td>1109±9.3</td>
<td>13.67±11.3</td>
<td>1590±13.8</td>
<td>1044±9.1</td>
<td>1510±12.4</td>
</tr>
<tr>
<td>5</td>
<td>V</td>
<td>17.92±4.8</td>
<td>23.69±4.1</td>
<td>7.85±4.0</td>
<td>BDL</td>
<td>37.78±6.0</td>
<td>10.79±4.0</td>
<td>11.55±12.4</td>
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<tr>
<td>6</td>
<td>Cr</td>
<td>16.63±2.3</td>
<td>38.53±2.1</td>
<td>15.16±2.1</td>
<td>16.93±2.5</td>
<td>17.71±3.1</td>
<td>9.118±2.1</td>
<td>14.13±2.6</td>
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<tr>
<td>7</td>
<td>Mn</td>
<td>18.62±3.9</td>
<td>34.71±3.7</td>
<td>34.31±3.7</td>
<td>33.68±4.1</td>
<td>47.43±5.3</td>
<td>27.36±3.5</td>
<td>27.03±4.5</td>
</tr>
<tr>
<td>Element</td>
<td>Atomic Number</td>
<td>Present PIXE Analysis, average of 7 samples</td>
<td>Previous Chemical analysis of pyroxene granulites from charnockitic rocks, Visakhapatnam [15]</td>
<td>Previous, Chemical analysis of allanite from Air Port Hill charnockite, Visakhapatnam [13]</td>
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<tr>
<td>Li</td>
<td>3</td>
<td>ND</td>
<td>18.08ppm</td>
<td></td>
<td></td>
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<td></td>
<td></td>
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<tr>
<td>Be</td>
<td>4</td>
<td>ND</td>
<td>-</td>
<td>&lt;4</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>F</td>
<td>9</td>
<td>ND</td>
<td>0.400ppm</td>
<td>-</td>
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<tr>
<td>Na</td>
<td>11</td>
<td>ND</td>
<td>Na₂O=1.591(Wt%)</td>
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<tr>
<td>Mg</td>
<td>12</td>
<td>ND</td>
<td>MgO=4.022(Wt%)</td>
<td>MgO=1.18(Wt%)</td>
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<tr>
<td>Al</td>
<td>13</td>
<td>ND</td>
<td>Al₂O₃=18.137(Wt%)</td>
<td>Al₂O₃=14.79(Wt%)</td>
<td></td>
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<tr>
<td>Si</td>
<td>14</td>
<td>ND</td>
<td>SiO₂=50.345(Wt%)</td>
<td>SiO₂=31.24(Wt%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>P</td>
<td>15</td>
<td>ND</td>
<td>P₂O₅=0.213(Wt%)</td>
<td>-</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Cl</td>
<td>17</td>
<td>424.2±18.62ppm</td>
<td>0.130ppm</td>
<td></td>
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<tr>
<td>K</td>
<td>19</td>
<td>4887.28±29.91ppm</td>
<td>K₂O=0.428(Wt%)</td>
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<td></td>
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<tr>
<td>Ca</td>
<td>20</td>
<td>2808±26.31ppm</td>
<td>CaO=11.376(Wt%)</td>
<td>CaO=11.01(Wt%)</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Sc</td>
<td>21</td>
<td>ND</td>
<td>23ppm</td>
<td>150ppm</td>
<td></td>
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<tr>
<td>Ti</td>
<td>22</td>
<td>1133.09±10.97ppm</td>
<td>TiO₂=1.314(Wt%)</td>
<td>TiO₂=1.62(Wt%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>V</td>
<td>23</td>
<td>18.26±5.88ppm</td>
<td>240ppm</td>
<td>V₂O₅=0.09(Wt%)</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Cr</td>
<td>24</td>
<td>18.31±2.4ppm</td>
<td>180ppm</td>
<td>70ppm</td>
<td></td>
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<tr>
<td>Mn</td>
<td>25</td>
<td>31.934±4.1ppm</td>
<td>MnO=0.089(Wt%)</td>
<td>MnO=0.28(Wt%)</td>
<td></td>
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<tr>
<td>Fe</td>
<td>26</td>
<td>5961.42±21.5ppm</td>
<td>Fe₂O₃=1.123(Wt%)</td>
<td>Fe₂O₃=5.10(Wt%)</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Co</td>
<td>27</td>
<td>ND</td>
<td>74ppm</td>
<td>150ppm</td>
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</tr>
</tbody>
</table>
4. Discussions

