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X-ray attenuation around *K*-edge of Zr, Nb, Mo and Pd: A comparative study using proton-induced X-ray emission and ^{241}Am gamma rays

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Abstract. Mass attenuation coefficients (μ/ρ) for Zr, Nb, Mo and Pd elements around their K-edges are measured at 14 energies in the range 15.744–28.564 keV using secondary excitation from thin Zr, Nb, Mo, Rh, Pd, Cd and Sn foils. The measurements were carried out at the K_α and K_β energy values of the target elements by two techniques: (1) Proton-induced X-ray emission (PIXE) and (2) ^{241}Am (300 mCi) source. In PIXE, 2 MeV proton-excited X-rays were detected by a Si(Li) detector. In the second case, X-rays excited by 59.54 keV photons from the targets were counted by an HPGe detector under a narrow beam good geometry set-up with sufficient shielding. The results are consistent with theoretical values derived from the XCOM package and indicate that the PIXE data have better statistical accuracy.

Keywords. X-ray attenuation; ^{241}Am; proton-induced X-ray emission; K-edge; thin foils.

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1. Introduction

The energy region near the photoelectric absorption edge in elements is interesting for many reasons. One reason is the validity of the mixture rule [1] for the attenuation coefficients at energies close to the absorption edges. This rule is valid when the effects of the molecular bonding and chemical or crystalline environment on the atomic wave functions are negligible. The attenuation coefficient values are believed

to be affected by chemical, molecular and thermal environments. These phenomena lead to deviations of the observed (μ/ρ) values from theoretical values. It was Jackson [2] who pointed out the possibility of non-validity of the mixture rule near absorption edges. Experimental observations of such effects have been reported by Tan *et al* [3] and Turgat *et al* [4]. Hubbell [5] has calculated the magnitude of the discrepancy between the estimated and theoretical K-edge cross-sections for elements from Ti to Zn to be in the range ± 3–$\pm 12\%$. Another reason for the interest in the K-edge energy region is the importance of the absorption jump factors and jump ratios in many fields of scientific applications like cancer therapy, industrial radiation processing, dosimetry computations, radiation shielding and X-ray fluorescence surface analysis. Also, accurate values of the above parameters make it possible to evaluate various atomic models. In order to study the X-ray interaction processes near the absorption edges, one requires several radiation energies around the edge energy. There are different techniques to obtain the required X-ray energies. Some of these are:

1. Using different radioactive sources which provide discrete X-ray energies, namely the characteristic X-rays of the daughter isotopes: However, availability of such sources is limited. The intensities of X-rays available from such sources also are comparatively low for scattering studies and photoelectric measurements, though they may be sufficient for attenuation measurements. The investigations by Ramachandran *et al* [6], Appaji Gowda *et al* [7,8], Budak *et al* [9], Tajuddin *et al* [10] are typical examples of the use of this method.

2. Using excitation of X-rays from targets with primary photons from radioactive sources: In this method, the incident photons excite the inner shells of the secondary excitation target and the subsequent decay of the excited atom results in the emission of the characteristic X-rays. The same qualitative arguments mentioned for the first method are applicable for this technique also. Several authors, for instance Mallikarjuna [11], Kerur *et al* [12], Angelone *et al* [13] and Turgut *et al* [14] have utilized this technique for attenuation and other types of interaction studies.

3. Using proton-induced X-ray emission (PIXE) which utilizes the secondary excitation from targets upon proton bombardment: The characteristic X-rays which originate from this process can be used to study the inner-shell phenomena as also for attenuation and cross-section measurements effectively at higher intensities. The technique has two definite advantages over the photon-induced X-ray production method. First, the incident flux of protons (accelerated in a particle accelerator) is much larger than the number of incident photons available from radioactive sources. Consequently, the flux of secondary X-rays will be much larger and the statistical accuracy is correspondingly increased. Secondly, the background is relatively less in the PIXE spectrum. However, the reported investigations on X-ray interactions using the PIXE technique are rather limited. Varier and Unnikrishnan [15] have reported attenuation coefficients of copper, tantalum and lead targets using the PIXE technique in the energy region 7–15 keV. The work by Braziewicsz *et al* [16] in the energy range 1–150 keV is also worth mentioning.

