CHAPTER - IV

MEASUREMENT OF K X-RAY FLUORESCENCE CROSS-SECTIONS AND RELATIVE INTENSITIES

4.1 Kα and Kβ XRF cross-sections:

The Kα and Kβ X-ray fluorescence cross-sections have been measured for 10 elements namely Ca(20), Ti(22), Cu(29), Zn(30), Ge(32), Se(34), Y(39), Ag(47), Sn(50) and Br(56) in the excitation energy range of 5.96 to 59.54 keV. All these target elements were taken in the form of thin foils of thickness varying from 70 to 160 μg/cm² evaporated on 6.3 μm thick mylar foil. These target materials were obtained from Micromatter (U.S.A.). Secondary exciters viz., Cu, Se, Y, Mo, Sn, Sm and Dy excited by the ²⁴¹Am (300 mCi) source, provided the exciting photons of energy 8.14, 11.37, 15.20, 17.80, 25.80 and 41.00 and 46.90 keV respectively. In the lower energy range, the 5.96 keV photons of ⁵⁵Fe and in the higher energy range 22.6 and 59.54 keV photons of ¹⁰⁹Cd and ²⁴¹Am respectively were used for direct excitation of the target K X-rays.

The fluorescent X-ray spectra were recorded with the X-ray detection system described in Section 3.2. To have a good counting statistics three spectra were run for each target material for time intervals ranging from 5000 to 40000 seconds. A typical spectrum of Cu at an excitation
Fig. 4.1 K X-RAY SPECTRUM OF Cu FOIL (129 μg/cm²) WITH $^{109}$Cd EXCITER
energy of 22.6 keV is shown in Fig. (4.1). It can be seen from the Fig. (4.1) that the $K_{\alpha}$ and $K_{\beta}$ X-rays are well separated in this spectrum. Similar spectra were observed for other elements also. The areas under the $K_{\alpha}$ and $K_{\beta}$ X-rays peaks have evaluated as described in Sec. 3.6.2.

4.1.1 Experimental evaluation of K XRF cross-sections

For the evaluation of experimental K X-ray fluorescence cross-sections the relation (3.9) can be rewritten as

$$\sigma_{K_{\alpha}} = \frac{N_{K_{\alpha}}(i)}{I_{e}G \beta_{i}e_{i}n_{i}}$$

(4.1)

where $N_{K_{\alpha}}(i)$ are the number of counts observed for the $K_{\alpha}$ X-ray line of the element $i$, per unit time, $I_{e}$ is the intensity of the exciting radiation, $G$ is the geometry factor, $e_{i}$ is the detector efficiency for the $K_{\alpha}$ X-ray line of element $i$, $n_{i}$ is the product $j_{i}d$ and is the thickness of the target element $i$ in g cm$^{-2}$ and $\beta_{i}$ is the target self-absorption correction for both the incident and the emitted radiations (characteristic X-rays). The method of evaluation of the absorption correction ($\beta_{i}$) used in these calculations has already been explained in Section 3.6.4. It may be added that an expression similar to eqn. (4.1) has also been used for the evaluation of $K_{\beta}$ XRF cross-section values from experimental data.
In eqn.(4.1), the factor $I_G$ represents the intensity of exciting radiation falling on the sample. It was calculated by running K X-ray spectra of a thin film of one element of known thickness and taking the theoretical X-ray fluorescence cross-section for that element as calculated from eqn.(4.2). All the experimental XRF cross-sections, therefore, stand expressed relative to that element.

The values of the various parameters used in the evaluation of experimental K XRF cross-sections are listed in Table 4.1. The experimental values of the K XRF cross-section are listed in table 4.2 along with the corresponding theoretical values.

### 4.1.2 Theoretical evaluation of K XRF cross-section

The theoretical values of the $K_\alpha$ X-ray fluorescence cross-sections have been calculated using the relation (2.1). This relation can be rewritten as

$$ \sigma_{K_\alpha} = J_{K_\alpha}(E) \sigma_{K}(E) \frac{f_{K_\alpha}}{\eta_{K_\alpha}} $$

where $\sigma_{K_\alpha}$ is the $K_\alpha$ X-ray fluorescence cross-section, $\sigma_{K}(E)$ is the K-shell photoionisation cross-section for the exciting energy (E), $\eta_{K_\alpha}$ is the K-shell fluorescence yield and $f_{K_\alpha}$ is the fractional X-ray emission rate for the $K_\alpha$ X-rays.

