**Kilde QUANTAX Microanalysis system based on energy-dispersive spectrometry. User manual. BRUKER-AXS Microanalysis GmbH, Berlin, Germany. Issue April 02, 2008.**

**Xflash® Spectrometer**

Detector type: Silicon drift droplet detector (SD3)

Collimator material: Zirconium

Cooling: Thermoelectric, convection (vibration free)

**Microanalysis Basics**

SEM scans the sample with a beam of high-energy electrons. During interaction of these electrons with atoms of the sample secondary electrons, backscattered electrons, characteristic X-rays and bremsstrahlung are emitted. While the SEM deliver images of surface structure and topology using SE and BSE respectively, the EDX gives detailed information about the chemical composition for the measured spot or area using characteristic X-rays.

*Qualitative analysis:* Each of the chemical elements in the sample emits characteristic X-rays. The energy of these lines are nearly independent of the chemical condition of the atoms. In the acquired energy dispersive spectrum the characteristic X-rays correspond to visible peaks. By identifying which elements characteristic X-rays that corresponds to each peak yields information about the elements that are present in the sample.

***IKKE OMSKREVET NOE AV DET SOM FØLGER!!!***

*Quantitative analysis:* Quantitative data about the composition of the sample are derived from the different peak intensities by an extensive mathematical process, often reffered to as ZAF or PhiRhoZ matrix correction. Bremsstrahlung X-rays, which form a continuous spectrum background, is used as additional source of information during the spectrum analysis with self-calibrating methods (P/B-ZAF)

*Analysis limits:* EDX is surface sensitive. The thickness of the surface layer from which most of the analytic signal is originating (information depth) is defined on one hand by the ability of the high-energy electron beam to penetrate into the sample, on the other hand by the length of the path the generated X-rays can travel back through the sample. Since this path is substantially longer than depthjs SE and BSE can penetrate the information dfepth of EDX is accordingly larger than that of SEM imaging.

***BILDE!*** Electron beam hitting a sample surface. The multicolored region is the volume which is penetrated by the multiply scattered primary electrons and exited to produce characteristic X-ray radiation. The yellow and red ranges are the volumes from which SE and BSE can escape from the surface respectively. The red and blue area are the origin of most of the characteristic X-rays and bremsstrahlung respectively.

*Depth distribution function*. The information depth spans from part of a micron to some microns depending on the high voltage of the elctron gun, the mean density of the sample matter, and the element content. Calculated depth distribution functions for a certain sample are available after quantitative spectrum analysis.

*Lateral scattering.* In deeper regions the electrons become scattered in lateral direction forming a pear shaped volume in which atoms are exited to produce X-rays. This lateral size in combination with the depth distribution defines the analysis volume, it’s normally in the order of magnitude 1 cubic micrometre.

The three dimensional size of the analysis volume is limiting the spatial resolution of X-ray microanalysis to values much less than the elctron image resolution of modern electron microscopes. At a certain point the spatial resolution becomes independentof decreasing spot size. In contrary, intentionally defocusing the electron beam can be used to decrease sensitivity to small artifacts. Moderate magnification values are adequate for X-ray mapping and similar applications.

As outlines abocve reducing the high voltage of the electron gun enhances the spatial resolution. Excitation using low energy characteristic X-ray lines (L- and M-line series) which will travel only a short distance in matter, can further enhance lateral resolution. But in this case detection of X-rayus becomes much more critical, and analysis will be increasingly sensitive to surface contamination and specimen condition.

Inhommogeneous samples require special consideration when using X-ray microanalysis . Point analysis inadvertently performed on textures material will lead to random results. Averaging a larger sample surface with inhomogeneous samples will generally not result in mean concentration values. At first this is due to the fact that numerical two-dimensional distribution of the constituents neither matches volume cntents nor mass concentrations. Secondly, none-linear relationsships of spectrum analysis prevent simple averagingof spectra from being adequate. The second reason also applies when averaging on rough sample areas, even if individual point analysuis results are correct.

Because of the small analysis volume X-ray microanalysis is extraordinary sensitive. Absolute sensitivity in the order od 0.1 pg and well below can be achived in many cases. In connection with thin sections – as used by TEM analysis – even total amount of 1.0E-19 g can be detected. This corresponds to only a few thousand atoms.

3.5.5. Limit of detection

Despite of the extraordinary low absolute detection limits of X-ray microanalysis, the limit of detection (LOD) of EDX in terms of concentration is restricted by the vrensstrahlung background, which is always present. In average and when using moderate measurement times, the detection limit for trace elements is rougly 0.1 % mass concentration.

Of course this value is modulated by the matrix, being lower within biological or geological matrices. Optimum analysis cnoditions abd very long aquistion times can shift the limits.

3.5.6. Accuracy and Precision

The precision of quantitative EDX is mainly governed by counting statistics of the discretely counted X-ray quanta. Using fast X-ray detectors statisticdal errors well below 1% can easily be achieved at moderate collecting times.

Accuracy is influenced by a great number of factors ranging from the property of the sample itself, the selection of elements to be analysid, the possibility of severe X-ray line overlap, to the selection of the general quantification method. In average cases advanced standardless X-ray microanalysis has proven to be reliable abd acurate, allowing error levels as low as 3 to 5 % relatively.

In principle accuracy of standard related analysis, is only limited by the quality of standards and the stability of the beam at the SEM. However, error levels down to statistical limits, require suitable samplesm and optimum operating condistions.