4. All the three techniques mentioned above suffer from one common disadvantage, that is the available γ energies are discrete. Continuously variable energy can be made available using Compton scattering of the primary photons from

radioactive sources at various angles. However, the Compton scattering method also requires strong primary photon sources. Also, the scattered radiation will not be strictly monochromatic. It is subject to an energy spread decided by the spread in the scattering angles arising from finite dimensions of the source and target. Budak and Polat [17] and Polat *et al* [18] have applied this technique for the measurement of the K-shell absorption jump factors and jump ratios in some rare earth elements in the energy range 48–59 keV. Again, very recently, we have carried out similar measurements [19] using Compton scattered ^{241}Am γ-rays for the rare earth elements Sm, Eu, Gd, Tb, Dy and Er.

5. An entirely new technique was developed recently by Tamura *et al* [20] to measure mass attenuation coefficients around the K-edge for elements Zr, Nb and Mo. Their technique involves the emission of X-rays around the Bragg angle when relativistic electrons are incident on a crystal plate like silicon. This process is called the parametric X-ray radiation (PXR). The X-rays generated by this technique are highly monochromatic, have continuously variable energy and are directed and coherent.

6. Another emerging method is the use of synchrotron radiation which is likely to be much more useful than any of the methods mentioned above. A typical example of the method based on synchrotron radiation is the work of Chantler *et al* [21] for the measurement of the X-ray mass attenuation coefficient of copper using 8.85–20 keV synchrotron radiation. The accuracies for attenuation coefficients of copper in the above energy range are 0.27–0.5%, with 0.02% reproducibility.

A large number of measurements have been reported over the years on the experimental determination of attenuation coefficients [19,22,23]. On the theoretical side, several compilations are available for obtaining theoretical values of the attenuation coefficients with which the experimental values can be compared. The XCOM package [24–26] is a very powerful and useful computer program and database which can be used to calculate, with a personal computer, photon cross-sections for scattering, photoelectric absorption and pair production. Total attenuation coefficients, in any element, compound or mixture can also be obtained at energies from 1 keV to 100 GeV. However, the XCOM has its own limitations. The cross-sections for elements in the XCOM database pertain to isolated neutral atoms, and do not take into account molecular and solid-state effects which modify the cross-sections, especially in the vicinity of absorption edges. Thus, it would be highly desirable to obtain accurate experimental values of the attenuation coefficients close to the absorption edges.

Under favourable conditions of proton energy and current, the PIXE method of X-ray production is expected to be superior to the photon-induced X-ray generation as a source of the X-radiations for the interaction studies. This is so, especially because of the comparatively lower background in the PIXE spectra and the relatively larger incident intensities available. However, as mentioned already, PIXE-based studies have been rather limited. We felt that it would be highly relevant to have a comparison of the above two methods for γ interaction studies.

To this end, we have carried out measurements of the attenuation coefficients near the K-absorption edges of Zr, Nb, Mo and Pd metals at 15.74, 16.58, 17.44, 17.63, 18.70, 19.6, 20.16, 21.12, 22.71, 23.11, 23.81, 25.19, 26.17, 28.56 keV energies [27] using secondary excitation from Zr, Nb, Mo, Rh, Pd, Cd and Sn foils. The

measurements are done by two independent techniques: (1) the PIXE method and (2) the radioactive ^{241}Am source. The details of the experimental arrangement and procedure, the results derived therefrom, a comparison with the theory and conclusions drawn are reported in this paper.

