List of Publications

Publications in International Journals

1. Development of regional PIXE facility at Panjab University cyclotron, Chandigarh (India)

2. Comparison of experimental and theoretical efficiency of HPGe X-ray detector

3. L x-ray production in $^{57}$La, $^{58}$Ce, $^{60}$Nd and $^{62}$Sm by 35 - 60 MeV carbon and oxygen ions

4. PIXE analysis and comparison of aerosol samples from relatively clean and industrially polluted cities in northern India
5. Measurement of wakefield intensity


6. Elemental analysis of Garnet, Citrine and Peridot Using PIXE technique


**Publications in Symposia/Conferences**


2. Identification of Gemstones Using PIXE technique, DAE BRNS Indian Particle Accelerator Conference (InPAC), November 2006, Mumbai.

3. Trace Elemental Analysis with the PIXE Facility at Chandigarh Cyclotron, AvH-sponsored Kolleg, March 2006, Palampur.
DEVELOPMENT OF REGIONAL PIXE FACILITY AT PANJAB UNIVERSITY CYCLOTRON, CHANDIGARH (INDIA)

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ABSTRACT

A description of modifications in the variable energy cyclotron (VEC) Chandigarh along with the installation of Proton Induced X-ray Emission (PIXE) setup is presented. A new main magnet power supply of 400A/725V is put with ±10 ppm stability and a new stabilized solid state power supply for RF oscillator has improved the beam characteristics substantially. A new chamber has been designed to cater for Proton Induced Gamma Emission (PIGE) and Rutherford Back Scattering (RBS) along with PIXE measurements. The HPGe x-ray detector, the Ge(Li) γ-ray detector and a silicon surface barrier (SSB) detector can be mounted simultaneously in the chamber for this purpose. A Turbo-Molecular vacuum pump is provided to produce a clean vacuum of the order of 10⁻⁶ mbar in the PIXE chamber. A remotely controlled stepper motor is provided to move the wheel having 12/24-position target holder. Beam size optimization along with the minimization of background has been done with the help of graphite collimators, thus making the setup suitable for practical applications. Preliminary experiments for the PIXE analysis of aerosol, gunshot residues and kidney stone samples are presented.

Keywords: PIXE set up; Chandigarh Cyclotron; beam size optimization; PIXE setup calibration; MDL.

I. Introduction

A large number of laboratories in the world are engaged in the Proton Induced X-ray Emission (PIXE) technique to exploit its applications in the field of Archaeology, Geology, Biomedical and Environmental sciences. In India PIXE facilities are available only at Institute of Physics (IOP), Bhubaneswar, Orissa and National Centre for Compositional Characterisation of Materials (CCCM), Bhabha Atomic Research Centre, Hyderabad. In northern region the Cyclotron Laboratory at Panjab University, Chandigarh having proton beam of 2-4 MeV is ideally suited for the particle induced X-ray emission (PIXE) technique. India provides an unique opportunity for exploring the archeological, biomedical, environmental and geological samples due to its distinct historical and environmental background.
Environmental pollution is one of the serious problems faced by the people in this country, especially in urban areas, which not only experiences a rapid growth of population due to high fertility, low mortality and increasing rural urban migration, but also industrialization which is accompanied by growing number of vehicles. In India, the increase of population has been tending towards alarming situation. This pollution problem kills thousands and leaves many more to suffer mainly from respiratory damage, heart and lung diseases. So it is important to study this problem thoroughly and PIXE is one of the useful techniques to study it.

Research across the country revealed that people in north India are more prone to stones and hence this region is called as stone belt [1]. A high calcium diet of milk and milk products and heavy consumption of red meat are main causes for this problem. Moreover, heavy physical work with lots of sweating due to very high temperature in summer and less intake of water increases the chances of the occurrence of kidney stones.

Elemental analysis of archaeological artifacts such as pottery, coins, paintings, books and documents can be used to determine the provenance of artifacts and also provide information about the technology used. The study of ancient artifacts from India has a special place in the field of archaeology as India is home to the world’s oldest civilization. We have already made an attempt to address some of the problems using PIXE facility at Institute of Physics, Bhubaneswar, Orissa, India. In these studies more than hundred coins from 200 A.D to 1500 A.D were analysed [2].

Above are some of the examples of important problems in different fields originating in India in which elemental analysis by PIXE technique can play an important role. Developments of such techniques are possible only at a regional center due to the cost and expertise available. Some of the applications of this regional facility planned are:

1. We plan to study the aerosol samples from the vicinity of thermal power plant. The primary goal of this study is to provide data on presence of different trace element in the ambient environment in the vicinity of power plant due to fly ash.
2. We plan to analyse the kidney stone samples collected from patients from northern region of India. The trace element analysis by PIXE will delineate any causal element present in the dietary regimen of north Indian population and thus can help the physician in suggesting remedial measure for future.
3. Blood samples from the orthodontic patients will be analysed for trace elements with PIXE facility at Chandigarh to know the elemental toxicity (mainly Ni) in the blood.

Present paper describes the development of PIXE setup at Chandigarh Cyclotron which includes the modifications of the cyclotron machine, fabrication of PIXE chamber, optimization of proton beam, calibration of PIXE setup and preliminary results of some of the prospective applications.

2. PIXE Set up

The cyclotron originally belonged to Rochester University, Rochester, USA and was gifted to India and installed at Chandigarh in 1976 [3]. The machine is a single Dee,
classical cyclotron, with arrangements for variable frequency. The modifications and additions done at the cyclotron laboratory to develop the PIXE technique as a regional facility are as follows:

Earlier the main magnet D.C. power was provided by a motor generator with shunt feedback amplifier. This system gave a magnetic field stability of about 1 in $10^3$, but this stability was not enough for PIXE work. Therefore, a new magnet power supply from M/S Danfysik, Denmark, Model 853 has been installed. The output power of this supply is 400A/125V with stability of ±10 ppm (8hrs). This modification has improved the orbital stability and focusing which in turn has improved the beam extraction efficiency and energy stability.

The RF oscillator is driven by RCA 5771 tube, which has 25 KVA power triode with water-cooled anode. It has plate voltage 10 kV and frequency range from 10 - 20 MHz. Earlier the plate supply to the oscillator was provided by using vacuum tube rectifiers. But now the power supply has been modified replacing the vacuum tube rectifiers with high voltage silicon diodes network. This has improved the stability of the dee voltage and thereby the beam characteristics.

In different PIXE laboratories, the design of the PIXE chamber somewhat varies [4-9]. However, all these chambers are designed keeping in mind the reproducibility and precision of sample handling, multi target holder arrangements, irradiation procedure, accurate measurement of beam current and the mounting of different radiation detectors (for gamma rays, x-rays and scattered charged particles). Keeping these considerations in mind a new PIXE chamber was designed indigenously and got fabricated from M/S New Poona Industries, Pune, India. Schematic diagram of PIXE chamber is shown in Figure 1.

![Schematic view of PIXE chamber](image)

Fig. 1. Schematic view of PIXE chamber (not to scale).
The entire chamber is made of stainless steel shell (4 mm thickness) of 300 mm inner diameter and of 300 mm height. At 90 mm height the chamber has six ports. Its design meets all requirements of a modern PIXE facility. It has provision for Si(Li) or Ge(Li), low energy HPGe, SSB detectors and a sample holder wheel to mount 12/24 samples at a time, which is insulated from the chamber through a teflon ring so as to enable the measurement of charge in case of thick targets. A Faraday cup is used in case of thin targets. Feedthroughs are provided to supply power to electron gun, current suppressor and SSB detectors. A view port to put close circuit TV camera; a port for attaching vacuum pump and two spare ports are also provided. A DN 100 CF flange at 90° is used to connect the turbomolecular vacuum pump assembly to the chamber with which the final pressure of $\sim 5 \times 10^{-6}$ mbar is achieved.

The chamber has two sample holder wheels. One wheel can hold equally spaced 24 targets of 13 mm and the other can hold 12 targets of 25 mm diameter. The stepper motor with torque 12 kg-cm is made by Sanyodenki, Japan while M/S Penta Designers and Engineers Pvt. Ltd., Pune, India has designed the stepper motor drive (Model MFB-4). The stepper motor and drive units are remotely controlled from the control panel. The stepper motor is placed at 0° and rotates the sample holder wheel coaxially from 0° to 360°.

The port at 135° has a guide of 35 mm ID and 100 mm length inside the chambers to bring the Low Energy HPGe detector as close as 50 mm from the target (centre of the chamber). The angle of 135° has the advantage of a two-fold reduction in bremsstrahlung as compared to 90° [10]. Accutrol Systems Pvt. Ltd. has supplied the HPGe detector (ICLET 06135) for low energy X-ray detection. The performance region with almost 100% efficiency is from 3 keV to 60 keV X-rays, which is ideal for PIXE applications. A thin Be window of thickness 12.7 μm is provided at the entrance which is about 90% transparent to 3 keV X-rays. The detector has an active diameter of 6 mm and active depth of 6 mm with energy resolution of 135 eV at 5.9 keV.

In the PIXE chamber, there is an arrangement for a ladder to hold different absorbers in front of the detector to avoid low energy peaks if necessary and to reduce the bremsstrahlung intensity. This is sometime required to improve the count rate of elements above the background with Z > 34 for which the K X-ray production cross section for 3 MeV protons are smaller as compared to element between Z = 20-30. Mylar of thickness 100-500 μm is ideal and convenient as absorber for biological specimens, since it generates no fluorescence X-ray line to interfere with PIXE spectrum. The compound filters, which acts both critical as well as funny filters, may be used in case of geological and archeological samples. The general view of PIXE setup at VEC, Chandigarh is shown in Figure 2.
3. Results and Discussion

3.1 Beam focusing and the background minimization

After installation of the PIXE setup the next step was to get a uniform focussed beam on the target and, thereby, avoiding any scattering of protons which may induce x-ray emission from the chamber and the target holder wheel made up of stainless steel. To achieve this, the spectra of polyatomic fluorescent material coated on aluminum target were recorded by passing the beam through the adjustable rectangular collimator made up of tantalum placed in the beam line about 30 cm before the target. A large number of counts of Cr, Ni, Mn and Fe were observed in addition to K and Au peaks of the polyatomic material [Figure 3a]. The expected source of these additional x-ray peaks was the target holder wheel made of stainless steel where the scattered protons fall. To verify this, the target was shielded with thick graphite block of thickness 10 mm. The spectrum with reduced background of Fe was observed [Figure 3b]. This confirms that wheel was the main source of Fe peak. To minimize further the scattered protons falling on the wheel, an additional collimator of tantalum with 2 mm diameter was placed ahead of the anti-scattering slit at a distance of ~250 mm from the target. This arrangement further reduced the contribution of the elements present in the stainless steel [Figure 3c]. The spectra were still not free from the contribution of the elements from stainless steel. So finally we removed the tantalum collimator altogether and placed the graphite collimator inside the chamber at a distance of ~35 mm from the target. This arrangement gave a clean spectrum with very little contribution of Fe X-rays [Figure 3d]. The negligible amount of Fe x-rays can be either from the steel chamber or from the iron impurities present in the target.
3.2 Setup calibration

After background minimization we calibrated the set up using thin film standards of rare earth elements namely La, Ce, Nd, Sm, Gd, Dy, Er and Yb whose spectra are shown in Figure 4. All these targets were prepared on aluminised polypropylene (6 μm thickness) backing by vacuum evaporation technique at Nuclear Science Centre, New Delhi, India. The thicknesses of these targets were checked by energy dispersive X-ray fluorescence (EDXRF) technique [11]. In the EDXRF measurements the thickness of each target was measured against the calibrated micromatter standards. It may be added that micromatter standards have been used extensively for the measurement of X-ray fluorescence cross sections [12] and our experience shows that the thickness accuracy of micromatter foils is better than 2%. Therefore, the accuracy of the thickness of unknown foils measured by EDXRF is at least better than 5%. For a reliable calibration of PIXE setup the elemental
thickness of these were evaluated using 3 MeV protons at cyclotron. The expression used to evaluate concentration is,

\[
C_Z \rho t = Y(Z) \times A_Z \times \sin \theta / N_A \times \sigma(E_0) \times \varepsilon_{\text{abs}} \times n_p.
\]

Where, \(Y(Z)\) is the number of counts in the characteristic x-ray line of the analyte element with atomic number \(Z\), \(N_A\) Avogadro's number, \(\sigma(E_0)\) the x-ray production cross section (barn/atom) at the incident proton energy \(E_0\), \(\varepsilon_{\text{abs}}\) the absolute detection efficiency (including all absorbing components of the set up) for X-ray line, \(n_p\) the number of incident protons, \(C_Z\) the concentration (W\% weight percent) of the analyte element in the specimen, \(\rho\) the specimen density (g/cm\(^3\)), \(t\) the specimen thickness (cm), \(A_Z\) the atomic mass of the analyte element and \(\theta\) the angle between the incident proton beam and the specimen surface. The product \(C_Z \rho t\) gives the thickness density (g/cm\(^2\)) per unit area.

