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STATUS OF DATA AND DATA NEEDS FOR XRF AND PIXE BASED ELEMENT ANALYSIS

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BHABHA ATOMIC RESEARCH CENTRE
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STATUS OF DATA AND DATA NEEDS FOR XRF AND PIXE BASED ELEMENT ANALYSIS

S.S.Kapoor and R.K. Choudhury
Bhabha Atomic Research Centre
Bombay

Abstract:

The status of data and data needs for X-ray fluorescence (XRF) and particle induced X-ray analysis (PIXE) are examined to determine the areas where additional and improved data are required to improve the accuracy, precision and sensitivity of quantitative element analysis by the above techniques.

1. Introduction

The techniques of particle induced X-ray emission (PIXE) and photon induced X-ray fluorescence (XRF) analysis involving energy dispersive method of X-ray analysis using high resolution Si(Li) detectors are finding increasing use in the elemental analysis of materials. A number of review articles[1-5] have been published over the years, which cover the details of these techniques and describe a number of practical applications to illustrate the usefulness of these physical techniques.

The photon induced X-ray fluorescence analysis is carried out by exciting the characteristic X-rays of the

sample by bombarding it with low energy photons ($6\ keV \lesssim E_\gamma \lesssim 60\ keV$) usually from a radioactive source or an X-ray tube. In the case of PIXE technique, the characteristic X-rays of the elements in the sample are commonly produced by bombarding it with low energy protons ($E_p \sim 2$-4 MeV) from an accelerator. The energy spectrum of the characteristic X-rays produced in the sample by the XRF or PIXE technique is then measured by a high resolution Si(Li) detector X-ray spectrometer; the energy resolution of these spectrometers is usually sufficient to resolve K X-rays of neighbouring elements of $Z \gtrsim 11$, and to resolve L X-rays of elements above $Z \gtrsim 50$. The main advantage of the XRF technique lies in its simplicity; it can be started in a laboratory with a minimum demand on infrastructural support. On the other hand PIXE technique is accelerator based. The choice of technique and also the details of analysis of the data depend on the ultimate aim of a particular analysis. Very often one is interested in a rapid qualitative or semi-quantitative scan of the elements present in the sample. This can be most conveniently done by the XRF technique in a matter of minutes by

non-destructive analysis of the sample in its original form. For quantitative trace element analysis, the samples are prepared in suitable form and both methods can be applied to carry out the analysis.

In both the methods, the energy of each peak in the observed X-ray spectrum is associated with the presence of a particular element and the intensity of the peak is related to the elemental concentration. For quantitative analysis of samples, the observed intensity of the various peaks have to be transformed to the concentration of the corresponding elements. Intrinsically, this transformation involves many physical quantities such as the intensity of the exciting radiation, ionization cross section, fluorescence yield, relative intensities of X-ray transitions, X-ray attenuation coefficients, detector efficiency, solid angle of detection etc; consequently an accurate knowledge of these quantities is required if an absolute quantitative analysis is carried out without use of standards. This is rarely done in practical applications and methods involving the use of standards have been developed for accurate quantitative analysis to minimize errors in the results due to uncertainties

in the fundamental X-ray related data or in the quantities dependent on a particular experimental arrangement. The quantitative analysis usually involves sample preparation and both thin and thick sample techniques are being employed. Any lack of uniformity and reproducibility of the samples in the thin-sample technique and matrix effects in the thick-sample technique can introduce extra sources of errors in the quantitative analysis.

The status of existing fundamental X-ray related data from the point of view of requirements of such data in the quantitative XRF or PIXE analysis is to be examined with reference to the methods commonly employed in such analysis. The present paper is aimed primarily to provide background information for such a review of the X-ray data although the status of the data without reference to XRF or PIXE analysis is also important for a basic understanding of the physical mechanisms involved.

2. Particle Induced X-ray Emission (PIXE) method

In this method, the mass per unit area of a particular element Z in the sample is given by

$$x = \frac{k N}{\sigma_p \Omega T \eta Q} \qquad (1)$$

x = mass per unit area of element Z (ng/cm^2)

N = number of counts in a peak

σ_p = X-ray production cross-section corresponding to the peak (barn)

Ω = solid angle of X-ray detector (Sr)

T = X-ray transmission factor from the point of production to the point of detection

η = X-ray detection efficiency in the detector

Q = proton current x time = integrated charge (μC)

k = a known constant (.00334 x atomic weight of the element)