With the comparison Table-2 of previous analytical studies of charnockites [12-15], the elements presented in charnockite composition are stated from Li and Be and but these are not presented and hence detected in the above PIXE at 3 MeV spectrum because of low energy k- X-rays, and these are absorbed by window of the Si(Li) detector and since PIXE experiments fit the elements from Z = 6 to Z = 60 for K X-rays nearly, from Z = 22 to 92 for L-X-rays, from Z = 72 to 93 for M-X-rays and also X-rays of the elements. The element Li, no X-ray and Be with X-ray energy K$_{\alpha}$ = 0.108 keV is not detected even though these elements previously reported in chemical analysis of Charnockites. Similarly the elements F with X-ray energy K$_{\alpha}$ = 0.677 keV and Na (K$_{\alpha}$ = 1.040keV) to P (K$_{\alpha}$ = 2.010 keV) not detected in present investigation due to above detector limits, but these elements analysed by previous analytical methods. Actually the analysis is started from element S (K$_{\alpha}$ = 2.309) but not present in Charnockite composition. The next element F is a trace or minor element in composition, but not trace out in this PIXE analysis due to the low energy < 2 keV characteristic X-ray. Similarly the major elements Na, Mg, Al, Si, and minor or trace P are present in the composition of charnockite by previous analytical techniques, but not identified in this investigation due to PIXE experiment limitations and hence low energy k-X-rays of the elements absorbed by the detector window. Therefore, PIXE unable to detect the low Z elements present like Li, Be, F, Na, Mg, Al, Si and P in charnockites due to detector limits, X-rays below or near the Cl cannot be seen because they are absorbed in either the detector window atmosphere or though any filter used. A possible disadvantage is that low energy X-rays from lighter elements attenuated in the air. The spectrum in above started from a peak of Cl because K$_{\alpha}$ = 2.622 keV, because detector configured limits starts from Cl element. From the element Cl onwards, all the elements within the detector limits,
and all are detected in the above spectrum of Charnockite sample.

The sensitive volume of Si (Li) detector ranges from order of 10 mm in diameter, and order of 5 inch thickness depending on the derived application. The smaller diameter detectors provide better energy resolution of elements at low X-ray energies, and the thicker detectors have higher detection efficiency at the energies of the X-rays above about 20 keV. The X-ray enters the crystal through a thin beryllium window to reach the detector. The beryllium windows of detectors are typically 8-25.4 pm thick. The thickness of the window sets the low energy limit for photons that can be detected by the detector. The X-ray of the order of 2 keV energies of low Z elements can be measured using this Si (Li) detector with the window thickness. Recently in place of beryllium foils very thin (of the order of 0.5 p) films of polycarbonate have been used which can withstand high pressure, and is resistant to environmental degradation. With these windows, low atomic number elements like carbon (0.282 keV K X-rays) has been measured.

From this investigation of Charnockite matrix composition, the knowledge of X-ray production cross sections for light elements is required for quantitative analysis by PIXE using fundamental parameter method. There is a simplest accepted universal expression of ionization cross-section for proton induced X-ray emission. The K and L shell ionization cross-sections as a function of incident proton energy and target atoms in the Charnockite composition. In this, the K-shell X-ray emission. The K and L shell ionization cross-sections for light elements is required for quantitative composition, the knowledge of X-ray production cross sections for light elements is required for quantitative analysis. The K X-rays of rare earth elements like Ti due to above overlapping peaks. PIXE could not perform well in the determination of Fe. Because the overlapped of Co-Kα and Fe-Kα causes the inability of proper detection and in addition to that Ti-Kα and Fe-Kα are same energy or nearly K X-ray energies, (4.509 keV and 4.647 keV). Sometimes it is difficult to deconvolve two peaks from neighboring elements. Some corrections are needed to calculate the exact concentration value of not only Fe, and also the near elements of Fe in Charnockites by PIXE [19].

Determination of Co by PIXE in Charnockites is not possible is due to mainly two reasons; Co-Kα and Fe-Kα and Ni-Kα and Co-Kα interferences of above two peaks trace of cobalt not detected, but in case of previous analysis Co also presented in Charnockites and its value only is low. In these spectrums Fe is the major component so that the X-ray emitted from this element will dominate the energy spectrum. Interference matrix, i.e. X-ray of Kα (Z+1) and the X-ray of Kα (Z) and by correction small interference value, PIXE once again proved in the non-determination of in case of trace elements completely due to matrix composition.

Tin 3.444 3.663 keV (L X-rays) is similar to the potassium K X-rays. Therefore Tin is not detected in charnockite composition by PIXE analysis. Barium 4.466 4.828 22 keV (L X-rays) since K X-rays limited in case of PIXE and for L X-rays, similar to titanium 4.512 4.933 (K X-rays). Therefore Ba is a present charnockite composition but not detected in PIXE analysis due to above reason. Cerium have 4.839 5.262 keV (L X-rays), these L X-ray energies are same as titanium 4.933 keV (K X-rays), and also the near elements of Fe in Charnockites by PIXE [19].

and L-X-ray energies are nearly equal to K-X-ray energies of Co, Ni, Cu, Zn, Ga and Ge. Gallium 9.251 10.267 keV (K X-rays) 1.098 1.125 keV (L X-rays) Germinium 9.886 10.982 keV (K X-rays) 1.188 1.218 keV (L X-rays). Therefore Hf, Ta, W and other rare earth elements are not presented in the PIXE spectrum of Charnockite composition in this PIXE at 3 MeV methodology. Similarly Au L-X-rays, 9.713 11.443 keV, Germinium 9.886 10.982 31 keV L- X-rays, Ga Gallium 9.251 10.267 keV L- X-rays have similar Characteristic X-rays, Au, Ge and Ga are not detected in PIXE but these are already presented in Charnockites in the form of traces by another previous techniques.