Fig. 4. PIXE spectra of thin foils of rare earth elements.
In the above expression the theoretical X-ray production cross section $\sigma'(E_x)$ were calculated using the equations given by Close et al [13], $n$ was evaluated from the charge collected in the Faraday cup and absolute intrinsic efficiency $\varepsilon_{abs}$ was calculated using expression:

$$\varepsilon_{abs} = \varepsilon_{in} \times \varepsilon_{geo} \times \varepsilon_{A}.$$  

Where,  
$\varepsilon_{in} = \frac{\text{Events registered}}{\text{Events impinging on detector}}.$  
$\varepsilon_{geo} = \Omega / 4\pi$ ($\Omega$ : Solid angle of detector).  
$\varepsilon_{A} = \text{Absorption in air and window of the chamber}.$

Here intrinsic efficiency ($\varepsilon_{in}$) of the HPGe detector was evaluated using GUPiX code [14], using parameters supplied by the manufacturer.

Using proton beam of 3 MeV each foil was run for a fixed charge of about 50-60 $\mu$C except Er (Q=120 $\mu$C) and Yb (Q=131 $\mu$C). The beam current was kept lower than 5 $nA$. The thickness of the foils measured with PIXE at cyclotron along with the values of parameters used in their calculations and EDXRF evaluated thicknesses are given in Table 1.

<table>
<thead>
<tr>
<th>Foil</th>
<th>EDXRF measured thickness in $\mu g/cm^2$</th>
<th>PIXE measured thickness in $\mu g/cm^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>La</td>
<td>59</td>
<td>57</td>
</tr>
<tr>
<td>Ce</td>
<td>66</td>
<td>64</td>
</tr>
<tr>
<td>Nd</td>
<td>58</td>
<td>47</td>
</tr>
<tr>
<td>Sm</td>
<td>47</td>
<td>45</td>
</tr>
<tr>
<td>Gd</td>
<td>48</td>
<td>56</td>
</tr>
<tr>
<td>Dy</td>
<td>70</td>
<td>68</td>
</tr>
<tr>
<td>Er</td>
<td>29</td>
<td>40</td>
</tr>
<tr>
<td>Yb</td>
<td>58</td>
<td>70</td>
</tr>
</tbody>
</table>

The excellent agreement between the EDXRF measured and the PIXE measured concentrations for these thin foils except for Er and Yb. The difference between EDXRF and PIXE results for these elements can be due to large overlapping for Er and Yb K X-ray lines with the 59.5 keV scattered peaks of Am$^{241}$ source used in EDXRF. It indirectly checks the accuracy of the proton energy, efficiency of the detector, thickness of the window at the detector port, and the air gap between the detector and the sample.

The minimum detection limit (MDL) of PIXE setup for thin and thick targets was obtained using the relation
\[ \text{MDL} = \frac{(3 \times \text{sigma})}{(C \times H \times Y_t \times \varepsilon \times \Gamma)} \]

Where,
- \( \text{sigma} \) = the standard deviation error, e.g. \( \sqrt{B} \)
- \( B \) = background area in one FWHM region
- \( C \) = the charge in \( \mu\text{C} \)
- \( H \) = standardization value used in conversion of area to concentration (contains solid angle etc.)
- \( Y_t \) = theoretical yield of x-rays/\( \mu\text{C} \cdot \text{sr} \cdot \text{mg/cm}^2 \) for thin targets or in units of \( \mu\text{C} \cdot \text{sr} \cdot \text{ppm} \) for thick targets in the 1 FWHM region centered about the elements principal peak.
- \( \varepsilon \) = relative detector efficiency
- \( \Gamma \) = transmission of x-rays through any absorber that is present.

For our PIXE setup the MDL for different trace elements in thin and thick targets are given in Table 2.

<table>
<thead>
<tr>
<th>Element</th>
<th>MDL (ng/cm²) for thin aerosol samples on polycarbonate filters (total charge 20.2 ( \mu\text{C} ))</th>
<th>MDL (ppm) for thick samples in graphite of purity 99.5% (total charge 3 ( \mu\text{C} ))</th>
</tr>
</thead>
<tbody>
<tr>
<td>S</td>
<td>7.56</td>
<td>2.05 e-2</td>
</tr>
<tr>
<td>K</td>
<td>6.55</td>
<td>1.19 e-2</td>
</tr>
<tr>
<td>Ca</td>
<td>6.21</td>
<td>8.97 e-3</td>
</tr>
<tr>
<td>Ti</td>
<td>3.82</td>
<td>4.42 e-3</td>
</tr>
<tr>
<td>V</td>
<td>2.49</td>
<td>4.55 e-3</td>
</tr>
<tr>
<td>Cr</td>
<td>1.88</td>
<td>3.04 e-3</td>
</tr>
<tr>
<td>Mn</td>
<td>2.11</td>
<td>2.86 e-3</td>
</tr>
<tr>
<td>Fe</td>
<td>2.30</td>
<td>2.94 e-3</td>
</tr>
<tr>
<td>Zn</td>
<td>5.13</td>
<td>4.93 e-3</td>
</tr>
<tr>
<td>Br</td>
<td>13.61</td>
<td>2.15 e-2</td>
</tr>
<tr>
<td>Pb</td>
<td>36.41</td>
<td>2.84 e-2</td>
</tr>
</tbody>
</table>

These are not only comparable but are even better than the one reported by Johansson et al. [15]. This may be due to the detector position of 135° where contribution due to bremsstrahlung is minimized. Next step after calibration was to use it for the estimation of trace elements in thin/thick targets. Presently we analysed two aerosol samples, two samples of the gun shot residue and two kidney stone samples. The results are discussed below.
Aerosol samples

The aerosol samples were collected from industrial town Gobindgarh, Punjab, India using aerosol sampling kit which involves Millipore diaphragmatic vacuum pump and Sequential filter unit (SFU) with coarse pored Nuclepore polycarbonate filter in such a manner that the particles passing through the 8 μm pore size filter are collected on the 0.4 μm pore size filter. The sampling time for the sample was 12 hrs with flow rate 8 l/min. The aerosol samples were collected on the flat rooftop of the building (20-40 feet high) to have an effective collection of the aerosol below 10 μm size. Typical PIXE spectra of these aerosol samples on filter papers of 8 μm and 0.4 μm pore sizes are shown in Figure 5 (a,b). Both samples have common trace elements namely K, Ca, Ti, V, Mn and Fe but Cr, Zn, Br and Pb present in aerosol sample collected on the filter paper of 0.4μm pore size while the concentrations of K, Ca, Ti, V, Mn and Fe are higher in aerosol sample collected on filter paper of 8 μm pore size. Quantitative analysis of both the samples are shown in Table 3. The presence of S, Zn, Br and Pb in addition to the other elements collected on 0.4 μm pore size filter paper shows that they are from the industrial activity, which may include coal and oil burning in addition to the rolling of iron ingots [16].

![Image](image_url)

**Fig. 5.** PIXE spectra of aerosol samples; (a) collected on 8.0 μm filter paper (b) collected on 0.4 μm filter paper.
Gun Shot Residue (GSR) samples

Two different pistols viz. 7.65 mm Indian made (sample-1) and 7.63 mm Germany made (sample-2) were used to fire at a filter paper target with cardboard and cotton wool backing from a distance of 3 feet. Two sheets of Whatman filter paper were used to form the target. The GSR were deposited on the front filter paper, while the back filter paper protects the front one from any contamination's due to the backing material. The proton beam was bombarded on the contact ring on the front filter paper and the PIXE spectrum was taken. These spectra are shown in Figure 6 (a, b). This was done to know the elemental composition of the fire arm discharge residue at the target from different firearms and ammunitions. The quantitative estimation of these spectra are shown in Table 4.

Table 3. Quantitative analysis of the aerosol sample collected on filter papers of 8 μm and 0.4 μm pore sizes.

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration in ng/m² (Pore size 8 μm)</th>
<th>Concentration in ng/m³ (Pore size 0.4 μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S</td>
<td>-</td>
<td>210</td>
</tr>
<tr>
<td>K</td>
<td>816</td>
<td>445</td>
</tr>
<tr>
<td>Ca</td>
<td>1861</td>
<td>469</td>
</tr>
<tr>
<td>Ti</td>
<td>180</td>
<td>46</td>
</tr>
<tr>
<td>V</td>
<td>36</td>
<td>17</td>
</tr>
<tr>
<td>Cr</td>
<td>-</td>
<td>23</td>
</tr>
<tr>
<td>Mn</td>
<td>33</td>
<td>24</td>
</tr>
<tr>
<td>Fe</td>
<td>1454</td>
<td>482</td>
</tr>
<tr>
<td>Zn</td>
<td>-</td>
<td>512</td>
</tr>
<tr>
<td>Br</td>
<td>-</td>
<td>189</td>
</tr>
<tr>
<td>Pb</td>
<td>-</td>
<td>445</td>
</tr>
</tbody>
</table>

Both the samples have common trace element namely Cl, K, Ca, Fe, Cu, Zn, Ba and Pb but Cr and Ni are present in the sample-1. Ba and Pb are from the primer residue where as Fe, Cu, Zn, Cr and Ni are due to the jacket of the bullet as well as the corrosion of the barrel. Cl, K and Ca are from the filter paper itself. The concentration of Cu and Ba are comparable in both the cases but the concentrations of Fe, Zn and Pb are remarkably higher in sample-2. The Difference in composition and concentrations of GSR samples from different firearms at same distance may provide useful information in forensic studies.
Kidney stone samples

Two kidney stone samples were collected from Urology department of Post Graduate Institute of Medical Research and Education (PGIMER), Chandigarh, India. These samples were dried in oven at 75°C and crushed to a fine powder. Equal amount of Graphite powder (purity 99.5%) are mixed and pallets of diameter 13mm were prepared with the help of the palletizer using pressure of the order of 60-70 kg-N. The samples were then irradiated with the proton beam of 3 MeV at Chandigarh cyclotron. Typical spectrum of kidney stone in graphite matrix along with graphite alone are shown in Figure 7 (a, b).

![Graph 1](image1)

![Graph 2](image2)

Fig. 6. PIXE spectra of GSR samples; (a) taken by firing a 7.65 mm Indian made pistol (b) taken by firing a 7.63 mm Germany made pistol.
Table 4. Quantitative analysis of the Gun Shot Residue sample; Sample-1: taken by firing a 7.65 mm Indian made pistol. Sample-2: taken by firing a 7.63 mm Germany made pistol.

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration in ng/cm² (sample-1)</th>
<th>Concentration in ng/cm² (sample-2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cl</td>
<td>125</td>
<td>44</td>
</tr>
<tr>
<td>K</td>
<td>66</td>
<td>125</td>
</tr>
<tr>
<td>Ca</td>
<td>222</td>
<td>549</td>
</tr>
<tr>
<td>Cr</td>
<td>104</td>
<td>--</td>
</tr>
<tr>
<td>Fe</td>
<td>360</td>
<td>1230</td>
</tr>
<tr>
<td>Ni</td>
<td>16</td>
<td>--</td>
</tr>
<tr>
<td>Cu</td>
<td>350</td>
<td>509</td>
</tr>
<tr>
<td>Zn</td>
<td>55</td>
<td>67</td>
</tr>
<tr>
<td>Ba</td>
<td>169</td>
<td>170</td>
</tr>
<tr>
<td>Pb</td>
<td>275</td>
<td>837</td>
</tr>
</tbody>
</table>

Fig. 7. PIXE spectra of: (a) kidney stone (b) graphite only.
The typical spectrum shows the presence of only Ca peaks and no other element was detected above the background. Ca is normally considered to be the main constituent of kidney stone in the form of calcium oxalate. The possible source of it can be high milk diet of the north Indian people.

