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SEM-EDS Quantitative Analysis of Aerosols ≥ 80nm: Impacts on Atmospheric Aerosol Characterization Campaigns

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Time, size and elementally resolved aerosol analysis is a necessity to fully characterize a pollution plume, whether it be under ambient conditions or for personal security in an industrial or military application. The US-EPA recently passed the PM 2.5 regulations that limit the emissions of particles with diameters less than 2.5 μm. These regulations have been put into place due to the increased understanding of the detrimental health effects of aerosols.[1]

As a result of the need for better aerosol collection and analysis techniques we have designed a rugged, cost efficient, time resolved field based aerosol collector for widespread deployment and sampling. Airborne particulates are collected via impaction techniques and stored for detailed analysis using lab based SEM-EDS. Recently several groups have analyzed atmospheric aerosols using electron microscopy coupled with X-ray analysis.[2-4] All of these groups have noted the benefits of analyzing sub micron particles on polymer coated copper grids. Laskin and Cowin make the specific point of ensuring the copper grid is mounted suspended such that X-rays are not generated from the specimen mount. We have taken a similar approach to the microanalysis of sub-micron particles, however as seen in Fig 1 we mount the polymer coated copper grids on a standard aluminum stub which has been hollowed out to achieve low background counts, see Fig 2.

Prior to analysis of atmospherically collected particles we are performing a calibration of the collector and X-ray analysis techniques. We have collected and manually analyzed NaCl, Al_2O_3, KCl, Fe_2O_3, SiO_2, and AgCl particles ranging in size from 80nm to 10μm at acceleration voltages of 5, 10 and 15keV. Historically, large numbers of environmental particles have been analyzed by automated systems using a combination of imaging techniques for particle location and standardless quantitative analysis. While this approach can work satisfactorily for large particles it breaks down for small particles(< 1 μm).[4] An example of this problem is illustrated in Fig. 3, which contains our recent results on Al_2O_3, showing errors as large as 20%. These errors will propogate to larger magnitudes upon the analysis of atmospherically collected heterogeneous aerosol particles and thus the data will have limited use. To alleviate these problems we are developing sophisticated quantitative analysis algorithms using Monte Carlo techniques which account for particle diameter and shape, variation in elemental excitation volumes, absorption of low energy X-rays, and variations in the mean ionization cross section for the accurate quantitative analysis of small particles. Initial results of these calculations will be discussed.

References

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[5] This research was supported by the NYS-DEC.

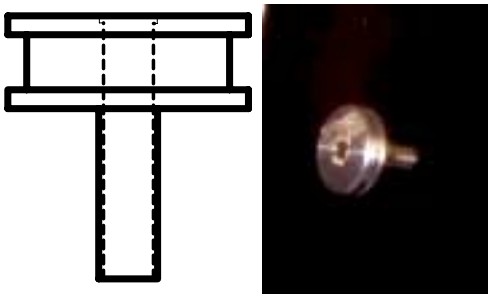

FIG 1. Schematic and picture of standard sample mount hollowed out and with a copper grid sample for minimum background generation

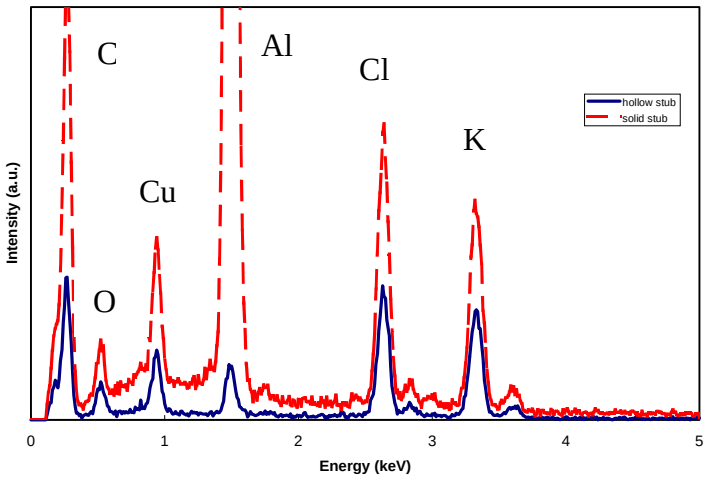

FIG 2. EDS spectra from 350 nm KCl particles mounted on standard Al stubs, hollowed out and solid

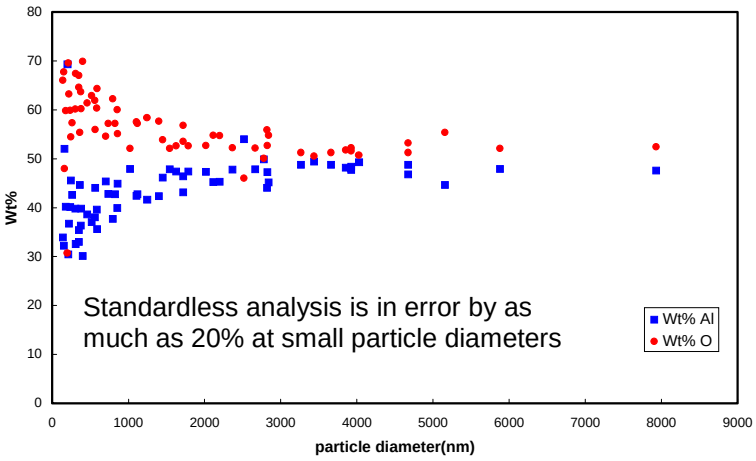

FIG 3. Standard quantitative analysis of Al_2O_3 particles as a function of particle diameter.

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