The X-ray production cross-section σ_p corresponding to the peak is given by

$$\sigma_p = \sigma_{ion}\, \omega\, f$$

where σ_{ion} = K or L shell ionization cross-section

w = fluorescence yield

f = fraction of the possible transitions giving rise to the observed peak

Thus, PIXE method offers the possibility of absolute quantitative analysis without the use of standards using Eq. (1). However, in order to remove uncertainties due to unknown factors of the experimental

arrangement like Ω, T and η , one establishes a reliable calibration of the analytical system with the use of standards. The standards which produce K X-ray peaks of medium energy (4.5 keV - 17.5 keV) are used to determine solid angle Ω and detection efficiency η , while the X-ray transmission factor T is best estimated with the lighter standard elements (X-ray energy range of 1.5 keV - 4.5 keV). One source of error in the calibration is the uncertainty in the stated thickness of the standard foils, which is often guaranteed by the manufacturers to only about 5%. It is clear that σ_p enters directly in the determination of concentration of an unknown element; the uncertainties in the knowledge of σ_p would therefore directly reflect on the accuracy of the quantitative analysis which can be achieved. It is,however, to be remembered that once the calibration is performed with a broad range of element standards, any overall systematic errors in σ_p will cancel out in the calibration procedure. Since usually the calibration is performed only with a few element standards, any systematic errors in the knowledge of σ_p in the region of the elements will affect the accuracy of analysis.

As the PIXE technique is used for trace element analysis, one is quite often interested not only in the accuracy of the analysis, but also in its sensitivity as determined by the minimum detectable concentration of an element. For an element to be detected, the number of counts in the peak, N_p, must satisfy the relation $N_p \gtrsim 3\sqrt{N_B}$, where N_B is the number of counts in the background continuum in an interval of width equal to the full width at half maximum (FWHM) of the peak. The high energy Compton background due to γ -rays from excited target nuclei and the bremsstrahlung radiation of higher energy electrons are two main causes of the background in the PIXE spectrum. The background therefore depends on the composition and thickness of the sample matrix and energy of the proton beam etc. The data related to background continuum such as its angular distribution and its dependence on the beam energy are not comprehensive. Such fundamental data are of use in optimizing energy and angle of detection to achieve best possible minimum detection limits, needed in many applications involving trace element analysis such as in the investigation of biological and environmental samples.

When the multielement standards and the samples under investigation are not infinitely thin, correction for matrix effects becomes necessary. These matrix effects are due to (i) slowing down of the charged particles in the matrix with subsequent decrease of cross sections, (ii) absorption of characteristic X-rays in the matrix, (iii) enhancement of trace element X-rays due to inner shell vacancy production by secondary electrons (iv) enhancement of trace element X-rays due to X-rays of heavier elements in the matrix. In analysing thick samples which are originally fluid such as natural water and blood, the concentration calibration can be carried out by the so called standard addition technique (Vis et al[6]), in which the matrix effects are cancelled. However, while analysing solid thick samples, these matrix effect corrections are usually done by computations, where relevant fundamental data are required as inputs. The status of the relevant data for PIXE analysis of thin and thick samples are discussed in more detail in section 4.

3. Photon excited X-ray fluorescence (XRF) analysis method

In this method, the expression for the number of X-rays produced in a thin sample per unit time for a

particular X-ray line may be written as

$$I_x \propto I_o \tau \left(1 - \frac{1}{J_{K,L}}\right) f \omega T \eta \Omega m_x \qquad (2)$$

where I_o = Intensity of exciting source

m_x = mass of the element in the sample (gm/cm^2)

$J_{K,L}$ = Jump ratio for K or L absorption edge

τ = total photoelectric cross section (cm^2/gm)

f = fraction of vacancies in the subshell giving the observed X-ray line

ω = fluorescence yield for the particular X-ray line of the element

T = transmission coefficient of X-ray

Ω = solid angle of X-ray detector

η = X-ray detection efficiency

Using the above expression, it is in principle possible to carry out quantitative analysis of samples without use of standards. However, the geometrical factors are better determined by use of standards. As in the PIXE method, the data required for the quantitative estimate of samples are the basic quantities such as the fluorescence yield and the photoelectric cross section etc., which should be known as accurately as possible. Apart from these, the effects such as thickness non-uniformity of samples, matrix corrections play a role for the thick samples.

Unlike the case of PIXE method, the origin of background in the XRF method is due to scattering of the incident photons from the sample and the surrounding material. The shape and intensity of the background is therefore a strong function of the source-sample-detector geometry and the mounting arrangement of the sample. In order to achieve the optimum detection limits, it is therefore necessary to select the energy of the exciting X-rays and proper geometry for excitation of sample X-rays.