Acknowledgement

The research grant from Department of Science and Technology is gratefully acknowledged under which this regional PIXE facility is developed. We are also thankful to the staff of the Cyclotron Laboratory for providing an excellent beam at the target.

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Comparison of experimental and theoretical efficiency of HPGe X-ray detector


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Abstract

The low energy high purity germanium (HPGe) detectors are being increasingly used for the quantitative estimation of elements using X-ray spectrometric techniques. The softwares used for quantitative estimation normally evaluate model based efficiency of detector using manufacturer supplied detector physical parameters. The present work shows that the manufacturer supplied detector parameters for low energy HPGe detectors need to be verified by comparing model based efficiency with the experimental ones. This is particularly crucial for detectors with ion implanted P type contacts.

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Keywords: HPGe detector; X-ray spectrometry; Efficiency; Si(Li) detector

1. Introduction

The Si(Li) detector has been studied theoretically as well as experimentally over last three decades and is well understood [1–5]. Until recently Ge detectors were normally used for medium and high energy γ-ray detection. With the improvement of fabrication techniques high purity germanium (HPGe) detectors are now available for low energy X-ray detection. The HPGe offers a superior energy resolution as compared to Si(Li) detector as the mean energy required to produce an electron–hole pair is 2.97 eV in Ge as compared to 3.76 eV in Si and the Fano factor of 0.08 for Ge and 0.1 for Si [6]. However, for HPGe detector the theoretical ratio of the intensity of the K escape peaks to the parent peaks reaches up to 15% just above the absorption edge, whereas for Si(Li) the maximum is 2% [7]. The low energy HPGe detector has a distinct advantage in the energy region 20–80 keV because its efficiency is higher by a factor of five as compared to Si(Li) detector [8]. In the energy region 2–14 keV both detectors have similar efficiency. The L X-rays of high Z (70–92) and the K X-rays of medium and low Z (13–40) elements fall in this energy region. The energy region above 15 keV is important for the fundamental studies of the atomic inner shell processes [9] in the medium and high Z region and the study of rare earth elements through their K-rays excited by heavy ions [10]. The most widely used quantitative X-ray analysis codes like Gupix [11] and QXAS [12] normally generate the detection efficiency data using physical parameters of the detector. In order to achieve sufficient accuracy and reliability in the quantitative analysis, exact physical parameters of the detector used should be supplied to these codes. Although the use of LEGe detectors in X-ray analysis is increasing, the efficiency and other characteristics of these detectors are much less studied in comparison to Si(Li) detectors.

Campbell et al. [13] measured the spectrum shapes of Si(Li) and Ge detectors using monochromatic X-rays from...
The Ge detector used by them was having surface barrier front contact. The recent version of GUPIX software also includes Ge detector in addition to Si(Li) detectors. The Au contact layer of 0.1 \( \mu \text{m} \) quoted for the Ge detector is included in the GUPIX library.

Salgado et al. [14] studied the HPGe detector response for 20–150 keV X-rays where the experimental observations were compared with Monte Carlo simulations. They found that the detector’s dead layer thickness of 0.5 \( \mu \text{m} \) quoted by the manufacturer had to be changed to 4 \( \mu \text{m} \) in order to explain the observed detector efficiency.

For the HPGe detector supplied by Canberra USA to us, we also observed a large deviation in the experimental efficiency when compared with the one evaluated using the empirical model. Both the detectors, the one used by us and the other by Salgado et al. [14] are having ion implanted front contact. However, Campbell et al. [13] had not reported such a difference in case of the detector used by them. Keeping these discrepancies in mind and increased use of HPGe detectors for X-ray detection we have carried a systematic study of the efficiency of HPGe detector supplied by Canberra Inc. USA.

2. Materials and methods

According to the manufacturers, the 5 mm thick LEGe detector crystal has planar geometry with circular face of 30 mm\(^2\) active area and is located at 5 mm behind the 25 \( \mu \text{m} \) thick beryllium window. The crystal has a front dead layer of 0.3 \( \mu \text{m} \) that is supposed to be the depth of the Ge crystal up to which the trivalent boron atoms are implanted for creating P type contact.

For experimental efficiency measurement, X-rays and \(\gamma\)-rays emitted by circular radioactive sources of \(^{55}\text{Fe}\), \(^{57}\text{Co}\), \(^{152}\text{Eu}\) and \(^{241}\text{Am}\) with a 2 mm active diameter were detected by the detector. The activities of \(^{55}\text{Fe}\), \(^{57}\text{Co}\) and \(^{241}\text{Am}\) were 8.8758, 4.3423 and 0.29957 \( \mu \text{Ci} \), respectively, at the time of the measurement after applying necessary decay corrections. The activity of the \(^{152}\text{Eu}\) radioisotope was not known. All the radioisotopes were supplied by the Board of Radiation and Isotope Technology (BRIT), India. The radioactive sources were placed inside a vacuum chamber which had a 20 \( \mu \text{m} \) thick Mylar exit window for the X-rays. The source and detector were placed in a parallel plane with the source centre laying along the detector axis as shown schematically in Fig. 1. The distance between source and detector crystal was 43 mm out of which 13 mm was air gap. Teflon collimators with 5.6 mm diameter aperture and total length of 15 mm were used in front of the detector to collimate the photons emitted by the source.

The absolute detection efficiency of a Si(Li) detector supplied by Ortec Inc. USA was also measured for the cross confirmation of our methodology. The Si(Li) detector has a 25 \( \mu \text{m} \) thick beryllium window and the crystal is 5 mm thick with 10 mm active diameter. On the front surface of the crystal, a gold layer of 200 Å thick makes the electrical contact and the thickness of Si dead layer is 0.1 \( \mu \text{m} \). This detector was also placed axially in front of the source at a distance of 21 mm. A 12 mm long circular collimator with 10 mm aperture was used in between the detector and the source.

![Fig. 1. Schematic representation of the source detector geometry used to determine the low energy HPGe detector efficiency.](image-url)
The photo peak areas of X-rays and γ-rays in the spectra recorded from the detectors were extracted using computer program FREEDOM [15]. The efficiency curve is estimated by using the relation

\[
e = \frac{\text{Number of particles detected (} N_d \text{)}}{\text{Number of particles emitted (} N_e \text{)}},
\]

where number of particles detected is the area under the photo peak of a particular X-ray or γ-ray line and the number of emitted particles is calculated using the following relation,

\[
N_e = N_0 e^{-\lambda t}.
\]

With \( I \) being the absolute intensity of the line of interest; \( N_0 \), the initial activity of the radioactive source; \( \lambda \), the decay constant and \( t \), the elapsed time. From this relation it is evident that accuracy of the efficiency curve relies mostly upon the peak area and the intensity of the line of interest. Final uncertainty in the measured efficiency was evaluated from the uncertainties in intensity of the X-ray or γ-ray lines, initial activity (≤1%), elapsed time (≤0.6%) and the peak area (≤3%). The major source of error was due to the uncertainties of the line intensities which vary for strong and weak lines. Finally, for most of the efficiency points, the total error in our case was below 6%.

The theoretical detector intrinsic efficiency was evaluated using detector parameters like thickness and density of detector crystal, contact layer and detector entrance window applying the model based on photon atom interaction [1]. The semi empirical model used for efficiency evaluation is described by the equation

\[
e = \left(\frac{\Omega}{4\pi}\right) \left(\exp\left[-\sum_{i}^n \mu_i d_i\right]\right) f_E(1 - \exp(-\mu_c D))
\]

where \(\Omega/4\pi\) is the fractional solid angle subtended by the detector crystal at the source and \( i \) denotes the medium between the source and the beryllium window, the window itself, a possible ice layer, the gold electrode, the frontal crystal dead layer etc. The \( d_i \) are the thickness of these absorbers and \( \mu_i \) are their linear absorption coefficients taken from XCOM [16] database. The term \( f_E \) represents the escape peak correction factor. The \( \mu_c \) is the photoelectric absorption coefficient of the detector crystal of thickness \( D \).

The fractional solid angle, \(\Omega/4\pi\), was calculated using the formula derived by Nelson Blachman [17,18] which is valid for a wide range of source detector geometries except for large sources close to the detector. If \( h \) represents the distance between source of radius \( r \) and the detector of radius \( R \) placed in a axial geometry, then the fractional solid angle subtended by the detector at the source is given by

\[
\frac{\Omega}{4\pi} = \frac{1}{2} \left[ 1 - \frac{1}{(1 + z)^{3/2}} \right] + \frac{\beta^2}{2} \left[ \frac{5}{16} \frac{\alpha}{(1 + z)^{7/2}} - \frac{35}{16} \frac{\alpha^2}{(1 + z)^{9/2}} \right]
\]

with

\[
\alpha = \left(\frac{R}{h}\right)^2, \quad \beta = \left(\frac{r}{h}\right)^2.
\]

3. Results and discussion

The experimental efficiency along with the model based efficiency for the Si(Li) detector is shown in Fig. 2. As it is evident from the figure, a reasonable good agreement between the experimental and the model efficiency is achieved. The maximum difference between the measured efficiencies and the corresponding model based value is ~6% i.e. within the experimental error. From this comparison we can conclude that the methods for the experimental as well as the model based calculations are reliable. An inappropriate selection of intensity values for the radioisotopes can give large errors as we found when the intensity values for different \( N_p \) L X-ray lines coming...
from $^{241}$Am were taken from the table of radioactive isotopes (TORI) [19]. Therefore, the use of already experimentally verified intensities as reported by Johnston [20] and the IAEA values [21] are recommended.

Based on the above experimental method and the model based calculation we compared the efficiency for the low energy HPGe detector as shown in Fig. 3. The detector parameters taken are those provided by the manufacturer which includes a dead layer of 0.3 $\mu$m thick. It is evident that there is a large deviation which, for a few energies deviates by a factor of 3. Initially it was thought to be the error in calculating the escape peak correction factor in low energy HPGe detectors. However, the existing literature shows that the maximum contribution of the escape peak in Ge detectors can be only up to 20% [6]. Except for the frontal crystal dead layer, the $\mu$ values of other absorber layers mentioned in Eq. (2) are quite small for X-rays with energy $> 3$ keV. Moreover, the thickness of the Mylar window and the air gap are measured quite precisely and the manufacturer declared value of Be window thickness are reliable. Hence these observed deviations can not be attributed to any uncertainties associated with these absorbers.

Recently Salgado et al. [14] have compared the experimental efficiency of HPGe detector with the Monte Carlo model based calculations in the 20–150 keV region. Their detector consisted of a planar crystal with $p^+$ ion implanted front contact. To the best of our knowledge this is the only detector of its type whose efficiency has been reported. In order to achieve a good agreement (within 5%) between the model efficiency points and the experimental ones they had to take a dead layer thickness of 4 $\mu$m instead of quoted value of just 0.5 $\mu$m. Recent studies by Shariff et.al. [22] and Campbell et al. [13] have not reported any comparison of the experimental and the modeled efficiency points and have mainly focused on the line shapes.

In order to understand the efficiency for the HPGe detectors, considering the above fact that all the physical quantities except the dead layer thickness are quite reliable, we tried to vary the crystal dead layer thickness to achieve a better fit between the experimental and the model calculations. For this purpose, in the model calculations, the dead layer thickness was varied from 0.3 to 20 $\mu$m. The percentage difference between the calculated values and experimental data were extracted for every dead layer thickness. The average percentage difference, determined using the absolute values of these differences for all the energy points, is found to be decreasing with increasing dead layer thickness up to 15 $\mu$m after which, it started increasing till 20 $\mu$m. A comparison of experimental efficiency points to that of model calculations are given in Table 1 for five different dead layer thicknesses. It can be observed that, for a dead layer of 15 $\mu$m thick, the model calculations become closest to the experimental values for majority of the energy points of our interest. As shown in Fig. 4, the experimental efficiency values match well with the calculated efficiency at almost all energies except at 3.3 keV which, may be because of a possible overestimation of the area under 3.3 keV line due its overlap with the Ge $K_b$ escape peak of 13.9 keV line.

