

SEA NO. 14 JUL.1998 Qualitative Techniques with SEA

1. Introduction

The SEA Bench-Top X-ray Fluorescence Analyzer was designed to be used by those without an extensive knowledge of X-rays. Nonetheless, the process of identifying elements from a spectrum requires some knowledge of X-rays. This application brief offers advice for performing qualitative analysis with SEA series instruments.

2. Auto ID Reliability

SEA instruments have an automatic identification function for identifying elements from the peak position of their spectrums. Unfortunately, this function is not totally reliable. Mistakes in identification can happen in the following situations:

- A. When measuring at a tube voltage of 15 kV
- B. In the case of rare earth elements
- C. When other element X-ray energies overlap

Typical examples are the L line of Mo and M line of Pb and S, M line of W and Si, L line of Rh and Cl, L line of Ba and Ti, Pb and As, L line of Br and Al.

3. Typical Interference Peaks

Peaks that appears in a spectrum include interference peaks generated by a number of causes other than fluorescent X-ray peaks from elements. Interference peaks must be understood in order to identify elements. There are four types of interference peaks:

- A. Scattered rays
- B. Escape peak
- C. Sum peak
- D. Diffracted rays

A. Scattered rays

When atoms from a sample reach the detector the primary X-rays from the X-ray tube are scattered by the atoms. There are two kinds of scattering: (1) Rayleigh Scattering, in which there is no change of energy and (2) Compton Scattering in which there is a drop in energy.

X-rays from the tube include continuous X-rays and X-rays characteristic of the target material. Scattered rays of continuous X-rays do not form a peak but those of characteristic X-rays are detected as peaks. As a result, the energy position of characteristic X-rays of the target metal appears as a peak. The fairly wide peaks of Compton scattering can be verified at somewhat lower energy positions.

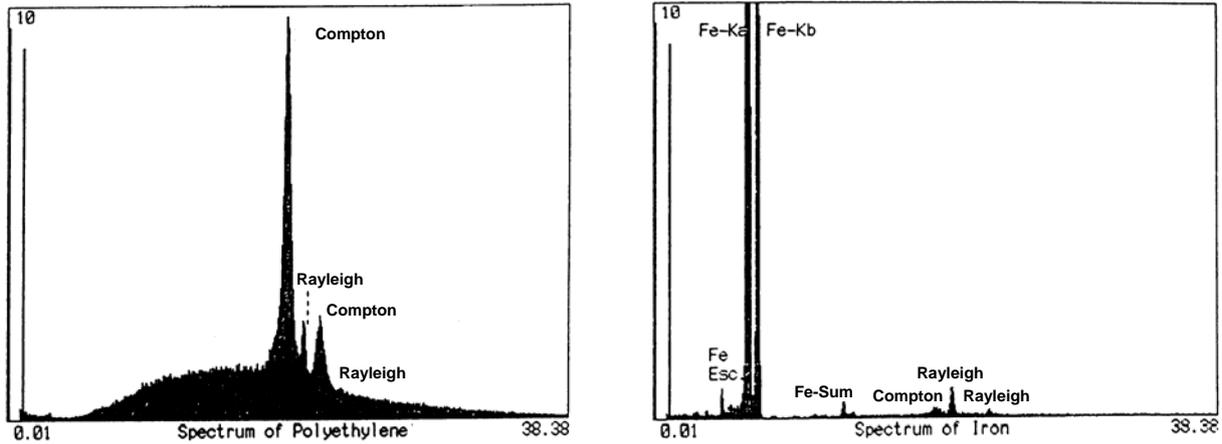


Figure 1 Scattered ray intensity changes according to main component atomic number

The scattered rays are more strongly detected as the atomic number of the sample decreases. Background increases when measuring light elements such as aluminum alloys or organic material, but this is due to the scattered ray intensity of continuous X-rays. The intensity of Compton scattering increases as the atomic number of the sample increases. This characteristic allows you to roughly estimate, by looking at the ratio of the Compton intensity vs. Rayleigh intensity, the existence of light elements whose X-rays cannot be detected.

B. Escape peak

The semi-conductor detector is a silicon crystal that is excited when hit by enough X-ray energy. As the X-rays lose energy to excitation, the value of the detector output decreases by the amount lost to excitation. As a result, a small peak will appear at an energy position lower than the main peak (only the energy needed to excite silicon). This peak is called the escape peak.

The position the escape peak can be accurately estimated from theory. The SEA2010 is able to display a marker that indicates that position. Normally, the peak is difficult to make out and is not displayed. The [ESC/ON] key on the ten key pad switches between display and not displayed.

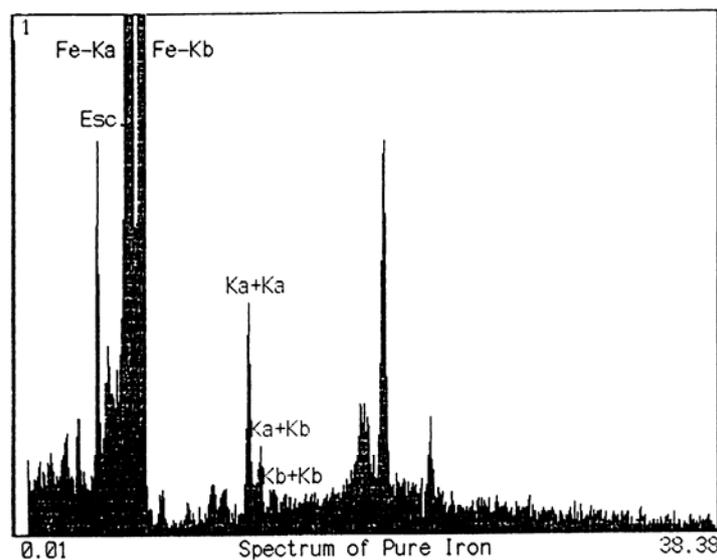


Figure 2 Escape peak and Sum peak

C. Sum peak

Individual X-ray photons are detected by the energy dispersion detector. Sometimes, however, two or more X-ray photons are detected at the same time. Detector output at this time is the same as one X-ray photon made up of the energies of two or more X-ray photons. The peak that appears at an energy position made up of two or more X-ray photons is called a sum peak.

