



## **Poster Session I. Aerosol chemistry**

### **OPTIMISATION OF LIGHT ELEMENT ANALYSIS OF INDIVIDUAL PARTICLES USING UTW-EPMA**

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Keywords: particle characterisation, instrument evaluation, light elements, ultra-thin window EPMA

#### **INTRODUCTION**

Conventional electron probe microanalysis (EPMA) has proven to be a powerful technique to analyse atmospheric particles. The combination of electron microscopic imaging with energy dispersive X-ray detection allows us to characterise large numbers of individual particles in an automated, computer-controlled way (detection limits 0.1 %, particle diameter > 0.2 µm). However, due to strong absorption of low energy X-rays in the thick beryllium window of the detector (8 µm), conventional analysis is limited to elements with atomic number  $Z > 11$ . To analyse low- $Z$  elements, the beryllium window should be removed (windowless EPMA, causing contamination of the detector crystal), or it should be replaced by an ultra-thin window (UTW). Since our research is in the field of atmospheric chemistry, we are very interested in the capabilities of UTW-EPMA, because aerosol particles contain high concentrations of light elements, like carbon, nitrogen and oxygen.

#### **CURRENT AND FUTURE RESEARCH**

The Jeol 733-JXA instrument in our facility has been equipped with an Oxford Link Pentafet Si(Li) atmospheric-thin window detector (resolution 133 eV for Mn-K $\alpha$ ). In earlier research (Ro *et al.*, 1999; Osán *et al.*, 2000; Szalóki *et al.*, 2000) we proved that windowless and UTW-EPMA are capable of at least semi-quantitative analysis of individual particles, using a modified version of the CASINO Monte Carlo simulation program by Hovington *et al.* (1997). Through iteration of the simulation results, the concentration of the detected elements in standard particles down to 0.3 µm can be calculated with good agreement between expected and calculated concentrations (within 3–8 % relative). Analysis on standard particles and real atmospheric particles (sampled in urban and marine environment), has pointed out that besides a more profound particle characterisation based on elemental results, also molecular information can be obtained.

Although the first results showed to be very promising, the method was not yet completely optimised. In our current research we mainly try to find answers to basic problems with which the method has to cope. For instance, we tried to compare different particle collection substrates on the basis of their physical and chemical properties: besides their purity and conductivity, special attention was paid to their influence on the background continuum and probable peak overlaps with particle peaks. A problem similar to the latter, is the fact that L- and M-lines of heavy elements (in the particles or in the substrate) may overlap with the K-lines of light elements. Work has been done on these effects, since spectrum analysis must take into account their intensity contributions to low- $Z$  peaks.

The influence of vacuum impurities was investigated, e.g. because oil coming from the rotation pumps might contribute to the carbon and oxygen intensity signals. Another vacuum problem can be found in the presence of volatile particle compounds. The combination of low pressure and local temperature rise due to the interaction with the electron beam, causes evaporation of e.g. ammonium sulphate and

nitrate. To (partly) prevent this from happening, the use of a cold sample stage, cooled with liquid nitrogen, has been studied.

A combination of the previous problems is the water content of particles: evaporation might occur in the vacuum, but the remaining water will contribute to the oxygen signal. Since there is no other element to trace water (hydrogen is not detected), a study of its influence has been carried out in function of the relative humidity to which standard particles had been exposed.

Besides answering basic questions and working out a quantification method for the simultaneous determination of light and heavy elements, our goal is to combine UTW-EPMA with other techniques. In the past, 'spot tests' have been developed (Bigg *et al.*, 1974): through microchemical particle transformations, specific compounds can be visually determined, e.g. nitrate, sulphate and chloride. Combining these tests with low-Z analysis might give more information on molecular species in the particles. Another method we want to combine, is the recent 'Grazing Exit' technique (Tsuji *et al.*, 1999), in which the respective orientation of detector and specimen is changed to enhance intensities or to specifically collect X-ray signals from the top layers of the particles. This might give us more information on particle heterogeneity.

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