An automated Scanning Electron Microscopy procedure for nanoparticle characterisation in workplaces

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Motivation

- Cover size range from nano to micro
- Improve particle detection (mixing state organic/inorganic)
- limit time of processing
- minimize user bias
- make available for a wide range of users and **applicable to workplace measurements**

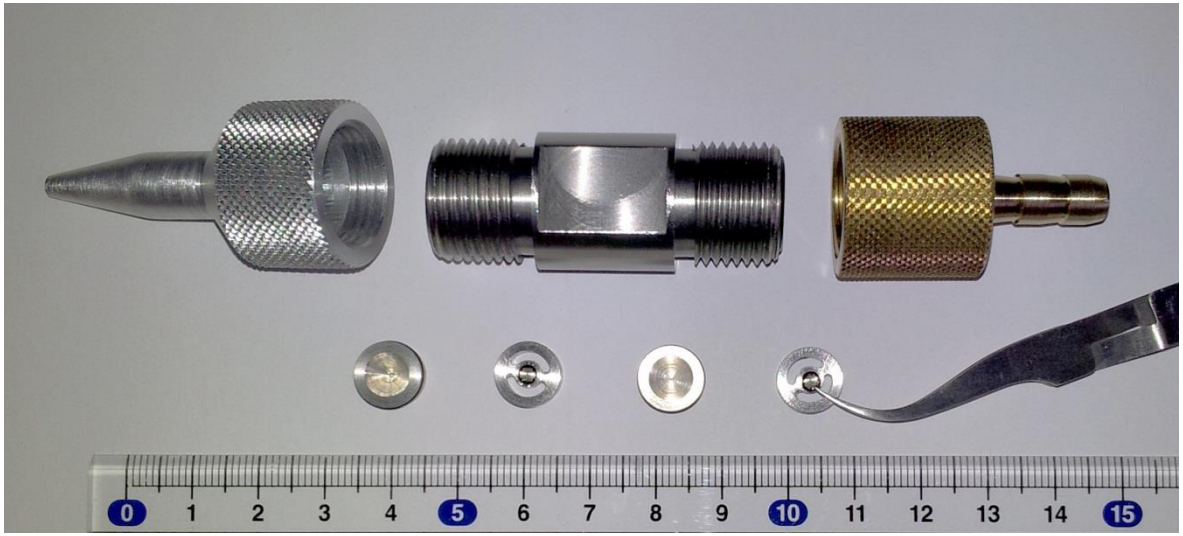
What is new?

The combination:

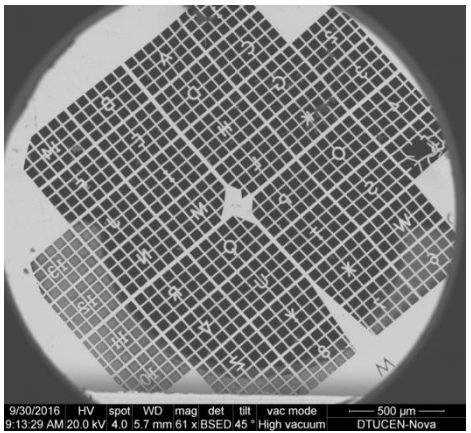
- Three stage impactor designed for EM (Kandler et al. 2007) *adjusted*.
- Automated analysis with a *STEM* setup
- Data processing with codes aiming for *use of data in exposure measurements* and more.

Micro Inertial Impactor sampling

- Short-term sampling of particles from tens of nanometers to micrometers
- 1 to 4 stages with distinct cut-off diameter on Ni-TEM and carbon substrate for single particle analysis in **scanning and transmission electron microscopy (SEM and TEM)**