$\sigma_{K}(E)$ was calculated using the relation

$$ \sigma_{K}(E) = \sigma(E)(1 - 1/\eta_{K}) $$

...(4.3)
Table 4.1: Value of different parameters used to evaluate experimental K X-ray fluorescence cross-sections.

**Excitation energy = 5.96 keV**

<table>
<thead>
<tr>
<th>Element</th>
<th>Kind of X-ray</th>
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<th>( \beta_1 )</th>
<th>( \varepsilon_1 )</th>
<th>Target thickness ( m_1 )</th>
<th>No. of counts/second ( N_{r_1} )</th>
</tr>
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<td>Ca(26)</td>
<td>( K_a )</td>
<td>( 1.69 \pm 0.14 \times 10^7 )</td>
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<td>1.24 \times 10^{-3}</td>
<td>68.3</td>
<td>66.32 \pm 0.06</td>
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<tr>
<td></td>
<td>( K_\beta )</td>
<td></td>
<td>0.989</td>
<td>1.30 \times 10^{-3}</td>
<td>68.3</td>
<td>9.8 \pm 0.02</td>
</tr>
<tr>
<td>Ti(22)</td>
<td>( K_a )</td>
<td></td>
<td>0.988</td>
<td>1.42 \times 10^{-3}</td>
<td>112</td>
<td>193.3 \pm 0.23</td>
</tr>
<tr>
<td></td>
<td>( K_\beta )</td>
<td></td>
<td>0.989</td>
<td>1.51 \times 10^{-3}</td>
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<td>25.3 \pm 0.09</td>
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**Excitation energy = 8.132 keV**

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<th>( \beta_1 )</th>
<th>( \varepsilon_1 )</th>
<th>Target thickness ( m_1 )</th>
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<td></td>
<td>( K_\beta )</td>
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<td>0.989</td>
<td>1.30 \times 10^{-3}</td>
<td>68.3</td>
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<td>( K_a )</td>
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<td>0.54 \pm 0.004</td>
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<td>0.989</td>
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<td>0.072 \pm 0.003</td>
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**Excitation energy = 11.37 keV**

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<th>( \beta_1 )</th>
<th>( \varepsilon_1 )</th>
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<td>( K_\beta )</td>
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<td>0.993</td>
<td>1.30 \times 10^{-3}</td>
<td>68.3</td>
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**Excitation Energy = 15.20 keV**

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<tr>
<td></td>
<td>$K_\beta$</td>
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<td>$1.51 \times 10^{-3}$</td>
</tr>
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<td>$2.32 \times 10^{-3}$</td>
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<tr>
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<td>Y(39)</td>
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**Excitation Energy = 22.6 keV**

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<tr>
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<td>1.30$\times$10$^{-3}$</td>
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<tr>
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<td>1.51$\times$10$^{-3}$</td>
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<td>2.32$\times$10$^{-3}$</td>
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<td>Y(39)</td>
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### Excitation Energy = 25.8 keV

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<th>$\sigma$</th>
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### Excitation Energy = 41.00 keV

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<td>Zn(30)</td>
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<th>Emission Line</th>
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<td>Kβ</td>
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<td>Se(34)</td>
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Excitation energy = 46.90 keV

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<td>Zn(30)</td>
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<th>Energy</th>
<th>Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ge(32)</td>
<td>Kα</td>
<td>0.998</td>
<td>2.28x10^{-3}</td>
<td>99</td>
<td>2.055±0.004</td>
</tr>
<tr>
<td></td>
<td>Kβ</td>
<td>0.998</td>
<td>2.32x10^{-3}</td>
<td>99</td>
<td>2.303±0.002</td>
</tr>
<tr>
<td>Se(34)</td>
<td>Kα</td>
<td>0.998</td>
<td>2.32x10^{-3}</td>
<td>104</td>
<td>2.806±0.005</td>
</tr>
<tr>
<td></td>
<td>Kβ</td>
<td>0.998</td>
<td>2.37x10^{-3}</td>
<td>104</td>
<td>0.458±0.003</td>
</tr>
<tr>
<td>Y(39)</td>
<td>Kα</td>
<td>0.999</td>
<td>2.39x10^{-3}</td>
<td>58</td>
<td>2.684±0.008</td>
</tr>
<tr>
<td></td>
<td>Kβ</td>
<td>0.999</td>
<td>2.39x10^{-3}</td>
<td>58</td>
<td>0.537±0.004</td>
</tr>
<tr>
<td>Ag(47)</td>
<td>Kα</td>
<td>0.998</td>
<td>2.31x10^{-3}</td>
<td>99</td>
<td>8.67±0.02</td>
</tr>
<tr>
<td></td>
<td>Kβ</td>
<td>0.999</td>
<td>2.17x10^{-3}</td>
<td>99</td>
<td>1.779±0.012</td>
</tr>
<tr>
<td>Sn(50)</td>
<td>Kα</td>
<td>0.999</td>
<td>2.16x10^{-3}</td>
<td>100</td>
<td>9.534±0.026</td>
</tr>
<tr>
<td></td>
<td>Kβ</td>
<td>0.999</td>
<td>1.91x10^{-3}</td>
<td>100</td>
<td>1.837±0.013</td>
</tr>
<tr>
<td>Be(56)</td>
<td>Kα</td>
<td>0.999</td>
<td>1.59x10^{-3}</td>
<td>76.8</td>
<td>6.984±0.016</td>
</tr>
<tr>
<td></td>
<td>Kβ</td>
<td>0.998</td>
<td>1.24x10^{-3}</td>
<td>76.8</td>
<td>1.294±0.012</td>
</tr>
</tbody>
</table>