### Table 1

<table>
<thead>
<tr>
<th>Source Energy (keV)</th>
<th>Efficiency ($\times 10^{-4}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Expt.</td>
</tr>
<tr>
<td>$^{241}$Am 3.30</td>
<td>1.46 (16)</td>
</tr>
<tr>
<td>$^{55}$Fe 5.90</td>
<td>3.34 (13)</td>
</tr>
<tr>
<td>$^{152}$Eu 46.60</td>
<td>7.94 (50)</td>
</tr>
<tr>
<td>$^{152}$Eu 45.40</td>
<td>9.26 (55)</td>
</tr>
<tr>
<td>$^{152}$Eu 42.99</td>
<td>10.49 (69)</td>
</tr>
<tr>
<td>$^{152}$Eu 42.31</td>
<td>10.59 (70)</td>
</tr>
<tr>
<td>$^{57}$Co 14.41</td>
<td>7.57 (19)</td>
</tr>
<tr>
<td>$^{55}$Fe 17.75</td>
<td>9.36 (37)</td>
</tr>
<tr>
<td>$^{152}$Eu 39.52</td>
<td>8.44 (53)</td>
</tr>
<tr>
<td>$^{57}$Co 26.34</td>
<td>7.33 (42)</td>
</tr>
<tr>
<td>$^{55}$Fe 33.20</td>
<td>9.36 (53)</td>
</tr>
<tr>
<td>$^{152}$Eu 40.12</td>
<td>8.38 (54)</td>
</tr>
<tr>
<td>$^{57}$Co 24.31</td>
<td>9.10 (50)</td>
</tr>
<tr>
<td>$^{152}$Eu 32.21</td>
<td>9.10 (50)</td>
</tr>
<tr>
<td>$^{57}$Co 40.45</td>
<td>9.10 (50)</td>
</tr>
<tr>
<td>$^{55}$Fe 46.60</td>
<td>7.94 (50)</td>
</tr>
<tr>
<td>$^{152}$Eu 46.85</td>
<td>12.04 (81)</td>
</tr>
</tbody>
</table>

The calculated efficiency for five dead layers viz. 0.3, 4, 10, 15 and 20 $\mu$m are given.

![Fig. 3](image-url)  
**Fig. 3.** Experimental efficiency points along with model based efficiency curve for a dead layer of 0.3 $\mu$m.

### 4. Conclusion

From the comparison of the modeled efficiency based on modified dead layer thickness and the experimental points, it is evident that the reported dead layer from the manufactures for the low energy HPGe detector can not
be relied upon for the model calculations. Therefore, it becomes necessary to cross check the model parameters with the experimental points before using them in different X-ray spectrometry softwares like Gupix for trace elemental concentration calculations. The use of low energy HPGe detectors is expected to increase in future due to their convenience of not requiring liquid nitrogen all the times and better energy resolution. The present study has tried to improve the understanding of the low energy HPGe detectors which is required for the usage of HPGe detectors in future for trace element evaluation applying X-ray spectrometry techniques.

References

[15] FREEDOM, Data acquisition and analysis system designed to support the accelerator based experiments at inter university accelerator centre (IUAC), New Delhi, India.
L x-ray production in $^{57}$La, $^{58}$Ce, $^{60}$Nd and $^{62}$Sm by 35–60 MeV carbon and oxygen ions

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$^{c}$ Department of Biophysics, Panjab University, Chandigarh 160014, India
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Abstract

L x-ray production cross sections have been measured for thin solid targets ($\sim$50 $\mu$g/cm$^2$) of $^{57}$La, $^{58}$Ce, $^{60}$Nd and $^{62}$Sm for 35–60 MeV C$^{4+}$ and O$^{5+}$ ions. There is an increase in these cross sections with the increasing projectile energy and the ratio of its atomic number to the atomic number of the target nucleus ($Z_1/Z_2$). The measured L x-ray production cross sections have been compared with the predicted values of the first Born approximation (FBA) and of the ECPSSR theory that accounts for the projectile energy loss and Coulomb deflection and the perturbed stationary-state and relativistic nature of the L-shell. For C$^{4+}$ ions, there is a reasonable agreement with the ECPSSR predicted values for the higher $Z$ elements (Nd and Sm), while for the lower $Z$ elements (La and Ce), the FBA is in a better accord with the data. For O$^{5+}$ ions, the FBA gives a better agreement for all the elements than the ECPSSR theory.

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Keywords: L-shell; Cross section; HPGe; Lanthanum; Cerium; Neodymium; Samarium; Carbon ions; Oxygen ions; First Born approximation; ECPSSR; MeV; Ion-atom collisions

1. Introduction

In heavy-ion-atom collisions, an inner shell vacancy is produced due to the direct ionization (DI) as well as from the electron capture (EC) by an
ionic projectile. The DI mechanism is dominant when $Z_1 / Z_2$ and $v_1 / v_2S$, while the EC process dominates if $Z_1 \leq Z_2$ and $v_1 \leq v_2S$, where $Z_1$ and $Z_2$ are the atomic numbers of the projectile and the target atom, and $v_1$ and $v_2S$ are the velocities of the projectile and the target inner-shell ($S = K-, L$-shell) electrons. The first-order Born approximation (FBA) describes the process using the plane wave Born approximation (PWBA) [1] for DI and the Oppenheimer–Brinkman–Kramers approximation of Nikolaev (OBKN) [2] for EC, whereas the ECPSSR theory for DI [3] and EC [4] accounts for the energy loss ($E$), Coulomb deflection ($C$) of the incident ion as well as for the perturbed stationary states ($PSS$) and the relativistic ($R$) nature of inner shell electrons.

A large database for the L-shell ionization by protons and light ions exists in the literature [5,6], whereas it is scarce for heavy-ion projectiles. Only a few measurements are available in the literature on the study of the L-shell ionization by carbon and oxygen ion beams for energies $E_1 \geq 30$ MeV. Table 1 surveys the published data on L-shell x-ray production by heavy-ion beams [7–20]. At energies above 30 MeV, the reports [7,8,10–12,15,18] of experimental x-ray production cross-sections are indeed rare. In the present work, L-shell x-ray production cross sections have been measured with thin (47–59 μg/cm²) solid targets of 57La, 58Ce, 60Nd and 62Sm for 35–60 MeV $C^{4+}$ and $O^{5+}$ ions. For this range of projectile-target systems, the 36 MeV $O^{5+}$ on 58Ce and 62Sm cross sections are the only other data [8]. The measured x-ray production cross sections are compared with the predictions of the FBA [1,2] and ECPSSR [3,4] theories, and the data of Pepper et al. [8] from three decades ago.

2. Experimental details and data evaluation

The experiment was performed with 15 UD pelletron at Nuclear Science Centre (NSC), New Delhi, India. The carbon and oxygen beam having

<table>
<thead>
<tr>
<th>Projectile</th>
<th>Target</th>
<th>Energy range</th>
<th>Type of x-ray measurements</th>
<th>Reference (year)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>Ag, Au</td>
<td>12–50 MeV</td>
<td>$L_{\text{total}}, L_y/L_p, L_y/L_x$ ratios</td>
<td>Bissinger et al. [7] (1974)</td>
</tr>
<tr>
<td>O</td>
<td>Ce, Pr, Sm, Eu, Dy, Ho</td>
<td>8–36 MeV</td>
<td>$L_{\text{total}}, L_y, L_{\beta_1}, L_{\beta_2}, L_{\gamma_1}, L_{\gamma_2,3}$</td>
<td>Pepper et al. [8] (1975)</td>
</tr>
<tr>
<td>C,N,O</td>
<td>Au</td>
<td>0.4–3.4 MeV</td>
<td>$L_{\text{subshells}}, L_{\text{total}}$</td>
<td>Sarkadi and Mukoyama [9] (1980)</td>
</tr>
<tr>
<td>C,O</td>
<td>Nd, Gd, Ho, Yb, Au, Pb</td>
<td>25 MeV C ($q = 4, 5, 6$), 32 MeV O ($q = 5, 7, 8$)</td>
<td>$L_{\text{total}}$</td>
<td>Andrews et al. [10] (1987)</td>
</tr>
<tr>
<td>Li, Be, C, N, F, Si</td>
<td>Yb, Au</td>
<td>0.5–3.0 MeV/u for Li, Be, C, F; and 0.5–2.6 MeV/u for N, Si</td>
<td>$L_{\alpha_{1,2}}, L_{\gamma_1}, L_{\gamma_2,3,6}$</td>
<td>Malhi and Gray [11] (1991)</td>
</tr>
<tr>
<td>C</td>
<td>Cu, Ga, Ge, Br, Y, Nd, Gd, Ho, Yb, Au, Pb</td>
<td>2–25 MeV</td>
<td>$L_{\text{total}}$</td>
<td>Mehta et al. [12] (1993)</td>
</tr>
<tr>
<td>C,O</td>
<td>Au, Bi</td>
<td>3.6–9.5 MeV for C, 4.0–7.2 MeV for O</td>
<td>$L_{\gamma_1}, L_{\gamma_2,3,6}, L_{444}$</td>
<td>Bhattacharya et al. [13] (1994)</td>
</tr>
<tr>
<td>C</td>
<td>La, Ce, Pr, Nd, Sm, Eu, Dy, Er</td>
<td>0.4–2.8 MeV/amu</td>
<td>$L_1, L_2, L_3$</td>
<td>Braziewicz et al. [14] (1994)</td>
</tr>
<tr>
<td>C,N</td>
<td>Hf, Ta, W, Os, Ir, Pt, Au, Bi, Th</td>
<td>0.4–1.8 MeV/amu</td>
<td>$L_1, L_2, L_3$</td>
<td>Semaniak et al. [16] (1995)</td>
</tr>
<tr>
<td>O</td>
<td>Ni, Cu, Zn, Ga, Ge</td>
<td>12 MeV</td>
<td>$L_{\text{total}}$</td>
<td>Yu et al. [17] (1999)</td>
</tr>
<tr>
<td>O</td>
<td>Au, Bi, Th, U</td>
<td>6.4–70 MeV</td>
<td>$L_1, L_2, L_3, L_{\alpha_1,2}, L_{\gamma_1}, L_{\gamma_2,3,4}$</td>
<td>Pajek et al. [18] (2003)</td>
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<tr>
<td>C</td>
<td>Re, Pt, Au</td>
<td>4–8 MeV</td>
<td>$L_1, L_2, L_3, L_{\alpha_1}, L_{\gamma_1}, L_{\gamma_2,3,4}$</td>
<td>Lapicki et al. [19] (2004)</td>
</tr>
<tr>
<td>C</td>
<td>Ce, Nd, Lu</td>
<td>4–10 MeV</td>
<td>$L_1, L_2, L_3, L_{\alpha_1}, L_{\gamma_1}, L_{\gamma_2,3,4}$</td>
<td>Lapicki et al. [20] (2005)</td>
</tr>
</tbody>
</table>
energies 35–60 MeV with charge states 4+ and 5+, respectively, were incident on the targets positioned at 45° to the beam direction in a scattering chamber. A low-energy HPGe detector (30 mm² × 5 mm, Canberra) was placed outside the target chamber with 20 μm thick Mylar window in the side port at 90° to the beam direction. To reduce the low-energy peak tailing that results from incomplete charge collection at the edge of the detector crystal, a 2 mm thick aluminum collimator with 5 mm hole was used in front of the x-ray detector. The distance between target and the detector crystal was 140 mm. The experimental L x-ray production cross sections for the peaks of the L-shell spectrum were evaluated using the relation

\[ \sigma = \frac{Y A}{N_A e N_t b} \]  

(1)

where \( Y \) is the number of net counts under the L x-ray peak, \( A \) is the atomic mass (in grams) of the target, \( N_A \) is the Avogadro’s number, \( e \) is the absolute detector efficiency (including all absorbing components of the set-up) for the x-ray peak, and \( N \) is the number of incident ions collected in the Faraday cup and counted with a charge integrator. With the target thickness \( t \) (in g/cm²) as