2. Experimental method

The Particle Irradiation Facility at the Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam, India, was used for the measurements using the PIXE method. A schematic drawing of the experimental arrangement is presented in figure 1. A collimated beam of protons (P) accelerated to 2 MeV, bombarded the target (T) mounted on an aluminium target ladder inside the PIXE chamber. The entire beam path from the ion source through the accelerating tubes and magnets to the target chamber was maintained at a pressure of 10^{-8} to 10^{-7} Torr. Turbomolecular pumps were installed at various locations for evacuating the entire proton beam path. The PIXE chamber, made of stainless steel, had a graphite beam collimator of 3 mm diameter to avoid the background X-rays originating from the scattered protons hitting the chamber walls. The vacuum inside the chamber was maintained through the port (V) and the ladder position could be adjusted by looking through the beam view port (B). In between the detector and the target, there was a 100 μ thick polythene filter to filter out the backscattered protons. The electronics and data acquisition system for the attenuation studies consisted of a CANBERRA Si(Li) detector (D) with an active area of 30 mm^2 kept at an angle of 45° with the incident beam direction. The 3 mm thick detector had a resolution of 155 eV at 5.9 keV and had a 0.025 mm thick berillium window. The measuring system also included a spectroscopy amplifier (ORTEC-672) and a multichannel analyser (FAST COMTEC) with 8K ADC. Self-supporting 25 μ thin foils of Zr, Nb, Mo, Rh, Pd, Cd and Sn of size 1.5 cm × 1.5 cm were procured from Johnson Matthey Chemicals India Pvt. Ltd., Mumbai. The target holder was an aluminium ladder of size 15 cm × 3 cm. The position of the targets mounted on the ladder could be changed from outside without breaking the vacuum. The X-ray beam (X)

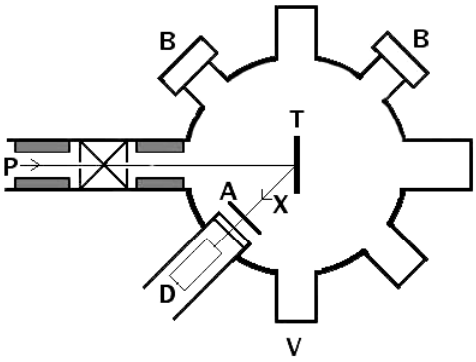

Figure 1. Schematic diagram of the experimental set-up for γ attenuation measurements using the PIXE method.

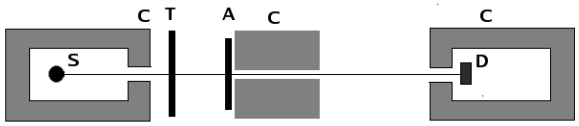

Figure 2. Schematic diagram of the experimental set-up for narrow beam geometry photon attenuation measurements using ^{241}Am source.

emerging from the target in the direction of the detector (D) could pass through the absorber (A). The absorbers were in the form of thin foils of size 1.5 cm $\times$ 1.5 cm and thickness 25 μ of Zr, Nb, Mo and Pd elements placed in front of the X-ray detector. The X-rays transmitted through the absorber were detected in a narrow beam good geometry by the Si(Li) detector. Because of limitations on the geometry and dimensions of the PIXE chamber, the distance between the target and the detector was 9 cm and the in-scattering angle in the narrow beam set up was around 2°. The corresponding in-scattering correction was calculated to be less than 0.1% and the attenuation set up could be considered to be a narrow beam good geometry arrangement, which was an essential requirement for attenuation measurements. The proton current entering the scattering chamber was maintained at several nA. The X-ray exciter targets were irradiated for a preset total charge of typically 10 μC, as measured on the target ladder by using an ORTEC model 439 current digitizer.

For the measurements using ^{241}Am source, we have used the set up shown in figure 2. A 300 mCi, ^{241}Am source for the present measurements had been procured from M/s Amersham, England. The source (S) was well shielded and a narrow γ-ray beam was obtained by means of a collimator (C). A secondary X-ray exciter target (T) was kept in front of the source. The targets and absorbers for the measurements were the same as used in the PIXE studies. X-rays generated in the targets in the forward direction were allowed to fall on the absorber (A). The transmitted X-rays after passing through lead collimators of diameter 1 cm were detected by an ORTEC HPGe Gamma X detector, placed at a distance of 60 cm from the absorber. This ensured a narrow beam good geometry set-up for attenuation measurements. The detector output pulses were shaped and amplified by an ORTEC 571 amplifier. Amplifier output pulses were then fed to a CAMAC-based data acquisition and analysis system, consisting of a kinetic CAMAC crate, crate controller, a 4k Quad ADC, supplied by the Electronics Division, BARC and connected to a personal computer through a suitable interface card. A Linux-based FREEDOM software [28], developed at the Inter University Accelerator Centre, New Delhi, India is used in the present investigations for both online data acquisition and for online as well as offline data analysis. In both the above methods, the peak areas for the direct X-rays and the X-rays transmitted through absorber were extracted from the spectra. In extracting the areas of the non-distinct component peaks, we did not attempt to resolve the separate contributions of the component peaks. The extracted attenuation coefficients are therefore really averages over the component peaks. The mass attenuation coefficients were then calculated from the relation