4. Status of data and data needs

From the above discussion, it is clear that for quantitative analysis of samples by means of both XRF and PIXE, a knowledge of the data connected with many physical quantities such as ionization and photoelectric cross-sections, X-ray fluorescence yields, relative X-ray line intensities, X-ray attenuation coefficients, charged particle energy loss, bremsstrahlung process etc. are needed. Apart from these data which are directly connected with the quantitative analysis of samples, there are other experimental data which are of interest for comparison with the existing theoretical models for

ionisation cross sections of inner shells by particle bombardment. In the following, the areas where further improvements in data are required will be briefly reviewed to evaluate the work needed to improve the precision, accuracy of the XRF and PIXE analysis.

4.1 Inner Shell Ionisation Cross sections

Extensive measurements of K, L shell ionisation cross sections due to bombardment of verious charged particles at different bombarding energies have been carried out in the past. These measurements provide data not only for PIXE analysis but also for testing of theoretical formalisms used for calculation of vacancy production cross sections. The status on these measurements have been reviewed in the past for both K-shell[7-9] and L-shells[10,11]. The experimental ionization cross section data for K-shell have a scatter of about 10% to 12% in most cases. Recently, a lot of attention has been given to measure the K X-ray production cross sections to better accuracies ($\sim$5%) by measuring accurately the ratio of X-ray to scattering cross sections[12-16].

Very often L-lines are used for calculating the element concentrations of heavier elements ($Z \gtrsim 50$). This requires knowledge of X-ray production cross sections for L-lines; this involves the L-shell ionization cross sections, fluorescence yields and relative intensities of X-ray lines from different subshells. Measurements for L-shell ionization cross-sections have not been done extensively, and the intensity ratio of different L-lines are not available for many elements. The recent compilation by Sokhi and Crumpton[11] brings out clearly the cases where the data is lacking. L-subshell X-ray production cross-sections have been measured in recent years[17-27] but the deviations in the data are very large (20% to 30%). Also for many cases data do not exist for cross sections of individual L-subshell transitions. For very heavy elements ($Z \gtrsim 82$), the M - X-ray production cross section data can be of use in PIXE analysis. The measurements in this region have discrepancies of the order of 25% to 50%. There is no compilation of the available data on M X-ray production cross sections. However, recently some measurements have been reported in literature[28-30]. It is, therefore, evident that more efforts are required in order to obtain complete

data on K, L and M X-ray production cross sections to accuracies of a few percent. Various theoretical methods have been proposed to calculate the ionisation cross section of inner shells[31-35]. The agreement of these calculations with the measured values is far from satisfactory and in most cases the theories overestimate the cross sections and some times the discrepancy is upto a factor of two or more.

4.2 Fluorescence yields

A number of compilations[36,39] exist on the values of fluorescence, Auger and Coster - Kronig yields for all the elements for K and L shells. The measurements have been carried out with photon excitations and these values are known to about a few percent. However, the same values need not be valid for other types of excitations. Recently it has been inferred[40,41] that the fluorescence yields, and the L_α and L_ℓ intensity ratio may depend on the method of excitation upto even ten percent. Due to multiple ionisations in particle induced X-ray emission studies, ω_K and ω_L values may also depend on the particle type and energy. Very little data exists on this aspect and more measurements are

necessary for a clear understanding of the fluorescence process. Investigations on the effect of multiple vacancies have been carried out[42] but no precise guideline is available to calculate this effect. However, some results are available for light atoms[41,43] on the effects of multiple vacancies on the fluorescence yields.

4.3 Relative intensity ratios of K X-ray lines (K_α / K_β) and L X-ray lines (L_l / L_α, L_β / L_α, L_γ / L_α) as function of bombarding energy and particle type

In PIXE analysis it is often found that the X-ray lines interefere with each other. This is particularly so for elements for which L X-rays are measured. The K_α /K_β ratio for an element is fairly independent of the energy and the nature of the ionizing particle and has been tabulated in literature[44]. On the other hand it has been found that L_β /L_α and L_γ /L_α ratios are dependent on both the energy as well as the character of the ionising radiation. The amount of data on the L intensity ratios is quite limited[45-50].

Systematic analysis of the data for L X-ray line intensity ratios and comparison with theory is required for a proper understanding of the mechanism of X-ray emission. The variation in L_β / L_α or L_γ / L_α over the proton bombarding energy of 1.0 to 3.0 MeV is of the order of 20% to 40% and so a knowledge of this is quite essential for accurate analysis of PIXE data. Also due to multiple ionisations, the K X-ray and L X-ray line intensity ratios can alter significantly. Multiple ionisations have been shown to be quite important by many calculations[51].