Excitation energy = 52.54 keV

<table>
<thead>
<tr>
<th>Element</th>
<th>Symbol</th>
<th>Intensity</th>
<th>Energy</th>
<th>Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu(29)</td>
<td>Kα</td>
<td>(8.19±0.34)x10^6</td>
<td>0.997</td>
<td>2.13x10^{-3}</td>
</tr>
<tr>
<td></td>
<td>Kβ</td>
<td>0.997</td>
<td>2.21x10^{-3}</td>
<td>129</td>
</tr>
<tr>
<td>Zn(30)</td>
<td>Kα</td>
<td>0.997</td>
<td>2.19x10^{-3}</td>
<td>125</td>
</tr>
<tr>
<td></td>
<td>Kβ</td>
<td>0.998</td>
<td>2.26x10^{-3}</td>
<td>125</td>
</tr>
</tbody>
</table>

...contd....
...contd....

| Element | 
|---------|---------|--------|-------|
| Ge(32)  | $K_{\alpha}$ | 0.998  | 2.28x10^{-3} | 99 | 1.499±0.005 |
|         | $K_{\beta}$ | 0.995  | 2.32x10^{-3} | 99 | 0.216±0.003 |
| Se(34)  | $K_{\alpha}$ | 0.998  | 2.32x10^{-3} | 104 | 2.046±0.005 |
|         | $K_{\beta}$ | 0.999  | 2.37x10^{-3} | 104 | 0.3535±0.002 |
| Y(39)   | $K_{\alpha}$ | 0.999  | 2.39x10^{-3} | 58 | 1.880±0.007 |
|         | $K_{\beta}$ | 0.999  | 2.39x10^{-3} | 58 | 0.365±0.004 |
| Ag(47)  | $K_{\alpha}$ | 0.999  | 2.31x10^{-3} | 99 | 6.412±0.015 |
|         | $K_{\beta}$ | 0.999  | 2.17x10^{-3} | 99 | 1.353±0.007 |
| Sn(50)  | $K_{\alpha}$ | 0.999  | 2.16x10^{-3} | 100 | 7.185±0.016 |
|         | $K_{\beta}$ | 0.999  | 1.92x10^{-3} | 100 | 1.415±0.008 |
| Br(56)  | $K_{\alpha}$ | 0.999  | 1.59x10^{-3} | 76.8 | 5.487±0.014 |
|         | $K_{\beta}$ | 0.999  | 1.24x10^{-3} | 76.8 | 1.056±0.009 |
where \( \sigma(E) \) is the total photoelectric cross-section of the given element at exciting energy (E) and \( J_K \) is the K shell jump ratio.

In the present calculations the values of \( \sigma(E) \) are taken from Stern and Israel, that of \( J_K \) from Mc Master et al, and the value of the product of \( f_{\alpha} \) from the tables of Krause and et al.

The \( K_\beta \) XRF cross-sections (\( \sigma_{K_\beta} \)) are calculated using the equation

\[
\sigma_{K_\beta} = \sigma_{K_\alpha} \frac{I_{K_\beta}}{I_{K_\alpha}}
\]

where the most probable value of the intensity ratio \( I_{K_\beta}/I_{K_\alpha} \) is taken from Khan and Karimi.