For example, the peak location of the characteristic X-ray of Fe is 6.4 keV (K alpha) and 7.0 keV (K beta). When Fe has a high density, small peaks can be found at the positions of 12.8 keV (K alpha + K alpha) and 13.4 keV (K alpha + K beta). A small peak also appears near 14.0 keV.

The probability of simultaneously detecting two or more X-ray photons increases as the number of photons increases. In the example above, the intensity of the high K alpha companion sum peak (12.8 keV) is the largest and becomes the next sum peak (13.4 keV) of K alpha + K beta.

When a sample has complex elements at high densities such as SUS, the sum peak becomes complex. However, you can easily determine whether a peak is a sum peak by "adding high peak energy companions."

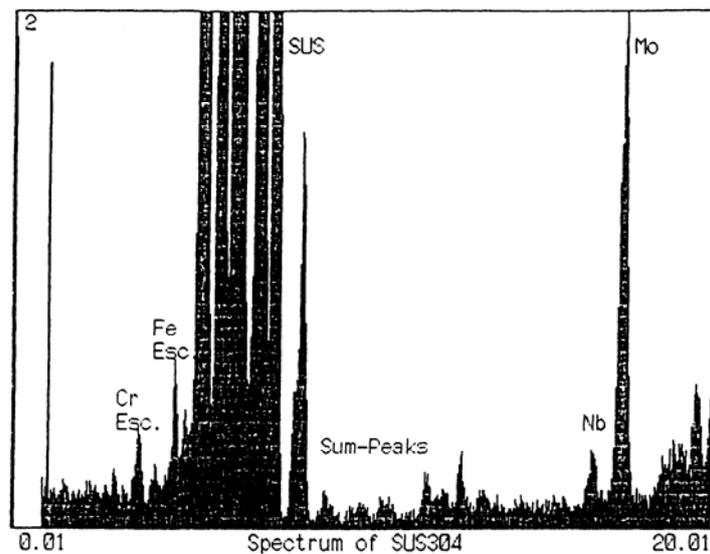


Figure 3 Sum Peak when multiple elements with high densities are included

D. Diffracted rays

X-ray diffraction occurs when a particular wavelength of X-ray is emphasized by the grating constant (distance between molecules) of a crystal and the angle of incidence when the X-rays strike the crystal. Usually the grating constant of a crystal is found (X-ray diffraction method) by measuring while changing angles at a fixed X-ray wavelength (using a monochromatic light source).

X-rays irradiate as an excitation source even in fluorescent X-ray analysis. Furthermore, since continuous X-rays are also irradiated, the diffraction conditions of only X-rays of a certain wavelength (energy) are satisfied and may be detected when measuring a crystal sample. The detected X-ray is called the diffraction line and the peak that appears at the energy position is called the diffraction peak.

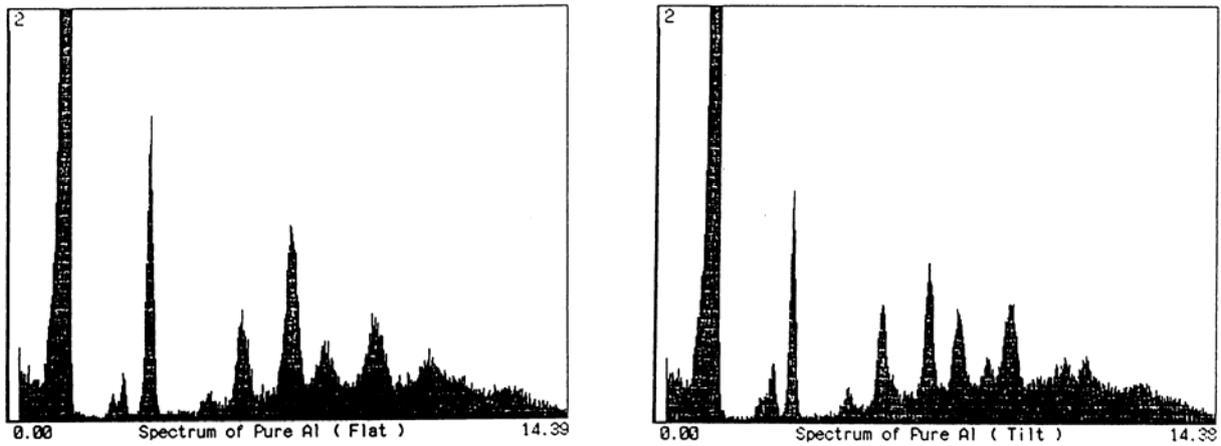


Figure 4 Diffraction changes according to the slope of the sample

Because the position cannot be specified, the diffraction peak cannot be identified on one spectrum, but by changing the angle of the crystal surface the position of the peak also changes. Therefore, tilting the sample and re-measuring can identify a peak. If the peak position changes it is a diffraction peak and if it does not change, then the cause is something else.

4. Summary

You can avoid most mistakes in identifying elements if you observe the points listed herein. However, in the end, there are many cases in which knowledge of the sample is a decisive factor. Please be aware that answers given by an analytical device is not absolute.