In the XRF analysis, the relative yields of K_α and K_β lines due to excitation by different photon energies have been measured in the past[52-54] and compared with the theoretical calculations given by Krause et al[55]. Measurements of L X-ray line intensity ratios and cross sections due to excitation by different energy photons have also been reported[56-61]. Experimental data are available with 5% to 10% accuracy. Although for some elements the observed line intensity ratios agree well with the theoretical values, for some others the deviations are beyond the experimental errors. This area needs further investigation.

4.4. Impact parameter dependence of K and L shell ionisation cross sections.

From theoretical understanding of the X-ray emission mechanism in particle induced reactions, it is quite useful to measure the ionization cross sections as a function of impact parameter of the collision. Since these cross sections are very small as compared to the total atomic cross sections, these measurements are quite difficult. Most of the earlier measurements have been done by using NaI crystals for X-ray detection[62-63]. Theoretical estimates of these quantities have been made based on binary encounter approximation[64] model and detailed tabulations are available in literature[65]. The measurements are quite few and have been carried out mostly for K-shell ionisation cross sections. The data for L shell ionisation and X-ray line intensity ratios as a function of particle type and bombarding energy will be quite useful in order to compare with the theoretical calculations.

4.5 Angular distribution of continuum and fluorescent X-ray spectra in proton bombardment.

The data for the angular dependence of fluorescent and continuum X-rays are quite sparse.

One would expect different types of anguler distributions for continuum and fluorescent X-rays. There have been some measurements of the continuum X-ray angular distributions[66-69] and it is shown that the optimum detector angle for PIXE analysis, in order to obtain lowest detection limits depends on the atomic number of the element being analysed. however to systematize the observations, more measurements in the angular distribution of the continuum background and fluorescent X-rays need to be carried out at different proton bombarding energies and for different sample backing materials.

4.6 Si(Li) detector response function to X-rays

For unfolding of the X-ray spectra, one normally uses a Gaussian peak shape for the X-ray line and a polynomial for the smooth background. However, recent studies indicate that depending on the energy of X-ray and the detector thickness, the response function can be quite complicated. In the case of Ge detectors hypermet functions have been used[70-72] for analysis of γ-ray spectra. Recently Campbell et al[73] have studied the shapes of monoenergetic X-rays in Si (Li) detectors. The response of the detector

can be represented as sum of two Gaussians (one for photopeak and the other for escape peak), and exponential and step functions for the tailing of the X-rays lines. Many interesting observations are made regarding the contributions of the various components as a function of X-ray energy. The ratio of intensities of degraded events to the photopeak Gaussian events, however, decreases quite sharply as a function of X-ray energy. Since the final accuracy of the PIXE and XRF analysis depends on the accuracy in unfolding of the X-ray spectra, a knowledge of the response function for detectors of different thickness to X-rays of various energy is essential.

4.7 Chemical effect on the X-ray cross sections and intensity ratios of K and L lines

Recently, there have been reports on the influence of the chemical state of the atoms on the X-ray production cross sections as well as the intensity ratios of the X-ray lines. The effect of the chemical state on the position of K-edge have been clearly demonstrated[74]. Chemical shifts on the energy of the K X-ray lines of Fe, Ni and other elements in that atomic number range have been studied. However, very little data

exists on the effect of the chemical state on the K_α/K_β and L X-ray line intensity ratios and X-ray production cross sections. Differences of the order of 5% in K_α/K_β ratio of transition elements, Cr, Mn, Fe and Cu have been observed due to chemical effects as reported by Brunner et al[76]. Recently Richter and Watjen[78] have also pointed out that the limiting factors on the accuracy of PIXE analysis may be the chemical effect on the K_α/K_β and L intensity line ratios. However, systematic data on the chemical effects are not available and such data will be quite useful both for PIXE and XRF analysis as well as for theoretical understanding of these effects.

4.8 Matrix effects in thick samples:

As mentioned earlier, in the case of thick samples various corrections need to be applied in the analysis which include; slowing down of the protons in the sample, absorption of characteristic X-rays in the target, electron induced X-ray emission anx X-ray induced X-ray emission etc. Calculations have been done[77,78] to estimate the effects of each of these corrections as a function of the atomic number of the elements. It is seen that with

the available computer programs incorporating the various matrix effects, it is possible to obtain absolute analysis of elements in thick samples to an accuracy of 10 to 15 per cent.

5. Conclusions

In the above, the available experimental data on various quantities relevant for the PIXE and XRF analysis have been summarized. The areas where data are lacking and measurements are needed to improve the accuracy in the analysis and also to obtain basic understanding of the processes have been outlined.

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