Table 2

<table>
<thead>
<tr>
<th>( Z_1/Z_2 )</th>
<th>Element</th>
<th>Projectile</th>
<th>( E_1 ) (MeV)</th>
<th>( v_1/v_2 )</th>
<th>Experiment</th>
<th>Expt/ECPPSSR</th>
<th>Expt/FBA</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.105</td>
<td>( ^{57}\text{La} )</td>
<td>( ^{6}\text{C} )</td>
<td>35</td>
<td>0.41</td>
<td>29.2 ± 10</td>
<td>2.43</td>
<td>1.57</td>
</tr>
<tr>
<td>0.105</td>
<td>( ^{57}\text{La} )</td>
<td>( ^{6}\text{C} )</td>
<td>45</td>
<td>0.46</td>
<td>30.8 ± 10</td>
<td>1.74</td>
<td>1.28</td>
</tr>
<tr>
<td>0.105</td>
<td>( ^{57}\text{La} )</td>
<td>( ^{6}\text{C} )</td>
<td>50</td>
<td>0.49</td>
<td>40.2 ± 13</td>
<td>1.99</td>
<td>1.53</td>
</tr>
<tr>
<td>0.103</td>
<td>( ^{58}\text{Ce} )</td>
<td>( ^{6}\text{C} )</td>
<td>35</td>
<td>0.40</td>
<td>16.4 ± 5</td>
<td>1.49</td>
<td>0.95</td>
</tr>
<tr>
<td>0.103</td>
<td>( ^{58}\text{Ce} )</td>
<td>( ^{6}\text{C} )</td>
<td>45</td>
<td>0.46</td>
<td>18.7 ± 6</td>
<td>1.15</td>
<td>0.83</td>
</tr>
<tr>
<td>0.103</td>
<td>( ^{58}\text{Ce} )</td>
<td>( ^{6}\text{C} )</td>
<td>50</td>
<td>0.48</td>
<td>23.8 ± 8</td>
<td>1.33</td>
<td>0.96</td>
</tr>
<tr>
<td>0.100</td>
<td>( ^{60}\text{Nd} )</td>
<td>( ^{6}\text{C} )</td>
<td>35</td>
<td>0.39</td>
<td>8.6 ± 3</td>
<td>0.93</td>
<td>0.59</td>
</tr>
<tr>
<td>0.100</td>
<td>( ^{60}\text{Nd} )</td>
<td>( ^{6}\text{C} )</td>
<td>45</td>
<td>0.44</td>
<td>12.6 ± 4</td>
<td>0.90</td>
<td>0.64</td>
</tr>
<tr>
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<td>( ^{6}\text{C} )</td>
<td>50</td>
<td>0.46</td>
<td>15.6 ± 5</td>
<td>0.96</td>
<td>0.72</td>
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<tr>
<td>0.097</td>
<td>( ^{62}\text{Sm} )</td>
<td>( ^{6}\text{C} )</td>
<td>35</td>
<td>0.37</td>
<td>8.7 ± 3</td>
<td>1.13</td>
<td>0.69</td>
</tr>
<tr>
<td>0.097</td>
<td>( ^{62}\text{Sm} )</td>
<td>( ^{6}\text{C} )</td>
<td>45</td>
<td>0.42</td>
<td>10.8 ± 4</td>
<td>0.90</td>
<td>0.63</td>
</tr>
<tr>
<td>0.097</td>
<td>( ^{62}\text{Sm} )</td>
<td>( ^{6}\text{C} )</td>
<td>50</td>
<td>0.45</td>
<td>15.3 ± 5</td>
<td>1.09</td>
<td>0.80</td>
</tr>
<tr>
<td>0.140</td>
<td>( ^{57}\text{La} )</td>
<td>( ^{8}\text{O} )</td>
<td>35</td>
<td>0.35</td>
<td>52.1 ± 17</td>
<td>4.87</td>
<td>2.06</td>
</tr>
<tr>
<td>0.140</td>
<td>( ^{57}\text{La} )</td>
<td>( ^{8}\text{O} )</td>
<td>50</td>
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<td>72.4 ± 24</td>
<td>3.31</td>
<td>1.89</td>
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<tr>
<td>0.140</td>
<td>( ^{57}\text{La} )</td>
<td>( ^{8}\text{O} )</td>
<td>60</td>
<td>0.46</td>
<td>105 ± 35</td>
<td>3.58</td>
<td>2.32</td>
</tr>
<tr>
<td>0.138</td>
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<td>( ^{8}\text{O} )</td>
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<td>22.5 ± 8</td>
<td>2.34</td>
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<tr>
<td>0.138</td>
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<td>( ^{8}\text{O} )</td>
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<td>41.7 ± 14</td>
<td>2.10</td>
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<tr>
<td>0.138</td>
<td>( ^{58}\text{Ce} )</td>
<td>( ^{8}\text{O} )</td>
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<td>66.6 ± 22</td>
<td>2.48</td>
<td>1.57</td>
</tr>
<tr>
<td>0.133</td>
<td>( ^{60}\text{Nd} )</td>
<td>( ^{8}\text{O} )</td>
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<td>0.34</td>
<td>15.5 ± 5</td>
<td>1.99</td>
<td>0.81</td>
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<tr>
<td>0.133</td>
<td>( ^{60}\text{Nd} )</td>
<td>( ^{8}\text{O} )</td>
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<td>0.40</td>
<td>28.1 ± 9</td>
<td>1.69</td>
<td>0.92</td>
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<td>( ^{8}\text{O} )</td>
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<td>0.44</td>
<td>39.6 ± 13</td>
<td>1.74</td>
<td>1.08</td>
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<tr>
<td>0.129</td>
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<td>( ^{8}\text{O} )</td>
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<td>0.32</td>
<td>11.5 ± 4</td>
<td>1.83</td>
<td>0.72</td>
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<tr>
<td>0.129</td>
<td>( ^{62}\text{Sm} )</td>
<td>( ^{8}\text{O} )</td>
<td>50</td>
<td>0.39</td>
<td>23.5 ± 8</td>
<td>1.69</td>
<td>0.90</td>
</tr>
<tr>
<td>0.129</td>
<td>( ^{62}\text{Sm} )</td>
<td>( ^{8}\text{O} )</td>
<td>60</td>
<td>0.42</td>
<td>35.9 ± 12</td>
<td>1.85</td>
<td>1.12</td>
</tr>
</tbody>
</table>
seen by the beam and the x-ray attenuation coefficient \(\mu\) (in \(cm^2/g\)) [21], the correction factor 
\[ \beta \equiv \frac{1-\exp(-\mu l)}{\mu l} \] for absorption of the emitted L x-rays in all measurements was \(\geq 0.98\). Energy loss of the incident ions in the present targets of thickness 20–60 \(\mu g/cm^2\) is less than 0.3%. The absolute x-ray detection efficiency \(\varepsilon\) of the HPGe detector calibrated by 14.4 keV \(\gamma\)-rays and the most prominent x-rays from radioactive sources of \(^{54}\)Cr, \(^{55}\)Fe, \(^{57}\)Co and \(^{241}\)Am placed at the target position. The efficiency points measured at {3.30, 5.41, 5.89, 7.06, 11.9, 13.9, 14.4, 17.7} keV with {Am(M\(_a\)), Cr(K\(_a\)), Fe(K\(_a\)), Co(K\(_a\)), Co(K\(_b\)), Am(L\(_a\)), Am(L\(_b\)), Co(\gamma-ray), Am(L\(_b\))} were fitted to within 13% with a polynomial shown in Fig. 1. Although for analysis of our L x-ray spectra \(\varepsilon\) was only required below 8 keV, the detector was also calibrated at higher energies to ensure that the required absolute values of \(\varepsilon\) are accurately normalized. The error in the measured cross sections is conservatively estimated at 30–40% and is attributed to the uncertainties in the peak area evaluation, charge collection, target thickness, \(\beta\) evaluation, and the largest error contributor – the uncertain and steep change by a factor 4 in the detector efficiency within the 3–8 keV range of our L x-ray spectra. Recent work [22] on the response functions of solid-state detectors points to the presence of peaks and backgrounds that due to pileup, signal processing and other mechanisms may seriously obscure the true nature of the measured x-ray spectra. It would be of interest to re-evaluate the extraction of the x-ray cross sections from our 3–8 keV spectra with the formulas derived by Papp [22] for the response function of the HPGe detector to low-energy (2–20 keV) x-rays.

3. Results and conclusion

The measured L x-ray production cross-sections of La, Ce, Nd and Sm for 35 MeV, 45 MeV and 50 MeV C\(^{\text{4+}}\) ions and for 35 MeV, 50 MeV and 60 MeV O\(^{\text{5+}}\) ions are tabulated in Table 2 along with their ratios to the predictions of the ECPSSR and FBA. These cross sections are plotted as a function of \(v_1/v_{2L}\) for C\(^{\text{4+}}\) and O\(^{\text{5+}}\) ions in the 3–8 keV range. Recent work [22] on the response functions of solid-state detectors points to the presence of peaks and backgrounds that due to pileup, signal processing and other mechanisms may seriously obscure the true nature of the measured x-ray spectra. It would be of interest to re-evaluate the extraction of the x-ray cross sections from our 3–8 keV spectra with the formulas derived by Papp [22] for the response function of the HPGe detector to low-energy (2–20 keV) x-rays.

![Fig. 2](image-url) Fig. 2. Measured L-shell x-ray production cross sections versus \(v_1/v_{2L}\) for \(^{57}\)La, \(^{58}\)Ce, \(^{60}\)Nd and \(^{62}\)Sm at 35 MeV, 45 MeV and 50 MeV C\(^{\text{4+}}\) ion beam and 35 MeV, 50 MeV and 60 MeV O\(^{\text{5+}}\) ion beam. Also shown are cross sections of \(^{58}\)Ce and \(^{62}\)Sm for 36 MeV O\(^{\text{5+}}\) ion beam from [8]. The data for \(^{58}\)Ce, \(^{60}\)Nd and \(^{62}\)Sm bombarded by carbon ions from [14] are not shown, because they were reported only after conversion to ionization cross sections.

As shown in Figs. 3 and 4, the measured cross sections in general, increase with the increase in \(v_1/v_{2L}\) and \(Z_1/Z_2\). For C\(^{\text{4+}}\) projectiles, the FBA predicts values that are closer than the ECPSSR in case of La and Ce, while for Nd and Sm the opposite is observed as shown in Figs. 2 and 3. For O\(^{\text{5+}}\) projectiles, the FBA calculations are in better agreement with our measurements for all elements. Cross sections for Ce and Sm by O\(^{\text{5+}}\) that were measured three decades ago [8] are significantly smaller than ours and in closer agreement with the ECPSSR theory. From Table 2...
keeping the velocity ratio \( v_1/v_{2L} \) and the target atomic number \( Z_2 \) unchanged, there is a remarkable increase in the total x-ray production cross sections for the heavier projectile i.e. for \( \text{O}^{5+} \) as compared to \( \text{C}^{4+} \).

The measured L x-ray production cross sections in general increase with energy as shown in Table 2 for the \( \text{C}^{4+} \) and \( \text{O}^{5+} \) ions, respectively. This is also predicted by both the FBA and ECPSSR theories. In fact, the FBA gives a larger contribution of EC as compared to the ECPSSR theory in our energy region [the EC/(EC + DI) percentages for the FBA range from 5% to 18%, while the same percentages in ECPSSR are between 0.8% and 3%] and thus the agreement with the FBA theory in case of \( \text{O}^{5+} \) may be attributed to the increased probability of the formation of the target L-shell vacancies created due to the electron capture into the L- and higher shells of the projectile. Andrews et al. [10] have shown that for projectile with one K-shell electron or bare projectile, without K-shell electrons, the target x-ray production cross sections are significantly enhanced over those for projectile with two or more electrons. The incident \( \text{O}^{5+} \) could well be stripped of its electrons, especially at higher energies; for fully-stripped oxygen, the EC/(EC + DI) percentages for the FBA would have ranged from 42% to 62%, while the same percentages in ECPSSR would be from 14% to 23%.

For \( \text{C}^{4+} \) ions, the ECPSSR gives a better agreement for Nd and Sm, while for La and Ce, the FBA yields better results. For \( \text{O}^{5+} \) ions, the measured cross sections for all the target elements are in closer agreement with the FBA values than the ECPSSR theory. The discrepancy between theory and experiment increases with energy and \( Z_1/Z_2 \), which may be attributed to multiple ionizations (for which no correction was made) and an underestimate of the EC contribution as \( Z_1/Z_2 \) increases. More extensive study and experiments – that would address problems associated with the HPGe efficiency in the difficult range of 3–8 keV spectra – are needed for better understanding of inner shell ionization by heavy ions.
Acknowledgements

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References

PIXE ANALYSIS AND COMPARISON OF AEROSOL SAMPLES FROM RELATIVELY CLEAN AND INDUSTRIALLY POLLUTED CITIES IN NORTHERN INDIA

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ABSTRACT
Particle induced X-ray emission (PIXE) technique developed at Variable Energy Cyclotron, Punjab University Chandigarh has been used for the detection of trace elements in the aerosol samples collected from relatively clean and industrially polluted city in northern India. Aerosol samples were collected from Jammu City of Jammu and Kashmir State, which is supposed to have a clean environment and from Mandi-Gobindgarh, one of the most polluted steel city of Punjab. The results from the analysis of these samples show some basic differences in the trace element profile of the two cities. The results further suggest a long term aerosol sample analysis to segregate anthropogenic and natural sources of air pollution. The mean concentration levels of different trace elements in PM_{10} (Particulate matter with aerodynamic diameter less than 10 μm) from these two cities of northern India are compared with some of the prominent cities of India.

