Spectral distribution from end window X-ray tubes

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ABSTRACT

Today little information still exists on spectra profiles emitted by x-ray tubes, although the development of mathematical matrix correction procedures for x-ray fluorescence analysis showed the demand of the knowlegde of accurate spectral intensity distribution from x-ray tubes. In this paper we present measured spectral intensity distributions from end window x-ray tubes (Au, Rh, Cu and Cr anodes) typically used in wavelength and energy dispersive spectrometers. The spectral distribution of the x-ray tubes measured with an energy dispersive method allows to compare the experimental data of the continuous spectra of the x-ray tube to a semiempirical expression based on the Kramers’ law. Furthermore, the knowlegde of different x-ray lines contributes to the total x-ray spectral distribution.

INTRODUCTION

In the quantitative analysis of elements by x-ray fluorescence techniques, it is essential to know the contribution of the continuous spectra to the excitation of the fluorescence lines relative to that excited by characteristic lines of the excitation beam. Modern x-ray generators are still built around the Coolidge tube, i.e. vacuum tube in which electrons are emitted by an heated W filament (cathode), accelerated by high voltage and focused on a water-cooled metal target (anode). Electron bombardement of solid targets is the most usual method to generate x-rays represented by spectral distributions of x-ray photons. These spectral distribution consist of a continuous spectrum (due to the Bremsstrahlung) on which are superposed a few characteristic lines depending on the target element. Spectral intensity distributions from x-ray tubes show that the continuous component is far from being negligible; the continuous spectrum represents 30 to 70% of the total intensity which is composed of this continuous spectrum and the anode’s characteristic lines.

It is surprising that it was long considered that the continuous spectrum was described by the Kramers’ law, while this law concerns the x-ray inner generation by electrons and not the x-rays leaving the anode or tube’s window. Before 1970, no discontinuities were reported due to the continuous spectrum’s self-absorption in the anode. Since 1968, the accurate spectral intensity distribution from x-ray tubes became more and more important for developing the fundamental parameter method. In these time new and more accurate measurements were realized. Important deviations between the experimental spectral distributions and the one given by Kramer’s law were reported.

These works were not carried on consequently, and therefore there exist only a few publications of which the most are principally devoted to former types of X-ray tubes. Up to now, no systematic study was carried out, although we know that the spectral distribution shape depends on the X-ray tube geometry.

Figure 1 shows measured spectral distributions for different types of X-ray tubes. The shape of the spectral distributions can be explained by a theory based on electron transmission. However, this theory is too elaborate to apply. Therefore, experimental measurements and semi-empirical relations are preferred nowadays.
EXPERIMENTAL METHODS

Different methods have been used to measure spectral distributions. The first of these methods has been used by many authors assuming that the spectrum, obtained by X-ray scattering by an organic sample agreed with real tube’s spectrum. Is difficult to fit such a spectrum to the real distribution. The exploitation of such a spectrum needs a lot of corrections.
Another method to study spectral distributions consists in exploiting spectra measured by an electronic microprobe, but again, results do not exactly match with reality. Furthermore, there exists two other experimental methods which allow to determine correctly the spectral distribution. The radiation at the exit of the tube is measured directly with a spectrometer, either wavelength or energy dispersive. Figure 2a,b represent the scheme of these experimental assemblies. These two methods also need corrections to link the measured x-ray intensity with x-ray intensity effectively emitted by the tube:

Fig. 2a,b Schemes of the experimental assemblies

**Wavelength dispersion**
- analysing crystal reflectivity
- detectors (scintillation and flow counters) efficiency
- pulse height analysis
- suppression of higher order reflections
- mylar window between detector transmission
- loss of counts due to the dead time of the detector
- total spectrum reconstruction from measurements with different analysing crystals

**Energy dispersion**
- detector quantum yield
- detector background
- escape peaks
- Compton effect in the detector
- pile-up peaks
- interference of fluorescence lines due to the spectrometer’s resolution
EXPERIMENTAL RESULTS

Measurements have been realized using the energy dispersion method, which is more easy to handle. After corrections, we record results in a table where intensity measured is noted for different excitation voltages. A full spectrum can be expressed as a distribution $N(E)dE$, where $N(E)$ is the number of accumulated counts corresponding to x-ray photon energies between $E$ and $E+dE$. Since wavelength is inversely proportional to energy ($\lambda=\frac{12.3981}{E}$), $d\lambda=-\frac{12.3981}{E^2}dE$ $N(E)$ can be written as $N(E)dE=N(\lambda)d\lambda$ where $N(\lambda)=N(E)E^2/12.3981$. Measurements have been made on front end window tubes (Beryllium windows thickness of 125µm) used for Siemens SRS303 spectrometers. Au, Rh, Cr and Cu anodes were studied. Figures 3,4,5,6 represent spectral distributions of the continuous spectra for different excitation voltages.

![Graph showing spectral distribution of the continuous spectra of an Au tube for different excitation voltages](image)

Fig 3. Spectral distribution of the continuous spectra of an Au tube for different excitation voltages
Fig 4. Spectral distribution of the continuous spectra of a Cu tube for different excitation voltages.