$$I = I_0 \exp[-(\mu/\rho)x\rho],$$

where μ is the linear attenuation coefficient (cm^{-1}), ρ is the density of the sample (g cm^{-3}), x is the thickness of the absorber (cm), I_0 is the count value without the absorber and I is the count value of the radiation penetrating through the absorber. In-scattering contributions in both the PIXE method and the ^{241}Am source method were found to be much small compared to the experimental uncertainties. We have carried out the measurement twice for each absorber element and the average attenuation coefficient values were determined. Errors in the extracted attenuation coefficient values have been estimated taking into account statistical uncertainties, the process of averaging of the two sets of measurements for each element, non-uniformities in the target thickness and the impurities in the target. We have ensured that the combined error due to the last two sources is less than about 0.5%.

3. Results

Typical X-ray spectra for the direct beam and the beam transmitted through the absorbers are shown in figure 3, for measurements using PIXE. Counting time was roughly around 30 min for each spectrum, keeping the total proton charge at a constant value of 10 μC, as measured by the current integrator.

The attenuation coefficient values, obtained in the present measurements by the two different techniques based on PIXE and ^{241}Am source are tabulated and compared with the corresponding XCOM values in tables 1 and 2.

The attenuation coefficients are also plotted as a function of the X-ray energy in figures 4–7. The errors associated with experimental (μ/ρ) data in each case, which are mainly due to statistical uncertainties are also shown. The respective K-edge energies are indicated in these figures. The XCOM values are plotted as solid curves for comparison. In addition, we have plotted the experimental results

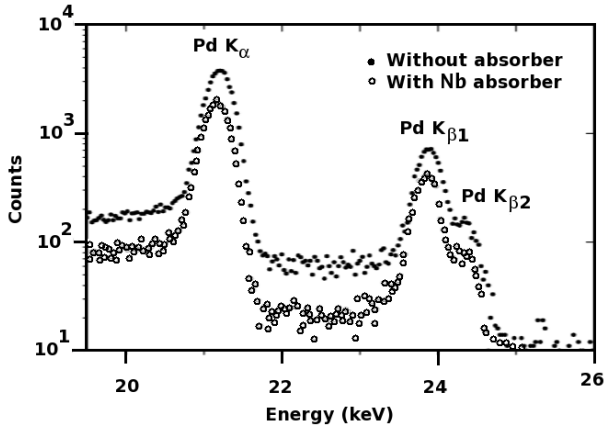

Figure 3. Spectra with and without the thin niobium foil absorber for proton-excited Pd X-rays.

Table 1. Comparison of the measured values of (μ/ρ) in cm^2/g with XCOM values for Zr and Nb.

	Zr			Nb		
Energy (keV)	PIXE	^{241}Am	XCOM	PIXE	^{241}Am	XCOM
15.74	20.78±0.36	19.03±0.55	21.58	21.19±0.43	26.80±0.91	23.41
16.58	16.58±0.28	20.48±0.44	18.74	17.96±1.92	17.92±0.73	20.33
17.44	14.88±0.29	17.47±0.53	16.34	15.93±0.32	14.27±0.41	17.72
17.63	18.33±1.47	12.42±1.30	15.87	15.83±0.64	18.51±0.82	17.21
18.70	81.20±1.75	83.68±3.86	85.92	12.92±0.39	11.87±0.77	14.68
19.60	69.71±2.02	71.94±2.99	76.23	76.40±2.94	74.38±3.40	81.01
20.16	64.83±3.76	67.40±3.45	70.87	77.21±4.48	80.01±4.30	75.52
21.12	59.80±1.33	66.40±2.67	62.90	64.93±2.53	68.23±2.86	67.16
22.71	53.65±3.70	50.33±3.51	52.07	52.93±3.65	48.45±3.20	55.72
23.11	46.82±1.42	47.93±1.87	49.78	50.47±1.94	53.78±2.61	53.29
23.81	43.83±2.19	39.88±2.40	46.02	46.30±2.69	45.07±2.69	49.29
25.19	38.69±0.86	36.20±1.40	39.64	40.81±1.29	41.98±2.11	42.48
26.17	33.84±1.65	37.43±1.90	35.83	35.85±2.45	39.09±2.28	38.41
28.56	26.23±1.54	25.40±1.61	28.30	28.07±1.54	33.41±2.30	30.04

Table 2. Comparison of the measured values of (μ/ρ) in cm^2/g with XCOM values for Mo and Pd.