Keywords: Aerosols; PM_{10}, PM_{2.5}, PIXE; trace elements.

1. Introduction

PIXE has established itself as a routine analytical technique in the study of atmospheric aerosols. The reason for this is the basic advantage of PIXE being a multi-elemental, sensitive, fast and non-destructive [1, 2]. Many research groups in world like Cahill group in California [3], PIXE group at Lund Institute of Technology in Sweden [4], Sao Paulo group in South America [5] and Milan group in Italy [6] are carrying elemental analysis of the aerosol samples collected from thickly populated urban areas and industrial centers. Data of elemental concentration of aerosols help to estimate the impact
of various natural and anthropogenic pollution sources on the ambient air quality. There is also an increasing awareness of the association of aerosols with the cardiovascular diseases and respiratory disorders [7].

In India data on elemental concentration of aerosol is scarce. However, the need for such measurements cannot be overemphasized due to the recent growth in industrialization, increasing rural urban migration and increasing vehicular density in cities. Recently we have developed a regional PIXE facility at Cyclotron Laboratory, Panjab University, Chandigarh [8]. This facility is one of the few facilities available in the country for elemental analysis of aerosol samples.

Keeping in view the above consideration, in the present paper, we report the preliminary results on the PIXE analysis of the aerosol samples from the Jammu city, having a relatively cleaner environment in comparison with the Mandi-Gobindgarh, one of the most polluted industrial cities of the Punjab having a large number of steel furnaces.

2. Experimental Method

2.1. Sample collection

Twelve aerosol samples for analysis were collected from six different sites of Jammu City by the Department of Environmental Sciences, Jammu University, Jammu, India in the year 2000. Site description along with sampling parameters and Suspended Particulate Matter (SPM), mainly with particulate matter of aerodynamic diameter less than 10 μm (henceforth defined as PM₁₀) are given in Table 1. These samples were collected with a high-volume sampler which is a two-stage high-speed blower capable of sampling ~1.1 m³/min through an EPM-2000 grade standard glass fiber filter (200×250 mm²). These blowers, mounted in protective shelter, have been used as the standard samplers for particulate matter for the National Air Sampling Network (NASN) [9]. They have a bypass rotameter or pressure tap for monitoring flow rate. The EPM-2000 grade paper was developed and produced especially for use in high volume air sampling equipment that collects atmospheric particulates and aerosols. It is manufactured from 100% pure borosilicate glass. EPM-2000 was selected by the Environmental Protection Agency (EPA) to be the standard filter for use in the nationwide network of Hi-Vol air samplers. The sampling was carried at the roof tops 40-60 feet. The PM₁₀ level in case of Jammu region is calculated using the relation

\[ \text{PM}_{10} (\mu g/m^3) = \frac{\text{Wt. of loaded filter (g) - Wt. of blank filter (g)}}{\text{Flow rate} (m^3/min) \times \text{Sampling time} (min)} \]

Five aerosol samples were collected from Mandi-Gobindgarh region by the Department of Community Medicine, Post Graduate Institute of Medical Education and Research (PGIMER) Chandigarh, India in the year 2000. These aerosol samples were collected using aerosol-sampling kit, which involves Millipore diaphragmatic vacuum
pump and Sequential Filter Unit (SFU) with coarse pored Nuclepore® polycarbonate filters for collection of fractionated samples of particulate matter under ambient conditions. With this sampler, outdoor aerosol can be separated in two fractions, which roughly can be described as fine fraction PM_{10} (diameter smaller than 2.5 μm) and coarse fraction PM_{2.5} (aerodynamic diameter between 2.5 to 10 μm). PM_{10} is the sum of PM_{2.5} and PM_{2.5}. The SFU consisted of 8 μm pore size filter of 25 mm diameter for PM_{2.5} followed by a 0.4 μm pore size filter of same diameter for PM_{2.5}, both held in a double filter holder of 25 mm diameter (Nuclepore Corp, Pleasanton, CA, USA) [10]. All the aerosol-sampling sites were located on the flat rooftops of building (20-40 feet high) to have an effective collection of the PM_{2.5} and PM_{2.5} aerosols.

Table 1. Site description of different sampling sites along with sampling parameters and PM_{10} level at various sites of Jammu region.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Site Name</th>
<th>Sampling Time (hrs)</th>
<th>Average Flow Rate (m³/min)</th>
<th>Initial Weight of Filter Paper (grams)</th>
<th>Final Weight of Filter Paper (grams)</th>
<th>PM_{10} (μg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-1</td>
<td>Department of Environmental Sciences</td>
<td>24</td>
<td>1.3</td>
<td>2.6835</td>
<td>2.8609</td>
<td>118</td>
</tr>
<tr>
<td>S-2</td>
<td>-do-</td>
<td>22</td>
<td>1.2</td>
<td>2.6980</td>
<td>2.8462</td>
<td>91</td>
</tr>
<tr>
<td>S-3</td>
<td>Mahesh pura Chowk</td>
<td>10</td>
<td>1.1</td>
<td>2.6940</td>
<td>2.9757</td>
<td>427</td>
</tr>
<tr>
<td>S-4</td>
<td>-do-</td>
<td>7</td>
<td>1.1</td>
<td>2.7089</td>
<td>3.0110</td>
<td>615</td>
</tr>
<tr>
<td>S-5</td>
<td>Police Line</td>
<td>6</td>
<td>1.1</td>
<td>2.6860</td>
<td>2.9533</td>
<td>691</td>
</tr>
<tr>
<td>S-6</td>
<td>-do-</td>
<td>6</td>
<td>1.0</td>
<td>2.6873</td>
<td>2.9053</td>
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<td>S-7</td>
<td>Old Hospital Road</td>
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<td>1.2</td>
<td>2.7174</td>
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<td>S-9</td>
<td>Talab Tilla</td>
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<td>-do-</td>
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<td>S-11</td>
<td>Satwari Chowk</td>
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<td>2.7005</td>
<td>2.9444</td>
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<tr>
<td>S-12</td>
<td>-do-</td>
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<td>1.0</td>
<td>2.6890</td>
<td>2.9168</td>
<td>633</td>
</tr>
</tbody>
</table>

The air through the filter membrane was sucked at a required flow rate with the help of a diaphragmatic vacuum pump (Millipore No. 5522050). The flow rate was kept constant by a critical orifice during the sampling period. The collection surface was directed downward to prevent particle collection by sedimentation and the filter holder was protected with a rain cover. The sampling time for the sample was 12 hrs with flow rate of 8 ltr/min.

2.2. Experimental details

The aerosol samples were collected with SFU on polycarbonate filters from Mandi-Gobindgarh and were as such fixed on the target holder wheel having provision for 12 targets of 25 mm diameter each. In case of aerosol samples from Jammu region which are on glass fiber sheets of dimensions (200 × 250 mm²), the sample of size 30 × 30 mm² were taken diagonally at three points to get the distribution representation across the total
filter paper which we found is uniform. The collected aerosol samples were irradiated by 3 MeV proton beam from Variable Energy Cyclotron (VEC), Panjab University, Chandigarh [11, 12]. The targets were oriented at the angle of 90° and the X-ray detector was at the angle of 135° to the beam line. The beam is passed through a carbon collimator (d=2 mm) before reaching the target. The beam current was kept below 10 nA. Characteristic X-rays were detected with a low energy HPGe detector with energy resolution of 135 eV at 5.9 keV. Data acquisition was done with PC based Multichannel Analyzer and the peak areas were calculated with the help of the computer software i.e. Personal Computer Analyzer-II (PCA-II).

2.3. Data analysis

The basis for the quantitative analysis is the relationship between the net area of an element's characteristic K or L X-ray photo peak line in the X-ray spectrum and the amount of element present in the sample. For proton bombardment and thin specimen, this relationship is given by the equation:

\[ C_{Z} \rho t = \frac{Y(Z) A_{Z} \sin \theta}{N_{A} \sigma_{p}(E_{p}) \varepsilon_{abs} p_{a}}. \]

Where, \( Y(Z) \) is the number of counts in the characteristic X-ray line of the analyte element with atomic number \( Z \), \( N_{A} \) Avogadro's number, \( \sigma_{p}(E_{p}) \) the X-ray production cross section (barn/atom) at the incident proton energy \( E_{p} \), \( \varepsilon_{abs} \) the absolute detection efficiency (including all absorbing components of the set up) for X-ray line, \( p_{a} \) the number of incident protons, \( A_{Z} \) the atomic mass of the analyte element and \( \theta \) the angle between the incident proton beam and the specimen surface, \( C_{Z} \) the concentration of the analyte element in the specimen, \( \rho \) the specimen density (g/cm\(^3\)), \( t \) the specimen thickness (cm). The product \( C_{Z}\rho t \) gives the concentration of the element in g/cm\(^3\).

The measurement and the evaluation of different parameters used in above equation for our PIXE setup are described by Puri et al. [13]. The theoretical X-ray production cross section \( \sigma_{p}(E_{p}) \) were calculated using the equations given by Close et al [14]. \( p_{a} \) was evaluated from the charge collected in the Faraday cup and absolute detector efficiency of the Low Energy HPGe detector has been experimentally measured using thin foils of Al (376 Å), Sc (230 Å), Ti (208 Å), Fe (149 Å), Cu (202 Å), Ge (240 Å) bombarded by 3 MeV protons and \(^{241}\)Am radioactive source. In the above expression, different parameters were rearranged to evaluate the \( \varepsilon_{abs} \) with thin foils. The intensities of different X-ray and gamma ray lines for \(^{241}\)Am were taken from Table of Isotopes [15]. The efficiency curve of the detector is given in Figure 1.

Elemental concentration in g/cm\(^2\) (on filter) is converted to g/m\(^3\) (in air) using relation:

\[
\text{Conc. in g/m}^3 = \frac{\text{Conc. on filter (g/cm}^2\text{)} \times \text{Area of the filter (cm}^2\text{)}}{\text{Flow rate (m}^3\text{/min)} \times \text{Sampling time (min)}}
\]
3. Result and Discussion

Typical X-ray spectra of the aerosol samples collected from Jammu region are shown in figure 2. Figure 2a indicates the trace elements present in the glass fiber filter while figure 2b shows the trace elements present in a typical aerosol sample from site S-4 as described in table 1 and figure 2c shows the spectrum obtained after subtraction of spectrum shown in figure 2a from spectrum shown in figure 2b. The maximum PM$_{10}$ level was found to be 1033 µg/m$^3$ in this region, which is higher as compared to the maximum PM$_{10}$ level of Mandi-Govindgarh region of 700 µg/m$^3$ as reported by Punjab State Council for Science and Technology [16].

The blank glass fiber filters have their own trace elements hence, the concentrations of these trace elements were estimated by irradiating blank filters. In the final tabulation (Table 2) the concentration from blank filters were therefore subtracted from the estimated concentration of the aerosol samples for a fixed charge collected in the faraday cup. After the subtraction of the background from the glass fiber filter, the elements S,
Cl, Ca, Fe and Cu were detected beyond doubt. However, the concentrations of elements like Si, K, Zn, Ba, Sr and Zr were found to be negligible, hence these elements are not tabulated in Table 2. The concentrations of Ca and Fe were found to be much higher as compared to other elements indicating typically the environment to be dusty in the Jammu region.

The X-ray spectra of the aerosol samples collected from Mandi-Gobindgarh region are shown in Figures 3 and 4. Figure 3a shows the X-ray spectrum of polycarbonate fiber and figure 3b shows the spectrum of PM$_{2.5}$, which indicates the presence of Si, Cl, K, Ca, Ti, V, Cr, Mn, Fe and Zn. Figure 4a shows the spectrum of blank filter and figure 4b shows the spectrum of PM$_{2.5}$, which indicates the presence of Si, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Zn, Br and Pb. The particulate matter PM$_{2.5}$ and PM$_{1.0}$ have common trace elements.

Figure 2. X-ray spectra of: (a) glass fiber filter, (b) aerosol sample from Jammu region collected on the glass fiber filter, (c) spectrum obtained after subtraction of (a) from (b).
Table 2. Average concentration (from two same sites) along with their standard deviations in ng/m$^3$ of trace elements in PM$_{10}$ of Jammu region.