Fig 5. Spectral distribution of the continuous spectra of a Rh tube for different excitation voltages.
Fig 6. Spectral distribution of the continuous spectra of a Cr tube for different excitation voltages

We note for the Au tube, discontinuities of absorption for M shells can not be seen, although characteristic lines are detected. This is due to the difficulty to obtain an accurate measurement in the discontinuity area because, first, the superposition of intense characteristic lines and second the poor detector resolution at low energies. In practice, this imprecision is negligible, since characteristic lines are widely prevalent.

Fig 7 accounts for continuous spectra importance according to the tube’s target for the same excitation voltage (here e.g. 40 keV).
We note that these experimental results can be used in order to fit the data to the semi-empirical equation:

\[ N(\lambda) \propto \left( \frac{\lambda}{\lambda_0} - 1 \right)^q \left( \frac{1}{\lambda^2} \exp(-Q'\mu'_{A,\lambda}) \exp(-\mu_{Be,\lambda} \rho_{Be} d) \right) \]

The first term agrees with the Kramers’ law, modified by the exponent q. The second term represents the radiation self absorption in the target, where the coefficients Q’ and n account for the mean depth electrons penetration in the target. Finally, the third term expresses the x-ray absorption by the beryllium window. Q’, n and q are fitted by a reiterative process until variations between theoretical and experimental graphs become a minimum. For example, for an excitation voltage of 50 keV, we obtain following coefficients ( n is kept constant to 1):
CHARACTERISTIC LINES
We have measured K\(\alpha\), K\(\beta\) lines of Cr and Cu tubes, L lines of Rh tube and L\(\alpha\), L\(\beta\), L\(\gamma\), L\(\delta\) and M lines of Au tube. Figure 8 represents measured intensity evolution for each characteristic line as a function of the exciting voltage.

<table>
<thead>
<tr>
<th>Tube</th>
<th>q</th>
<th>Q'</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr</td>
<td>1.502</td>
<td>0.00127</td>
</tr>
<tr>
<td>Cu</td>
<td>1.491</td>
<td>0.00144</td>
</tr>
<tr>
<td>Rh</td>
<td>1.426</td>
<td>0.00096</td>
</tr>
<tr>
<td>Au</td>
<td>1.206</td>
<td>0.00041</td>
</tr>
</tbody>
</table>

Fig. 8 Measured intensity evolution for each characteristic line as a function of the exciting voltage

We have represented in table 1 these characteristic lines leaving the x-ray tubes, at different voltages, that is, after all corrections required by the energy-dispersive method. Results are expressed in per cent in respect to the total spectrum (Bremsstrahlung plus characteristic lines)
<table>
<thead>
<tr>
<th>[kV]</th>
<th>20</th>
<th>25</th>
<th>30</th>
<th>35</th>
<th>40</th>
<th>45</th>
<th>50</th>
</tr>
</thead>
</table>

**Cr tube**

Brems. 34.09 31.10 29.60 28.02 25.73 26.53 26.84
K_α 57.92 60.54 61.64 63.18 65.15 64.45 64.23
K_β 7.99 8.36 8.76 8.80 9.12 9.02 8.93

**Cu tube**

Brems. 58.41 51.64 47.27 44.97 43.64 41.45 39.08
K_α 36.52 42.50 46.38 48.49 49.59 51.44 53.42
K_β 5.07 5.86 6.35 6.54 6.77 7.11 7.50

**Rh tube**

Brems. 55.82 54.73 55.07 55.07 53.57 54.82 55.21
L 44.18 54.27 44.28 43.47 43.88 41.41 39.74
K_α 0.55 1.25 2.16 3.21 4.21
K_β 0.10 0.21 0.40 0.57 0.84

**Cr tube**

Brems. 71.79 69.19 67.01 65.36 63.97 63.76 63.39
M_{a,b} 21.80 19.45 17.54 15.78 14.47 12.83 11.97
Mn4 0.51 0.38 0.34 0.23 0.26 0.21 0.21
L_{α} 0.16 0.37 0.42 0.52 0.60 0.64 0.64
L_α 3.71 6.45 8.70 10.46 11.90 12.72 13.37
Lβ 1.87 3.80 5.26 6.72 7.68 8.60 9.22
Lγ 0.15 0.37 0.72 0.93 1.12 1.24 1.19

**Table 1** Characteristic line intensities leaving the X-ray tubes at different voltages

Figure 9 illustrates these variations in per cent as a function of the exciting voltage for each X-ray tube.
CONCLUSION

Our collected spectral data are used in different software packages with fundamental parameter coefficients method. It is interesting to know the importance of different x-ray lines in order to determine their contributions to element analysis. In practice, the accuracy of the results is widely satisfied. In order to have more accurate measurements, it will be necessary to measure the discontinuities area (at low energies) by energy and wavelength dispersive method, and then to adjust the two measured spectra. Furthermore, it will be possible to realize a statistical study for determine the reliability of this method (exactness). We finally note that the spectral distribution does not remain constant during the x-ray tube lifetime. Characteristics can change by ageing and impurities.

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