4.1.3 Results and Discussion

The experimental values of \( K_\alpha \) and \( K_\beta \) XRF cross-sections for ten elements at different excitation energies are listed in Table 4.2. The statistical error in the counts for \( K_\alpha \) X-ray peak is estimated to be less than 2% and that in \( K_\beta \) X-ray peak it is 1-6%, depending on the excitation energy. The uncertainty in the detector efficiency is estimated to be of the order of 3%, the error in \( \tau_1 \) is of the order of 2% and error in the values of absorption correction (\( \beta \)) is fairly small because of the fact that thin foils were used.

The uncertainty in the area of the K X-ray peak was evaluated by taking the weighted average of the area from different runs.
Table 4.2: Measured and calculated $K_\alpha$ and $K_\beta$ X-ray fluorescence cross-sections for elements $20 \leq Z \leq 56$ at different excitation energies (results are expressed in cm$^2$/g).

<table>
<thead>
<tr>
<th>Excitation Energy in keV</th>
<th>Element</th>
<th>Measured</th>
<th>Others</th>
<th>Calculated $K_\alpha$ X-ray</th>
<th>Measured $K_\alpha$ X-ray</th>
<th>Calculated $K_\beta$ X-ray</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.96</td>
<td>Ca(20)</td>
<td>46.93±1.91</td>
<td>-</td>
<td>48.140</td>
<td>6.07±0.25</td>
<td>6.152</td>
</tr>
<tr>
<td></td>
<td>Ti(22)</td>
<td>(a)</td>
<td>-</td>
<td>72.823</td>
<td>8.96±0.37</td>
<td>9.540</td>
</tr>
<tr>
<td>8.14</td>
<td>Ca(20)</td>
<td>19.53±0.87</td>
<td>-</td>
<td>20.782</td>
<td>(b)</td>
<td>2.639</td>
</tr>
<tr>
<td></td>
<td>Ti(22)</td>
<td>(a)</td>
<td>-</td>
<td>31.684</td>
<td>3.69±0.21</td>
<td>4.151</td>
</tr>
<tr>
<td>11.37</td>
<td>Ca(20)</td>
<td>7.17±0.31</td>
<td>-</td>
<td>8.133</td>
<td>(b)</td>
<td>1.033</td>
</tr>
<tr>
<td></td>
<td>Ti(22)</td>
<td>12.21±0.51</td>
<td>-</td>
<td>12.596</td>
<td>1.49±0.08</td>
<td>1.650</td>
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<tr>
<td></td>
<td>Cu(29)</td>
<td>(a)</td>
<td>-</td>
<td>51.515</td>
<td>7.12±0.36</td>
<td>7.058</td>
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<tr>
<td></td>
<td>Zn(30)</td>
<td>6.54±2.5</td>
<td>-</td>
<td>60.662</td>
<td>8.22±0.34</td>
<td>8.370</td>
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<tr>
<td>15.20</td>
<td>Ca(20)</td>
<td>3.39±0.14</td>
<td>-</td>
<td>3.544</td>
<td>0.46±0.027</td>
<td>0.450</td>
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<tr>
<td></td>
<td>Ti(22)</td>
<td>5.47±0.23</td>
<td>-</td>
<td>5.562</td>
<td>0.66±0.04</td>
<td>0.729</td>
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<tr>
<td></td>
<td>Cu(29)</td>
<td>(a)</td>
<td>-</td>
<td>23.807</td>
<td>3.24±0.13</td>
<td>3.282</td>
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<td>Zn(30)</td>
<td>27.86±1.13</td>
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<td>27.792</td>
<td>3.91±0.16</td>
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### Table