In case of PIXE at 3 MeV spectrum analysis of the elements present in Charnockites, the elements Z≤32 an interference is encountered between the Kα X-ray of next element and the Kβ X-ray of previous element which have virtually the same energy or between the X-ray K lines of media elements and X-ray L lines of heavy elements. By PIXE at 3 MeV, the L X-rays energies from 4-9 keV of REE in Charnockites strongly overlap with K X-rays energies of light elements which are presented in the Chrnockite composition (20 to 31 Z) hence the quantification of REE by PIXE with 3 MeV becomes very difficult in this study of Charnockites.

Th and U did not detected by using PIXE analysis Table-1. Because of Uranium contain 98.440 keV, 111.303 keV (k-X-rays), 13.614 keV 17.220 keV (l-X-rays) 3.171 keV 3.336 keV (M-X-rays) and Thorium contain 93.351 keV 105.605 keV (k-X-rays), 12.968 keV 16.202 keV (L-X-rays) 2.996 keV 3.149 keV (M-X-rays). The X-ray energy from this PIXE experiment should only lies 2-20 keV detected in case of PIXE detection, but K-X-rays more energy, therefore detection is not possible through K-X-rays. But the atomic number of Th and U > 83, according to PIXE theory, M X-rays are limited to PIXE technique because for M-X-rays, atomic number validity is 72<Z<83. Finally L X-rays energies of Th and U are similar K-X-ray energies of Br 13.292 keV, Rb 13.396 keV, Sr 15.835 keV, Mo 17.480 keV Table-1. Therefore, due to above reason U and Th not detected in Charnockite composition by PIXE at 3 MeV spectrum analyses.

Conclusions

The drawback of PIXE at 3 MeV to running in this configuration is that below the Cl from spectrums of PIXE are not detected in this work due to low energy X-rays, because they are absorbed in either the detector window atmosphere or through any filter or detector with dimensions used. Therefore the low Z elements Li, Be, F, Na, Mg, Al, Si and P present in Charnockites are not detected. For this purpose low energy PIXE whose cross sections suitable and high efficiency detector or by changing the dimensions of detector, low energy X-rays of light elements can be determined.

In case of PIXE analysis of the elements present in Charnockites with Z≤31 an interference is encountered between the X-ray of Kα (Z+1) and the X-ray of Kβ (Z), which have the same characteristics X-ray energy and also between the X-ray K lines of media elements and X-ray L lines of heavy elements. Due to this the concentration value of Ca, K, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Ru, Ag, Pb are not exactly equal to the concentrations when compared to the other analytical techniques. Few elements like Li, Be, F, Co, Ga, Ge, Ce, Sn, Ba, Hf, Ta, W, Au and REE are not detected due to the various experimental reasons as explained above. Micro PIXE may be suitable to obtain better resolution of wide range of elements in complex material at middle Z elements with same PIXE technique.

By PIXE at 3 MeV, the L X-rays energies from 4-9 keV of REE in Charnockites strongly overlap with K X-rays energies of light elements (20 to 31 Z) and the quantification of REE by PIXE with 3 MeV becomes very difficult detect in this study of Charnockites. Therefore REE are not detected in Charnockite composition by PIXE at 3 MeV. For L X-rays determination of REE, whose cross section is very high, high energy PIXE is required.

In case of PIXE spectrum analysis of the wide range of elements present in Charnockites, interference is encountered between the same energy or between the X-ray K lines of media elements and X-ray L lines of heavy elements. So PIXE with high resolution detector is required to obtain traces of Th and U in case of complex matrix composition.

The elements Thorium and Uranium are not determined in Charnockite composition because of matrix composition contained a long range of elements present in that composition. Also the characteristic X-rays of any series of elements presented are at similar to characteristic X-rays of Thorium and Uranium in the composition. In case of PIXE spectrum analysis of the wide range of elements present in Charnockites, interference is encountered between the same energy or between the X-ray K lines of media elements and X-ray L lines of heavy elements. So PIXE with high resolution detector is required to obtain traces of Th and U in case of complex matrix composition.

From the above, the experimental factors are Si (Li) detector, incident proton energy and cross sections, resolution, Detector limits etc. After study the X-ray spectrum of PIXE in case complex matrix composition, apply the complimentary analytical techniques like PIGE, NAA, EPMA, ICPMS and others at elements in matrix composition which are not detected by PIXE.
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