<table>
<thead>
<tr>
<th>Element</th>
<th>S-1.2</th>
<th>S-3.4</th>
<th>S-5.6</th>
<th>S-7.8</th>
<th>S-9.70</th>
<th>S-11.12</th>
<th>Mean</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>S</td>
<td>134 ± 78</td>
<td>48 ± 20</td>
<td>87 ± 21</td>
<td>192 ± 103</td>
<td>41 ± 17</td>
<td>166 ± 101</td>
<td>111 ± 63</td>
<td></td>
</tr>
<tr>
<td>Cl</td>
<td>439 ± 192</td>
<td>224 ± 73</td>
<td>404 ± 179</td>
<td>430 ± 169</td>
<td>188 ± 153</td>
<td>361 ± 261</td>
<td>341 ± 169</td>
<td></td>
</tr>
<tr>
<td>Ca</td>
<td>9698 ± 7270</td>
<td>4299 ± 3291</td>
<td>9624 ± 4583</td>
<td>10297 ± 4747</td>
<td>5061 ± 3241</td>
<td>5528 ± 3331</td>
<td>7418 ± 2728</td>
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</tr>
<tr>
<td>Fe</td>
<td>7799 ± 3160</td>
<td>5037 ± 1858</td>
<td>8392 ± 6123</td>
<td>10398 ± 1971</td>
<td>9401 ± 1958</td>
<td>4321 ± 3365</td>
<td>7591 ± 2426</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>100 ± 48</td>
<td>96 ± 24</td>
<td>140 ± 68</td>
<td>113 ± 44</td>
<td>72 ± 56</td>
<td>113 ± 63</td>
<td>106 ± 23</td>
<td></td>
</tr>
</tbody>
</table>

Figure 3. X-ray spectra of: (a) polycarbonate filter, (b) aerosol sample of PM$_{10}$ from Mandi-Cobindgarh.
Figure 4. X-ray spectra of: (a) polycarbonate filter, (b) aerosol sample of PM$_{2.5}$ from Mandi-Gobindgarh.

namely S, Cl, K, Ca, Ti, V, Cr, Mn, Fe and Zn. However, Br and Pb were present only in PM$_{2.5}$. This observation clearly shows that while other elements have coarse as well as fine particulate matter but Br and Pb are present in the form of fine powder only. From table 3, we observe that the concentrations of K, Ca, Ti, V and Fe are higher in PM$_{CSP}$ as compared to PM$_{2.5}$ while concentration of S, Cl, Cr, Mn and Zn are higher in PM$_{2.5}$ as compared to PM$_{CSP}$ indicating that these elements to be present more in the form of fine powder. The higher levels of Ca and Fe in Jammu as compared to Mandi-Gobindgarh indicate more suspended natural dust in Jammu. S levels in Mandi-Gobindgarh are higher than Jammu shows higher industrial activities. Pb and Br are completely absent in Jammu region and their concentrations in Mandi-Gobindgarh are higher than Jammu again indicating industrial activities.

The overall error in the estimation of trace element concentrations is about 15%. This is attributed due to the errors incurred due to the flow rate during sampling and the X-ray photopeak statistics. The mean concentration levels of different trace elements for Jammu and Mandi-Gobindgarh region of northern India are compared with some of the prominent cities of India [17] in Table 4.
Table 3. Elemental concentration in ng/m³ of trace elements in PM$_{2.5}$, PM$_{10}$ and total PM$_{10}$ in aerosol samples collected from Mandi-Gobindgarh region.

<table>
<thead>
<tr>
<th>Element</th>
<th>PM$_{2.5}$</th>
<th>PM$_{10}$</th>
<th>PM$_{10}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>S</td>
<td>70 ± 32</td>
<td>276 ± 66</td>
<td>346 ± 73</td>
</tr>
<tr>
<td>Cl</td>
<td>23 ± 5</td>
<td>256 ± 48</td>
<td>279 ± 48</td>
</tr>
<tr>
<td>K</td>
<td>741 ± 428</td>
<td>649 ± 157</td>
<td>1391 ± 456</td>
</tr>
<tr>
<td>Ca</td>
<td>1780 ± 1136</td>
<td>285 ± 92</td>
<td>2056 ± 1140</td>
</tr>
<tr>
<td>Ti</td>
<td>166 ± 103</td>
<td>14 ± 5</td>
<td>180 ± 104</td>
</tr>
<tr>
<td>V</td>
<td>31 ± 15</td>
<td>29 ± 13</td>
<td>59 ± 20</td>
</tr>
<tr>
<td>Cr</td>
<td>17 ± 10</td>
<td>20 ± 5</td>
<td>38 ± 11</td>
</tr>
<tr>
<td>Mn</td>
<td>44 ± 14</td>
<td>69 ± 68</td>
<td>113 ± 70</td>
</tr>
<tr>
<td>Fe</td>
<td>3022 ± 1985</td>
<td>1516 ± 1705</td>
<td>4538 ± 2617</td>
</tr>
<tr>
<td>Br</td>
<td>-</td>
<td>205 ± 47</td>
<td>205 ± 47</td>
</tr>
<tr>
<td>Pb</td>
<td>-</td>
<td>532 ± 318</td>
<td>532 ± 318</td>
</tr>
</tbody>
</table>

Table 4. Comparative study of elemental concentration in ng/m³ of total PM$_{10}$ from Mandi-Gobindgarh, Chandigarh, Jaipur and Jammu region of India.

<table>
<thead>
<tr>
<th>Element</th>
<th>Mandi-Gobindgarh (exp.)</th>
<th>Chandigarh (ref. 17)</th>
<th>Jaipur (ref. 17)</th>
<th>Jammu (exp.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>-</td>
<td>-</td>
<td>7610 ± 2460</td>
<td>-</td>
</tr>
<tr>
<td>Si</td>
<td>-</td>
<td>-</td>
<td>14400 ± 3320</td>
<td>-</td>
</tr>
<tr>
<td>S</td>
<td>346 ± 73</td>
<td>4702 ± 3855</td>
<td>440 ± 1880</td>
<td>111 ± 63</td>
</tr>
<tr>
<td>Cl</td>
<td>279 ± 48</td>
<td>3015 ± 2608</td>
<td>1060 ± 2330</td>
<td>341 ± 109</td>
</tr>
<tr>
<td>K</td>
<td>1391 ± 456</td>
<td>4633 ± 4768</td>
<td>1710 ± 3020</td>
<td>-</td>
</tr>
<tr>
<td>Ca</td>
<td>2065 ± 1149</td>
<td>7827 ± 8699</td>
<td>5000 ± 2290</td>
<td>7416 ± 2728</td>
</tr>
<tr>
<td>Ti</td>
<td>180 ± 108</td>
<td>599 ± 703</td>
<td>390 ± 3010</td>
<td>-</td>
</tr>
<tr>
<td>V</td>
<td>59 ± 20</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Cr</td>
<td>35 ± 11</td>
<td>47 ± 73</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Mn</td>
<td>113 ± 70</td>
<td>318 ± 381</td>
<td>100 ± 2320</td>
<td>-</td>
</tr>
<tr>
<td>Fe</td>
<td>4538 ± 2617</td>
<td>7720 ± 9394</td>
<td>4450 ± 2420</td>
<td>7591 ± 2426</td>
</tr>
<tr>
<td>Ni</td>
<td>-</td>
<td>14 ± 17</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Cu</td>
<td>-</td>
<td>32 ± 44</td>
<td>57 ± 2030</td>
<td>106 ± 23</td>
</tr>
<tr>
<td>Zn</td>
<td>2291 ± 2085</td>
<td>1042 ± 1484</td>
<td>170 ± 2460</td>
<td>-</td>
</tr>
<tr>
<td>Br</td>
<td>205 ± 47</td>
<td>27 ± 12</td>
<td>12 ± 13</td>
<td>-</td>
</tr>
<tr>
<td>Sr</td>
<td>-</td>
<td>62 ± 82</td>
<td>39 ± 2190</td>
<td>-</td>
</tr>
<tr>
<td>Pb</td>
<td>532 ± 318</td>
<td>1225 ± 1910</td>
<td>80 ± 1810</td>
<td>-</td>
</tr>
</tbody>
</table>
It is clear from the Table 4 that concentration of trace element namely Br from Mandi-Gobindgarh region is found to be higher as compared with those measured at Chandigarh and Jaipur, which has higher level of out door air pollution due to Industrial activities. The concentration values of trace elements namely Ca and Fe from Jammu region are found to be comparable with those measured at Chandigarh and Jaipur, which are comparatively clean cities in India.

4. Conclusions

We have analyzed the aerosol samples of the two important cities of India viz. Jammu, which is comparatively cleaner and Mandi-Gobindgarh, which is a highly polluted industrial city in Punjab region. In case of samples from the Jammu region, the concentration of the trace element Ca is higher by a factor of 15 and Fe by a factor of approximately 3 as compared to the Mandi-Gobindgarh region. This indicates the dusty environment of Jammu region with the major source of pollution from the soil dust [18]. This is further confirmed since the maximum PM$_{10}$ level in Jammu region as measured by us is 1033 µg/m$^3$ which is higher as compared to the maximum PM$_{10}$ level of Mandi-Govindgarh region of 700 µg/m$^3$ as reported by the Punjab State Council for Science and Technology. The presence of trace elements like Ti, V, Cr, Mn, Br and Pb in the aerosol samples from Mandi-Gobindgarh region indicates that these are from the industrial activity of steel furnaces and are produced due to the burning and the rolling of the iron ingots. The higher concentration of Br in Mandi-Gobindgarh as compared to other cities in India shows the existence of high outdoor air pollution due to industrial activities. The concentration of Ca and Fe from Jammu are comparable with those reported from Chandigarh and Jaipur. Further investigations from industrial and non-industrial towns with large number of aerosol samples are needed in future.

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References

Measurement of wakefield intensity

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Abstract

We report on the measurement of the wakefield of an ion moving through a beam–foil plasma using an indirect experimental method. The standard beam–foil time-of-flight technique is used to measure the lifetime of the perturbed H-like V $2s^2S_1/2$ state due to its passage through the wakefield. This measured lifetime along with the theoretical lifetimes of pure H-like V $2s^2S_1/2$ and $2p^2P_1/2$ states has been used to determine the Stark mixing parameter of H-like $2s^2S_1/2$ and $2p^2P_1/2$ states, which is further exploited to determine the wakefield at the surface of the exit foil. The wakefield intensity during the passage of 160 MeV $^{23}$V ion through thin C-foil as well as Au-foil is found to be as high as $3.2 \pm 0.07 \times 10^7$ V cm$^{-1}$.

When an ion beam passes through a thin solid foil the beam–foil interaction yields a beam-foil plasma [1] in the foil. Subsequently, as rear charge particles of the same beam pass through this plasma, the beam scatters electrons, which causes an uneven distribution of charge between the scattered particles and the plasma ions. To restore an even distribution, the electrons are pulled back towards the positive plasma ions that have congregated towards the rear of the beam pulse. But the electrons overshoot their original positions, creating a wake-like disturbance called a wakefield oscillation at the surface. The existence of such a wake in the beam–foil plasma has been known for a long while [2, 3]. The wake-induced electric field at the projectile nucleus was found to be strong enough to produce measurable energy splitting of $n = 2$ manifold of light H-like ions [4]. Observation of anomalous substate populations of projectile ions emerging from solid targets was explained in terms of the wakefield-induced Stark mixing of the substates in a solid [5]. In order to know whether the wakefield is a surface effect or a bulk effect the following facts must be recalled: (i) the excited states are formed at the surface, not in the bulk [6] and (ii) the ion–solid interaction gives rise to a stopping power field in the bulk of the foil [7], not at the surface. Since the stopping power field cannot affect the excited states forming at the surface, the wakefield must be playing its role on the excited states. It is thus of great fundamental interest to quantify its strength in the laboratory. However, till today no quantitative determinations of wakefield intensities in the laboratory have been attempted.