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<tr>
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<th>Error</th>
<th>Value</th>
<th>Error</th>
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<tr>
<td>Ge(32)</td>
<td>34.3±1.4</td>
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<td>34.875</td>
<td>0.21</td>
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<tr>
<td>Se(34)</td>
<td>42.9±1.75</td>
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<td>42.434</td>
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<td>Cn(26)</td>
<td>2.27±0.09</td>
<td>2.55±0.17</td>
<td>2.332</td>
<td>0.17</td>
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<tr>
<td>Ti(22)</td>
<td>3.70±0.16</td>
<td>3.53±0.25</td>
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<tr>
<td>Cu(29)</td>
<td>15.6±4.7</td>
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<td>2.15±0.5</td>
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<tr>
<td>Zn(30)</td>
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<td>18.6±5</td>
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<tr>
<td>Ge(32)</td>
<td>22.9±1.9</td>
<td>22.3±1.09</td>
<td>22.737</td>
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<td>28.7±1.17</td>
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<td>28.326</td>
<td>4.5±0.18</td>
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<tr>
<td>Y(39)</td>
<td>45.8±2.07</td>
<td>31.6±1.54</td>
<td>46.6±3</td>
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</tr>
<tr>
<td>Ca(20)</td>
<td>1.63±0.04</td>
<td>1.1±0.08</td>
<td>1.096</td>
<td>0.14±0.01</td>
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<tr>
<td>Ti(22)</td>
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<tr>
<td>Cu(29)</td>
<td>8.37±0.34</td>
<td></td>
<td>7.865</td>
<td>1.15±0.05</td>
</tr>
<tr>
<td>Zn(30)</td>
<td>9.25±0.38</td>
<td>9.48±0.38</td>
<td>9.256</td>
<td>1.129(5)</td>
</tr>
<tr>
<td>Ge(32)</td>
<td>11.37(40)</td>
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<td>11.775</td>
<td>1.67(7)</td>
</tr>
<tr>
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<td></td>
<td>14.560</td>
<td>2.25(9)</td>
</tr>
<tr>
<td>Y(39)</td>
<td>(a)</td>
<td></td>
<td>24.767</td>
<td>4.63(19)</td>
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<th>Value 2</th>
<th>Error 2</th>
<th>Value 3</th>
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<tr>
<td>25.8</td>
<td>Cu(29)</td>
<td>5.69±0.23</td>
<td>-</td>
<td>5.456</td>
<td>0.79±0.03</td>
<td>0.747</td>
</tr>
<tr>
<td></td>
<td>Zn(30)</td>
<td>6.83±0.28</td>
<td>-</td>
<td>6.362</td>
<td>0.97±0.04</td>
<td>0.884</td>
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<tr>
<td></td>
<td>Ge(32)</td>
<td>8.55±0.35</td>
<td>-</td>
<td>8.561</td>
<td>1.26±0.05</td>
<td>1.301</td>
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<tr>
<td></td>
<td>Se(34)</td>
<td>10.89±0.44</td>
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<td>10.548</td>
<td>1.71±0.07</td>
<td>1.698</td>
</tr>
<tr>
<td></td>
<td>Y(39)</td>
<td>(a)</td>
<td>-</td>
<td>17.824</td>
<td>3.06±0.13</td>
<td>3.262</td>
</tr>
<tr>
<td>41.00</td>
<td>Cu(29)</td>
<td>1.450±0.06</td>
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<td>1.410</td>
<td>0.19±0.01</td>
<td>0.193</td>
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<tr>
<td></td>
<td>Zn(30)</td>
<td>1.75±0.07</td>
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<td>1.670</td>
<td>0.25±0.01</td>
<td>0.232</td>
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<td>Ge(32)</td>
<td>2.21±0.09</td>
<td>-</td>
<td>2.166</td>
<td>0.32±0.01</td>
<td>0.329</td>
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<tr>
<td></td>
<td>Se(34)</td>
<td>2.84±0.12</td>
<td>-</td>
<td>2.751</td>
<td>0.46±0.02</td>
<td>0.443</td>
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<tr>
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<td>Y(39)</td>
<td>(a)</td>
<td>-</td>
<td>4.817</td>
<td>0.89±0.04</td>
<td>0.881</td>
</tr>
<tr>
<td></td>
<td>Ag(47)</td>
<td>9.24±0.38</td>
<td>-</td>
<td>9.001</td>
<td>1.98±0.08</td>
<td>1.908</td>
</tr>
<tr>
<td></td>
<td>Sn(50)</td>
<td>10.19±0.42</td>
<td>-</td>
<td>10.461</td>
<td>2.21±0.09</td>
<td>2.322</td>
</tr>
<tr>
<td></td>
<td>Ba(56)</td>
<td>13.74±0.56</td>
<td>-</td>
<td>13.454</td>
<td>(b)</td>
<td></td>
</tr>
<tr>
<td>46.9</td>
<td>Cu(29)</td>
<td>0.99±0.04</td>
<td>-</td>
<td>0.946</td>
<td>0.14±0.006</td>
<td>0.130</td>
</tr>
<tr>
<td></td>
<td>Zn(30)</td>
<td>1.18±0.05</td>
<td>-</td>
<td>1.123</td>
<td>0.17±0.007</td>
<td>0.156</td>
</tr>
<tr>
<td></td>
<td>Ge(32)</td>
<td>1.49±0.06</td>
<td>-</td>
<td>1.464</td>
<td>0.22±0.008</td>
<td>0.222</td>
</tr>
<tr>
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<td>Se(34)</td>
<td>1.90±0.08</td>
<td>-</td>
<td>1.865</td>
<td>0.30±0.01</td>
<td>0.300</td>
</tr>
</tbody>
</table>
(a) The K X-rays of these elements have been utilised to evaluate the I\textsubscript{c}G values.

