Technical Note

On the Reduction of Background in Energy Dispersive X-Ray Fluorescence Analysis[†]

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1. Introduction

To obtain the high sensitivity in energy dispersive X-ray fluorescence (XRF) analysis, measurement with low background is required. Though many important elements with atomic numbers smaller than about 23 are contained in environmental, biological or medical samples, little trial to reduce the background has been reported in the few keV X-ray energy region. In this paper is described the reduction of X-ray background under the condition that a Si(Li) X-ray detector is almost free from irradiation of backscattered photons by the use of a large volume vacuum chamber.

2. Experimental Setup

Experimental setup is shown in Fig. 1. X-ray detection was made with a Si(Li) detector of 3.8 mm effective diameter through a 7.6 μ m Be window. A 78 MBq (2.1 mCi) ⁵⁵Fe annular source was used as the primary photon source. In the detection of a few keV X-rays, it is desired to place both a sample and a detector in vacuum to eliminate attenuation of the X-rays and generation of the Ar-K line from the air. Moreover, since the majority of primary photons transmit a thin sample, a large volume evacuated chamber is necessary to minimize the X-ray background due to back-scattering from the chamber wall. Therefore, a lucite cylinder of

110 cm in length and 31 cm in diameter was employed (see Fig. 1(A)); lucite was used to avoid the generation of interfering X-rays. For comparison, a smaller lucite cylinder (50 cm long $\times$ 5 cm diameter) was also tested (see Fig. 1(A')). As shown in Fig. 1(B), a lucite colli-

Fig. 1 (A) Experimental arrangement with large lucite chamber.

(A') Arrangement with small chamber designed for preliminary test.

(B) Partial cross section of a Si(Li) detector, an annular source of ⁵⁵Fe and a sample with 2.5 μ m Mylar backing.

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material was attached to the detector so that the Si(Li) X-ray detector could view only the sample layer and the far distant part of the lucite cylinder.

3. Background Spectra

In Fig. 2, observed background spectra are shown. Spectra (A') and (A) correspond to the measurements using smaller (Fig. 1(A')) and larger (Fig. 1(A)) cylinders, respectively. Both of them were obtained with the ^{55}Fe source and a $2.5\text{ }\mu\text{m}$ Mylar film attached on the sample holder. The comparison of spectra (A') and (A) clearly shows the advantage of use of large volume chamber.

Fig. 2 Background spectrum obtained with a small chamber (Fig. 1(A')).
 (A') ^{55}Fe and $2.5\text{ }\mu\text{m}$ Mylar.
 Background spectra obtained with a large chamber (Fig. 1(A)).
 (A) ^{55}Fe and $2.5\text{ }\mu\text{m}$ Mylar.
 (B) ^{55}Fe only.
 (C) Natural background.
 (D) Calculation of photoelectron bremsstrahlung (PEB) for ^{55}Fe and $2.5\text{ }\mu\text{m}$ Mylar.
 Mn-K X-rays (^{55}Fe) directly observed by the detector are shown by the dashed curve (E).

The spectrum (B) exhibits the similar measurement as (A) but the Mylar film is removed.

Spectrum (C) shows the natural background without the ^{55}Fe source. The drastic decrease of backscattering peaks in the case of (B) (no Mylar film) as compared with (A) implies that the coherent scattering mainly comes from the Mylar film. Contrary to this, the difference between (A) and (B) in the low energy region (1~4.5 keV) is at most 10%. This fact means that the emission of photoelectron bremsstrahlung (PEB) caused in Mylar film is of minor contribution to the observed background, whereas in an ideal XRF measurement PEB is considered to be a dominant factor of the background. The roughly estimated PEB spectrum is shown in curve (D) in Fig. 2. The calculation is based on the procedure described by Goulding, et al.¹¹ Clearly, curve (D) differs from (A) in both magnitude and spectrum shape.

The dashed curve (E) in Fig. 2 shows the normalized Mn-K X-ray spectrum obtained when the ^{55}Fe source was placed at the sample position. The low energy tail of the $\text{K}\alpha$ line due to incompleteness of charge collection is found to contribute little to the background in low energy region concerned. In our ^{55}Fe source, the existence of ^{60}Co (5.9 kBq (0.16 μCi)) was observed by the γ -ray measurement using a Ge(Li) detector. We estimate that the origin of low energy background in the spectra (A) and (B) is partially due to the low energy Compton tail of γ -rays emitted from ^{60}Co source, and partially due to the primary Mn-K X-rays multiply scattered from lucite walls.

4. X-Ray Spectrum

Typical XRF spectrum in the present apparatus for the elemental analysis of Chinese medicinal herb "Furmariaceae" is displayed in Fig. 3. In the figure is also drawn the X-ray spectrum measured by PIXE method, in which the same sample was irradiated by 20 MeV α particles supplied by the tandem accelerator of University of Tsukuba. In Fig. 4, XRF detection limits obtained by our apparatus are shown as function of sample atomic numbers. The samples were evaporated onto a $2.5\text{ }\mu\text{m}$ Mylar and the detection limit in Fig. 4 means the amount of sample element expressed in unit of g/cm^2 when the ratio of K X-ray to background areas becomes

Fig. 3 Comparison of observed X-ray spectra between XRF (^{55}Fe) and PIXE (20 MeV α) for the sample of Chinese medical herb.

unity within the FWHM of X-ray peak.

In conclusion, it can be said that a large volume lucite chamber is useful to reduce the background induced by the backscattering of primary photons, and the background in XRF measurement is attributed to the measuring apparatus itself. In contrast to PIXE where the background is mainly composed of the bremsstrahlung due to the secondary electrons in a sample, contribution of the photoelectron bremsstrahlung (PEB) is more than one order of magnitude lower than the observed background.

Fig. 4 Observed XRF detection limits for K X-rays as function of elemental atomic numbers.

Therefore, the attainment of high sensitivity in XRF detection depends on the improvement of apparatus the background of which may come close to the one due to PEB.

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Reference

- 1) F.S. Goulding and M.J. Jaklevic: *Nucl. Instr. and Meth.*, 142, 323 (1977)