	Mo			Pd		
Energy (keV)	PIXE	^{241}Am	XCOM	PIXE	^{241}Am	XCOM
15.74	23.12±0.44	21.29±0.62	24.97	30.80±0.16	34.41±1.07	32.56
16.58	19.80±1.85	18.90±0.59	21.66	25.97±0.17	27.26±0.57	28.29
17.44	16.01±2.45	21.41±0.39	18.86	22.43±0.19	21.04±0.47	24.66
17.63	16.93±0.49	15.25±0.59	18.30	22.24±0.35	18.83±0.77	23.94
18.70	14.11±0.54	13.08±0.68	15.61	19.86±0.38	24.24±1.35	20.43
19.60	11.23±0.34	16.20±0.63	13.79	16.98±0.47	17.43±0.68	18.00
20.16	76.44±3.68	74.38±4.14	79.49	12.50±0.72	14.40±0.63	16.67
21.12	74.22±2.24	80.23±2.29	77.11	14.50±0.27	12.59±0.41	14.70
22.71	70.31±4.15	63.71±4.41	68.10	09.28±0.62	09.24±0.59	12.09
23.11	62.08±2.00	60.02±1.89	65.39	12.19±0.36	13.28±0.44	11.54
23.81	56.39±2.88	54.34±2.13	60.43	10.52±0.60	08.98±0.48	10.65
25.19	48.21±1.39	47.84±1.67	50.90	54.15±0.73	49.96±1.34	54.15
26.17	43.05±2.47	51.30±2.34	44.83	49.46±2.40	52.00±2.89	49.18
28.56	35.19±1.78	28.03±1.41	33.08	35.00±1.88	35.59±1.78	39.32

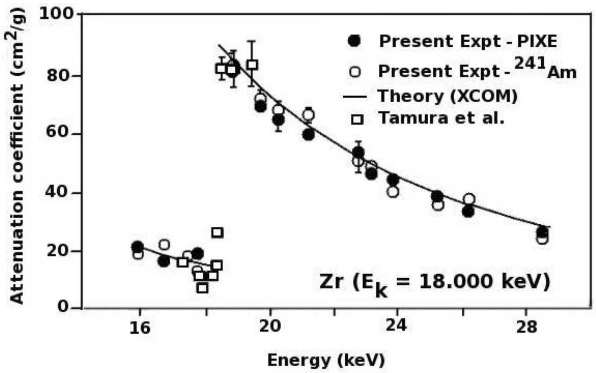

Figure 4. Comparison of the present experimental results obtained by the PIXE and ^{241}Am methods with XCOM values for Zr.

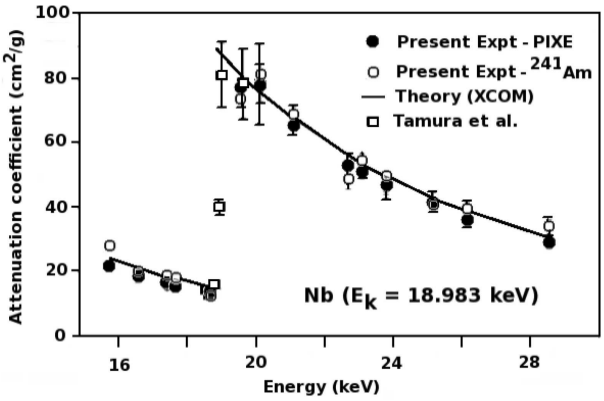

Figure 5. Comparison of the present experimental results obtained by the PIXE and ^{241}Am methods with XCOM values for Nb.

of Tamura *et al* [20] for Zr, Nb and Mo elements. For Pd, we have compared our results with the values of Deslattes [29] as quoted by Saloman and Hubbell [23].