(b) The area under the K\textsubscript{p} X-ray peaks was so poor that no worthwhile estimates could be made for these energies.

(c) Results of Watson et al (43).

(d) Results of Bhan et al (14).

<table>
<thead>
<tr>
<th>Element</th>
<th>Value 1</th>
<th>Value 2</th>
<th>Value 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Y(39)</td>
<td>3.298</td>
<td>0.66±0.03</td>
<td>0.604</td>
</tr>
<tr>
<td>Ag(47)</td>
<td>6.47±0.26</td>
<td>6.224</td>
<td>1.41±0.06</td>
</tr>
<tr>
<td>Sn(50)</td>
<td>7.52±0.31</td>
<td>7.242</td>
<td>1.64±0.07</td>
</tr>
<tr>
<td>Ba(56)</td>
<td>9.74±0.40</td>
<td>9.417</td>
<td>2.32±0.10</td>
</tr>
<tr>
<td>Cu(29)</td>
<td>0.48±0.02</td>
<td>0.466</td>
<td>0.066±0.003</td>
</tr>
<tr>
<td>Zn(30)</td>
<td>0.58±0.02</td>
<td>0.554</td>
<td>0.084±0.004</td>
</tr>
<tr>
<td>Ge(32)</td>
<td>0.74±0.03</td>
<td>0.724</td>
<td>0.105±0.005</td>
</tr>
<tr>
<td>Se(34)</td>
<td>0.94±0.04</td>
<td>0.927</td>
<td>0.166±0.007</td>
</tr>
<tr>
<td>Y(39)</td>
<td>1.658</td>
<td>0.32±0.01</td>
<td>0.303</td>
</tr>
<tr>
<td>Ag(47)</td>
<td>3.43±0.14</td>
<td>3.264</td>
<td>0.77±0.03</td>
</tr>
<tr>
<td>Sn(50)</td>
<td>4.67±0.17</td>
<td>3.759</td>
<td>0.9±0.04</td>
</tr>
<tr>
<td>Ba(56)</td>
<td>5.49±0.23</td>
<td>4.969</td>
<td>1.36±0.06</td>
</tr>
</tbody>
</table>
Hence overall error in the $K_\alpha$ XRF cross-sections normally comes to be equal to or less than 5%, and that in the $K_\beta$ XRF cross-sections it is estimated to be of the order of 4-7% in all the present measurements.

The theoretical values of $K_\alpha$ and $K_\beta$ XRF cross-sections are given in Table 4.2. These values are compared with the corresponding experimental results. The theoretical results are shown by thick curves in Fig. (4.2 a,b), and the experimental values are shown by points over these. It is clear from these plots that the experimental results agree fairly well, within experimental error, with the theoretical results for all the excitation energies.