The $2s^2S_1/2$ state of H-like ions is a long-lived state that decays to $1s^2S_1/2$ ground state either by two-photon emission or by single-photon M1 emission [8]. However, it is very close to the $2p^2P_1/2$ state, a suitable electric field can mix these two states resulting in a shorter lifetime for the perturbed $2s^2S_1/2$ state. Minimum electric field ($F$) required for complete Stark mixing of the H-like $2s^2S_1/2$ and $2p^2P_1/2$ states [9] can be estimated from $\Delta E = 3Fh(n-1)/Z$, where $\Delta E$ is the Lamb shift in cm$^{-1}$, $F$ in a.u. and $n$ is the principal quantum number. For example, the Lamb shift between the $2s^2S_1/2$ and $2p^2P_1/2$ states in H-like V ion is only 2980 cm$^{-1}$. The value of $F$ turns out to be 11.423 a.u. ($1.78 \times 10^8$ V cm$^{-1}$). If the above-mentioned wakefield can induce an electric field comparable to this value, partial Stark mixing might take place and its effect could be reflected in the measured lifetime. We measured the lifetime of the perturbed H-like V $2s^2S_1/2$ state of 47 $\pm$ 2 ps instead of 756.7 ps [10], the theoretical mean lifetime of the pure H-like V $2s^2S_1/2$ state. Using the theoretical lifetimes of pure H-like V $2s^2S_1/2$ and $2p^2P_1/2$ states, and the measured lifetime for the perturbed H-like V $2s^2S_1/2$ state, we determined the mixing parameter that could be exploited to evaluate the wakefield intensity.

The details of the experimental setup used in these measurements have been described in our earlier paper [11]. Briefly, the experiment was performed at the Inter-University Accelerator Center, New Delhi using the 15 UD tandem Pelletron accelerator. A beam of 160 MeV $^{24}$V$^{12+}$ ions passed through a 60 $\mu$g cm$^{-2}$ carbon foil to obtain the highest charge state [12] and subsequently to produce all kinds of excited states in each ion. A germanium ultra-low energy detector (GUL 0035, Canberra Inc.), having a resolution of 150 eV at 5.9 keV, was used without any absorber foil to measure the
x-ray spectra. Time-resolved x-ray spectra were recorded at different delay times (time taken to travel the distance between the foil and the detector slit) by changing the distance between the foil and the detector window with high precision (<1 μm). Thus, the error in delay-time measurements was restricted mainly to the uncertainty in determining the initial foil position (2.2 ± 0.1 mm). Silicon surface barrier detectors were used to monitor the incident ion beam by detecting the elastically scattered projectiles from an Au foil which was placed 10 mm downstream of the detector slit. Intensity decay curves were normalized using this method, which agree to within 3–5% with the charge normalization method using a deep Faraday cup. In addition, the recoil carbon atoms scattered by the projectiles were also used to verify the normalization method. Vacuum was maintained to better than 1 × 10⁻⁶ Torr. A liquid nitrogen trap was introduced in the vacuum chamber to reduce the foil thickening effect due to the hydrocarbons. At this point it may be worth noting that since the wakefield is formed at the exit surface, normally one would think that the experiment must be performed at ultra-high vacuum conditions. However, the normal incidence of the ion beam as used in the present work cleans up both entrance and exit surfaces in a few seconds due to annealing effects caused by ion energy loss at the foil.

The x-ray detector was placed at right angles to the beam axis. In order to get rid of the faster transitions, it was kept 2.2 ± 0.1 mm from the exiter foil. The x-ray spectra were calibrated with a ²⁴¹Am source. The spectra display a composite peak at 5.17 keV corresponding to three unresolved lines (namely, 1s2p⁴⁴P₀–1s²⁴S₀ (M2) line at 5.1758 keV and 1s2s³S₁–1s²⁴S₀ (M1) line at 5.1857 keV in He-like V and 1s2s2p⁴⁴P⁰–1s²²S₂1/2 (M2) line at 5.1423 keV in Li-like V [13]) is seen along with the 2E1 decays in H-like as well as He-like ions, a small peak at 5.42 keV and even a smaller peak at 6.1 keV (can be seen by expanding the intensity scale). To verify genuineness of the small peaks we reduced the beam energy to 100 MeV and saw the lines at 5.42 keV and 6.1 keV disappear completely. Further, on passing the beam through a blank foil holder, the spectrum shows a flat background to reveal that the lines do not originate from any experimental artefact. Since the lines are observed in the delayed spectra, they could only be attributed to the forbidden transitions. The theoretical value of He-like V M2 line 1s3p³P₂–1s²⁴S₀ is 6.114 keV and H-like V M1 line 2s²S₁/2–1s²S₁/2 is 5.427 keV [13].

In the next step, we recorded the x-ray spectra by keeping the C-foil at different distances from the detector window so that the mean lifetime of the upper state of the line could be determined. Intensities of the 5.42 keV line so obtained were normalized with respect to a fixed number of elastically scattered ions from the Au foil. The normalized x-ray line intensities have been plotted as a function of delay time that fit a single exponential with rms error of 0.089. The cause of cascade-free transition was considered as follows. Cascades through high-lying p states could repopulate the 2s²S₁/2 states downstream of the foil and distort the decay. The ground state decays np → 1s contribute as a tail to the x-ray spectrum in the region above 5.42 keV. However, the spectra hardly show such contribution. The reason is the low beam energy used in our experiment. Even cascade contribution at much higher beam energy was very small [14].

In order to identify the origin of the 5.42 keV peak one can consider that the ions may leave the foil in a number of excited states that may have been created by (i) direct excitation by charge transfer from the surface [15] and (ii) transition cascades from higher excited states. Although the energy resolution of the measured x-ray spectra is not sufficient to differentiate these two processes, however, the lifetime measurement gives a clear indication of the excitation process involved. If the transition cascades occur from non-circular states the decay curve shows a strong cascade fitting with a power law (see for example [16]). Whereas if the higher excited state belongs to circular states, transition cascades are yrast chain. Since they are unbranched, the decay curve displays a hump-like structure after a long while as shown in our recent experiments [17, 18]. The cause of the hump-like structure is the population of short-lived 2p²⁴P₁/₂ state occurring late due to the yrast chain. However, the observed decay curve did not show such trends; a simple one exponential decay trend favours the direct excitation process. Hence the weaker peak at 5.42 keV was assigned to H-like M1 transition (2s²S₁/2–1s²S₁/2). Our spectral analysis resembles well that of [19].

The single exponential fitting displays a lifetime of 49.3 ± 3 ps instead of 756.7 ps [10], theoretical mean lifetime of the H-like V 2s²S₁/2 state. The above experiment was carried out with C-foil; for a further check with other elemental targets we repeated the experiment with a 120 μg cm⁻² Au-foil. A typical x-ray spectrum obtained with 158 MeV ⁵¹V ion beam is shown in figure1(a). The measured lifetime (see figure 1(b)) was around the same (45.7 ± 3 ps) as that obtained with the C-foil. Although the foil materials were different, results show no difference indicating that the wakefield intensity at C- or Au-surfaces is nearly equal. The mean of these two measured values provides the lifetime of 47.5 ± 2 ps. Although the 5.42 keV peak is quite small compared to 5.17 keV line at 2.2 ± 0.1 mm, the relative intensity at their origin can be estimated using the lifetimes. The lifetime measured for 5.17 keV peak was 242 ± 5 ps [20] and the relative intensity of the two peaks turns out to be 2 ± 0.1 only.

We looked for any other effects which could also cause reduction of the 2s²S₁/2 lifetime. In view of this hyperfine mixing was considered: as ⁵¹V has a finite nuclear magnetic moment and a non-zero nuclear spin, hyperfine mixing can have no measurable effect on its decay rate as the 2s²S₁/2 state predominantly decays through the 2E1 emission channel [21]. Thus, such reduction in lifetime could only be attributed to the wakefield-dominated Stark mixing between the 2s²S₁/2 and 2p²⁴P₁/₂ states in the H-like V ion. In the non-relativistic (NR) limit, the two-photon decay rate [22] is \( \omega_{NR}(2E1) = 8.229.38Z²\mu g^{-1} \). The M1 decay rate in the Dirac theory [10] is \( \omega_{NR}(M1) = 2.496 \times 10^{-6}Z^³\mu g^{-1} \). The larger two-photon decay branch is used for lifetime measurement of the 2s²S₁/2 state of H-like ions for Z < 43. A precise lifetime measurement (error about 1%) for the 2s²S₁/2 state of H-like Ni [23] through the two-photon channel showed very good
agreement (within 1 standard deviation) with theoretical values [10, 22]. Thus, H-like 2s2S1/2 system is well understood and we can make use of theoretical H-like 2s2S1/2 lifetime for any application. For such a situation the perturbed 2s2S1/2 state and its decay rate become [14]

\[
\psi'_{2s} = \psi_{2s} + \epsilon \psi_{2p}, \\
\omega'_{2s} = \omega_{2s} + |\epsilon|^2 \omega_{2p},
\]

where \(\epsilon\) is the mixing parameter. By putting the measured \(\omega'_{2s}\) in this experiment and theoretical values of \(\omega_{2s}\) \((1.32 \times 10^9 \text{ s}^{-1} [10, 22])\) and \(\omega_{2p}\) \((1.8 \times 10^{14} \text{ s}^{-1} [13])\), \(|\epsilon|\) turns out to be 0.01046. Electric field \(F'\) responsible for such mixing can be calculated from \(|\epsilon| = \frac{F'}{475 Z^2} [9, 14]\). It is found to be \(3.2 \pm 0.07 \times 10^7 \text{ V cm}^{-1}\) which is comparable to the minimum Stark field \((1.78 \times 10^8 \text{ V cm}^{-1})\) required for complete mixing of the two states [9]. In the error estimation, uncertainty in the lifetime measurement (4.2\%) is only introduced in evaluating \(|\epsilon|\) as no uncertainty in theoretical transition rates can be considered. Uncertainty in \(|\epsilon|\) is equal to error in \(F'\) and it is only 2.1\%.

The stopping power, energy loss per unit foil thickness, is the magnitude of electromagnetic force. This electromagnetic force is electric in origin and its magnitude will differ from one ion to another because of their different charge states. The corresponding electric field can be obtained by dividing the stopping power by the charge state of the ion. Instead of determining the electric field from the stopping power one can measure the electric field, as we have done in the present experiment, to find the stopping power from the product of the electric field and the charge state of the ion. The stopping power corresponding to the wakefield, we call it wake stopping power, measured in the present experiment turns out to be only \(0.07 \pm 0.0015 \text{ MeV } \mu \text{g cm}^{-2}\), about 92 and 316 times smaller than the bulk stopping power in C-foil and Au-foil [7], respectively (see footnote 3). Hence, one can disentangle the bulk stopping power and the wake stopping power in an experiment like the present one.

Burgdörfer suggested [24] that while a fast positive ion traverse a thin foil with greater than the Fermi velocity, three competing mechanisms: the surface barrier, the dynamical image force and the surface plasmon, can be involved at the surface leading to the wakefield. Hence, the wakefield is the sum of the electric-field magnitudes due to the surface barrier, the dynamical image force and the surface plasmon, which is measured to be \(3.2 \pm 0.07 \times 10^7 \text{ V cm}^{-1}\) for 160 MeV 51V ions in C- or Au-foil.

We must mention here that the M1 peak was used for the H-like Ag 2s2S1/2 lifetime measurements [19] and the measured lifetime was in conformity with the theoretical value [25]. This implies that for the H-like Ag case the wake stopping power field was not enough to mix the two states having a Lamb shift of 4.341(2) eV [26]. Considering the theoretical prediction [27] that wakefield is directly proportional to the charge state of the impinging ions and is not dependent on the ion velocity, we get the wakefield that can be responsible for Stark mixing the 2s2S1/2 state with 2p2P1/2 is \(6.69 \times 10^7 \text{ V cm}^{-1}\). This value is about 2 orders of magnitude smaller than the minimum Stark field \((4.26 \times 10^9 \text{ V cm}^{-1})\) required for mixing the two states 2s2S1/2 and 2p2P1/2. In other words, it can cause little mixing between the two states (\(\epsilon = 6.14 \times 10^{-4}\)). In fact, this verification ascertains the reliability of the present measurement.
In conclusion, we have demonstrated that the wakefield-induced Stark mixing can lead to mixing the fine-structure sublevels $2s^2S_{1/2}$ and $2p^2P_{1/2}$ in H-like V ions. The measured Stark mixing parameter can be used to determine the wakefield as well as the wake stopping power of an ion through a solid target. The present experiment reveals that although the wake stopping power is about 2 orders of magnitude smaller than the bulk stopping power in the MeV energy range, still the two contributions can be resolved experimentally.

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