flow = 0,48 lpm

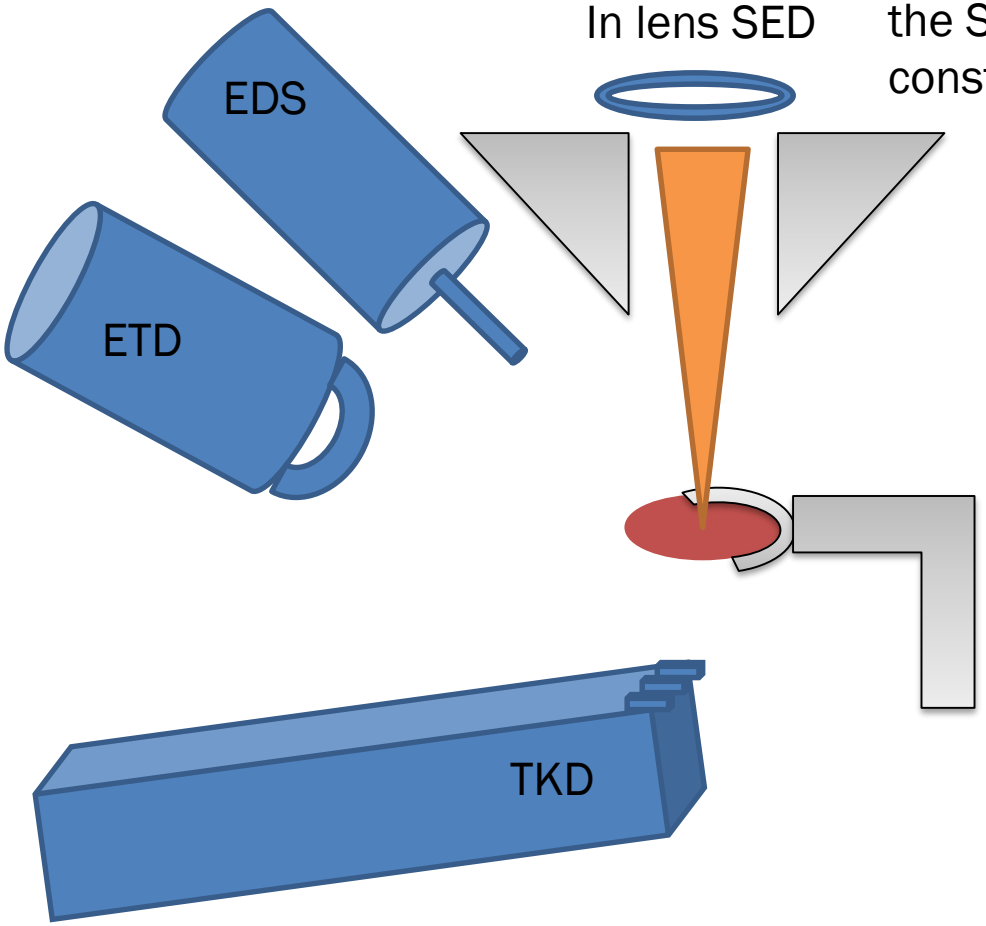


Setting up the impactor

1) Close to the source, supporting ”NF” measurements with *short term* peak emissions
2) *Background*, or in the so called ”FF”, covering the overall composition of aerosol

- Sampling times must be adjusted to concentration levels (in best case size resolved)
- Multiple stages prevent from overloading and insure complete sampling, when D_{aero} is not know.

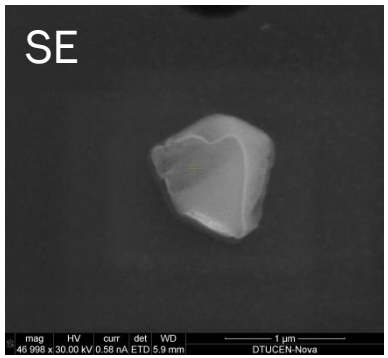
STEM setup

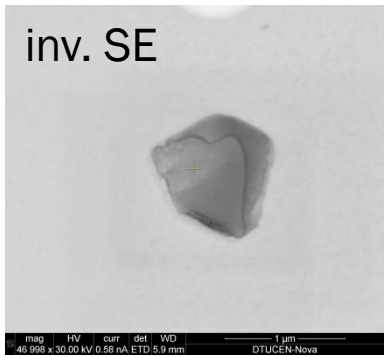


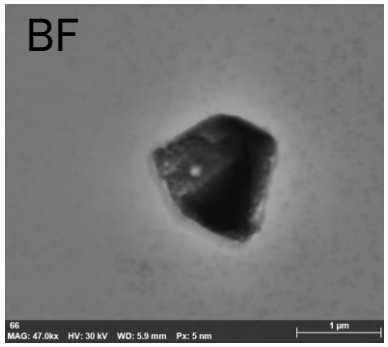
In lens SED

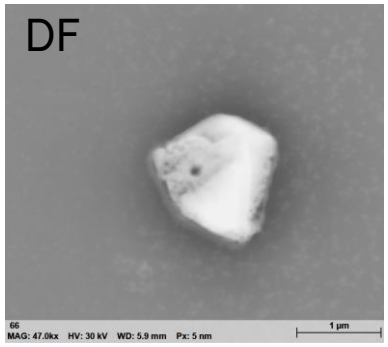
BSE detector can also be used, but the STEM DF exhibits clearer Z constrast