Relative $K_\alpha$ XRF cross-sections in barns/atom reported by Watson et al and Bhan et al have also been converted into absolute $K_\alpha$ XRF cross-section in $\text{cm}^2/\text{g}$ by taking the $K_\alpha$ XRF cross-sections for Cu equal to 15.604 and 7.865 $\text{cm}^2/\text{g}$ at 17.8 and 22.6 keV excitation energies respectively. These results are given in Table 4.2. It can be seen from the table that the results of Watson et al\textsuperscript{43} are in good agreement with the present measured results for the atomic-number-range covered in this study. It may be pointed out that around atomic number 39, the experimental values of Watson et al\textsuperscript{43} differ significantly from the theoretical values. However, our experimental value in this atomic number range agree well with theory. It is likely that this discrepancy in the results of Watson et al\textsuperscript{43} is caused by the additional
Fig. 4.2(a) COMPARISON OF MEASURED AND THEORETICALLY CALCULATED VALUES OF $K_{\alpha}$ XRF CROSS-SECTIONS FOR ELEMENTS WITH $20 \leq Z \leq 56$ AS A FUNCTION OF ENERGY
Fig. 4.2 (b) COMPARISON OF MEASURED AND THEORETICALLY CALCULATED VALUES OF K XRF CROSS-SECTIONS FOR ELEMENTS WITH $20 \leq Z \leq 56$ AS A FUNCTION OF ENERGY
excitation due to bremsstrahlung. As pointed out by Watson et al.\textsuperscript{(43)}, over most of the atomic number range investigated, the extra fluorescence due to bremsstrahlung is nearly compensated with that caused in Copper, the reference element. But as the target binding energy approaches the Mo absorption edge, the fraction of the bremsstrahlung intensity which is effective in producing $\alpha$-vacancies begins to decrease. At this point the extra fluorescence due to bremsstrahlung no longer compensates the one that is produced in Copper. Therefore, the measured XRF cross-sections begin to fall short of the calculated values. In radioisotope excitation, used in the present work, bremsstrahlung is almost absent leading to good agreement between theoretical and experimental results in the total range of the atomic number. The results of Bhan et al.\textsuperscript{(44)} are in good agreement with the calculated values of $K_\alpha$ XRF cross-sections as is found in our case, because Bhan et al.\textsuperscript{(44)} have also used radioisotope excitation.

These measurements serve two purposes. Firstly, they provide an indirect check on the reliability of the available values of the basic parameters used in the calculations of XRF cross-section for excitation energies ranging from 5.96 to 59.54 keV. Secondly, these results confirm that excitation by radiation either directly from the radioisotopes or indirectly from the secondary exciter system does not involve any error due to bremsstrahlung as observed by Watson et al.\textsuperscript{(43)}. 
Further, it is established that these experimental values of K XRF cross-sections can be used with confidence for trace element analysis while making measurements using fundamental parameter approach.

4.2 \( \frac{I_{K\beta}}{I_{K\alpha}} \) X-ray relative intensities

Khan and Karimi\(^{35}\) have compiled a set of most probable values of \( \frac{I_{K\beta}}{I_{K\alpha}} \) intensity ratios from the available data and from some of their own measurements. These results agree well with those obtained from Scofields\(^{26}\) theoretical calculations based on the relativistic Hartree Fock model. When compared with the Scofield's latest theoretical calculations based on relativistic Hartree Slater model\(^{24}\) the most probable values of Khan and Karimi differ from the theory. In the range of 20 ≤ Z ≤ 56 the difference are 7.5 - 19\% . To check this discrepancy and the validity of the revised theory, \( \frac{I_{K\beta}}{I_{K\alpha}} \) intensity ratios for 20 elements in the range of atomic number 20-56 have been measured. These elements are Cr, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ge, Se, Y, Zr, Nb, Mo, As, In, Sb, and Ba. Most of the targets whose \( \frac{I_{K\beta}}{I_{K\alpha}} \) intensity ratios are measured were taken in the form of thin foils of thickness varying from 70 to 160 μg/cm\(^2\), evaporated on 6.3 μm thick mylar foil. For the elements whose thin foils could not be obtained thin pellets of the salts of required elements were prepared, thickness of these
pellets varied from 50 mg/cm² to 70 mg/cm². To measure $I_{K_p} / I_{K_{\alpha}}$ intensity ratios for elements with $Z \leq 32$, $33 \leq Z \leq 42$ and $43 \leq Z \leq 56$, the secondary exciters of Mo, Sn and Dy were used respectively. Although $I_{K_p} / I_{K_{\alpha}}$ intensity ratio is independent of the excitation energy even than the X-rays from different secondary exciters have been used to excite the characteristic X-rays of the elements falling in different atomic number ranges. It was necessitated to have sufficiently high counting rate.

The $I_{K_p} / I_{K_{\alpha}}$ intensity ratios have been obtained from the peak areas using the relation

$$
I_{K_p} / I_{K_{\alpha}} = \frac{N_{K_p}}{N_{K_{\alpha}}} \frac{\beta_{K_p}}{\beta_{K_{\alpha}}} \frac{\epsilon_{K_p}}{\epsilon_{K_{\alpha}}} \ ... \ ...(4.5)
$$