4. Discussion and conclusions

It can be seen from figures 4–7 that the two sets of experimental results for the attenuation coefficients obtained in the present work agree reasonably well with each other within the experimental uncertainties, except at one or two energies in each case. The reasonable agreement between the results of the two sets of measurements gives confidence in the measurement techniques adopted in the two sets of measurements. The data of Tamura *et al* [20] in the case of Zr, Nb and Mo, plotted in figures 4–6 for the range of energies from 17.22–20.56 keV are also seen to be consistent with our data. However, the present experiment covers a still wider range, 15.74–28.56 keV. Our data for the element Pd are also in agreement with

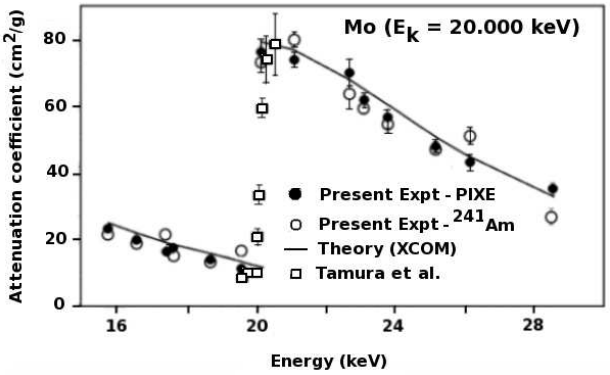

Figure 6. Comparison of the present experimental results obtained by the PIXE and ^{241}Am methods with XCOM values for Mo.

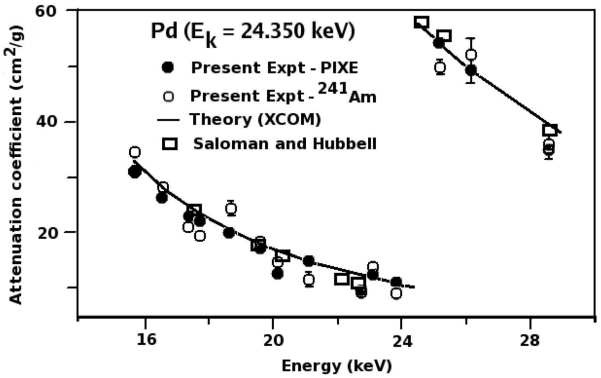

Figure 7. Comparison of the present experimental results obtained by the PIXE and ^{241}Am methods with XCOM values for Pd.

those of Deslattes [29] as given by Saloman and Hubbell [23]. The experimental results of Angelone *et al* [13] at 17.44 keV for Zr, Nb, Mo and Pd, and at 19.6 keV for Nb and Pd are also consistent with the present values.

Saloman and Hubbell [23] in their compilation of X-ray attenuation cross-sections for energies 100 eV to 100 keV for the elements $Z = 1$–92, have presented several other sets of experimental values for the attenuation coefficients for Zr, Nb, Mo and Pd elements in the energy range of the present measurements. It is seen that there is an overall agreement to within $\pm 10\%$ for most of the reported values for the attenuation coefficients with the corresponding XCOM values in the case of Zr, Nb and Pd. In the case of Mo, agreement is observed only to within $\pm 15\%$ at energies below the K-edge. However, a gross mismatch is seen at the above edge energies where the experimental values are consistently below the XCOM values.

Figures 4–7 show that our experimental results are, in general, close to the theoretical values (XCOM), represented by the solid curves. However, some of the PIXE values tend to be on the lower side. We feel that more experimental investigations are needed to resolve this discrepancy.

The better accuracy of the PIXE results is worth mentioning in this context. This is obviously due to the larger intensities of the incident X-rays available with this technique as well as the relatively lower background levels. We feel therefore that further investigations on X-ray attenuation near K-edge energies using the PIXE technique would be highly desirable for other elements also. Keeping in view the fairly close comparison of the present results with theory, calculation of dispersion corrections to the forward Rayleigh scattering amplitude [8,30] of Zr, Nb, Mo and Pd elements has been carried out therefrom. The results are being communicated elsewhere.

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