where $N_{K_p}$ and $N_{K_{\alpha}}$ are the counts/sec under the peaks corresponding to $K_p$ and $K_{\alpha}$ X-rays respectively, $\beta_{K_p}$ and $\beta_{K_{\alpha}}$ are the target self absorption corrections, $\epsilon_{K_p}$ and $\epsilon_{K_{\alpha}}$ are the detection efficiencies of the detector for $K_p$ and $K_{\alpha}$ X-rays respectively. The error in the experimental values of K X-ray intensity ratios is due to errors in the various parameters (eqn.4.5) involved in the evaluation of the experimental results. The errors in different parameters have already been explained in Section 4.1.3. As we are taking the ratios of K X-ray intensities, the target thickness and incident beam intensity are automatically cancelled out. As a result of this the errors in these quantities
does not contribute to errors in the relative intensities. Thus overall error in the measured K X-ray relative intensities is reduced and is estimated to be 3 to 5%. The results of these measurements are summarised in Table 4.3. The theoretical values calculated both from the relativistic Hartree Fock Model\(^{26}\) and the relativistic Hartree Slater Model\(^{24}\) are also listed in this table. Most probable values of Khan and Karimi\(^{35} \) are also given for comparison. The present \( \frac{I_{K\alpha}}{I_{K\beta}} \) intensity ratios agree well with the theoretical results of Scofield based on relativistic Hartree Fock Model\(^{26} \) and not with that of Hartree Slater Model\(^{24} \). Therefore it can be concluded that while making calculations of K XRF cross-sections one should preferably use the values of emission rates based on Hartree Fock Theory\(^{26} \).
### Table 4.3: $I_{K\beta}/I_{K\alpha}$ intensity ratios

<table>
<thead>
<tr>
<th>Element</th>
<th>Present</th>
<th>Most Probable value of Khan and Karimi</th>
<th>Theoretical calculations</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hartree Fock Model</td>
</tr>
<tr>
<td>Ca(20)</td>
<td>0.131±0.005</td>
<td>0.127</td>
<td>0.1315</td>
</tr>
<tr>
<td>Ti(22)</td>
<td>0.123±0.005</td>
<td>0.131</td>
<td>0.1355</td>
</tr>
<tr>
<td>V(23)</td>
<td>0.130±0.004</td>
<td>0.132</td>
<td>0.1367</td>
</tr>
<tr>
<td>Cr(24)</td>
<td>0.132±0.005</td>
<td>0.133</td>
<td>0.1377</td>
</tr>
<tr>
<td>Mn(25)</td>
<td>0.134±0.005</td>
<td>0.134</td>
<td>0.1385</td>
</tr>
<tr>
<td>Fe(26)</td>
<td>0.133±0.004</td>
<td>0.134</td>
<td>0.1391</td>
</tr>
<tr>
<td>Co(27)</td>
<td>0.137±0.004</td>
<td>0.136</td>
<td>-</td>
</tr>
<tr>
<td>Ni(28)</td>
<td>0.134±0.004</td>
<td>0.136</td>
<td>0.1401</td>
</tr>
<tr>
<td>Cu(29)</td>
<td>0.138±0.003</td>
<td>0.137</td>
<td>0.1379</td>
</tr>
<tr>
<td>Zn(30)</td>
<td>0.144±0.003</td>
<td>0.139</td>
<td>0.1410</td>
</tr>
<tr>
<td>Ge(32)</td>
<td>0.152±0.002</td>
<td>0.152</td>
<td>0.1504</td>
</tr>
<tr>
<td>Se(34)</td>
<td>0.161±0.002</td>
<td>0.161</td>
<td>0.1624</td>
</tr>
<tr>
<td>Y(39)</td>
<td>0.183±0.002</td>
<td>0.183</td>
<td>-</td>
</tr>
<tr>
<td>Zr(40)</td>
<td>0.186±0.004</td>
<td>0.187</td>
<td>0.1913</td>
</tr>
<tr>
<td>Nb(41)</td>
<td>0.192±0.004</td>
<td>0.191</td>
<td>-</td>
</tr>
<tr>
<td>Mo(42)</td>
<td>0.197±0.004</td>
<td>0.195</td>
<td>0.1981</td>
</tr>
<tr>
<td>Ag(47)</td>
<td>0.208±0.004</td>
<td>0.212</td>
<td>0.2130</td>
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<tr>
<td>In(49)</td>
<td>0.214±0.006</td>
<td>0.213</td>
<td>-</td>
</tr>
<tr>
<td>Sn(50)</td>
<td>0.220±0.005</td>
<td>0.222</td>
<td>0.2230</td>
</tr>
<tr>
<td>Fe(56)</td>
<td>0.243±0.006</td>
<td>0.237</td>
<td>0.2433</td>
</tr>
</tbody>
</table>