Image output



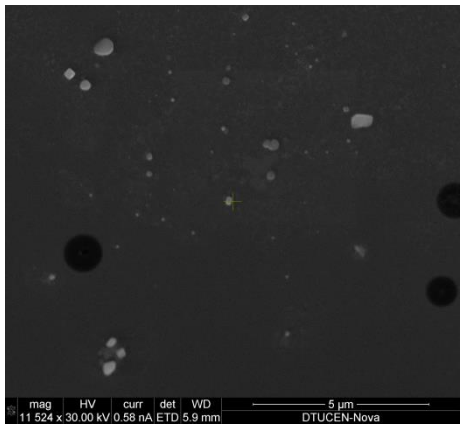


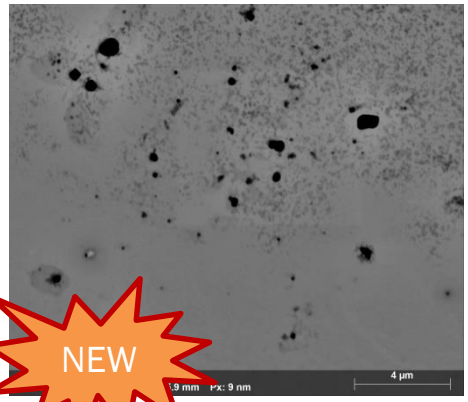




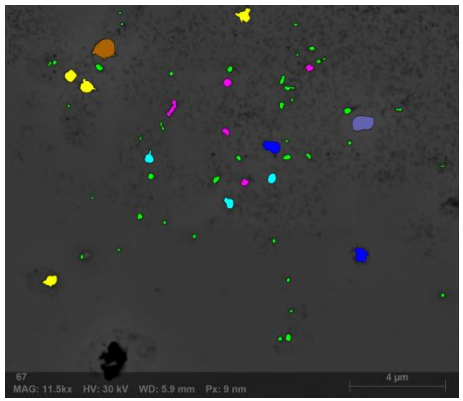
You can use a BSE detector mounted upside down undernaeth your sample as a STEM

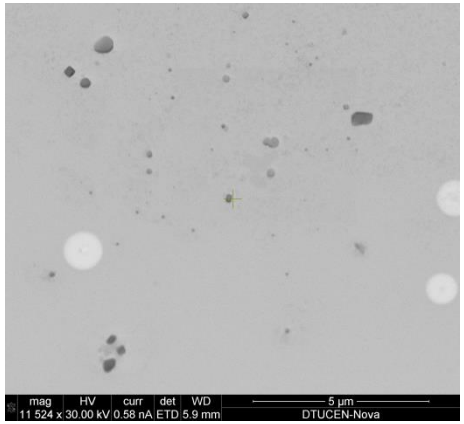
automated particle detection

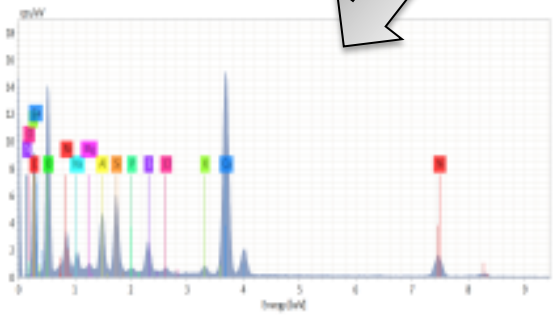




NEW







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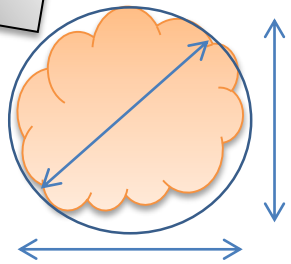
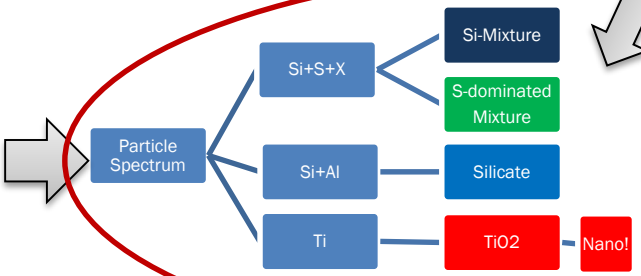
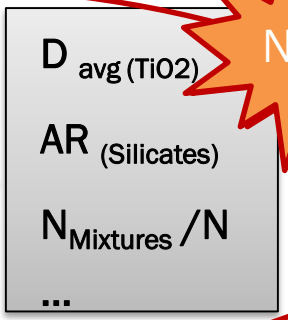


Particle	Size	Si	Mg
P1	XX nm	… w%	… w%
P2	XX nm	… w%	… w%
P3	XX nm	… w%	… w%
P4	XX nm	… w%	… w%



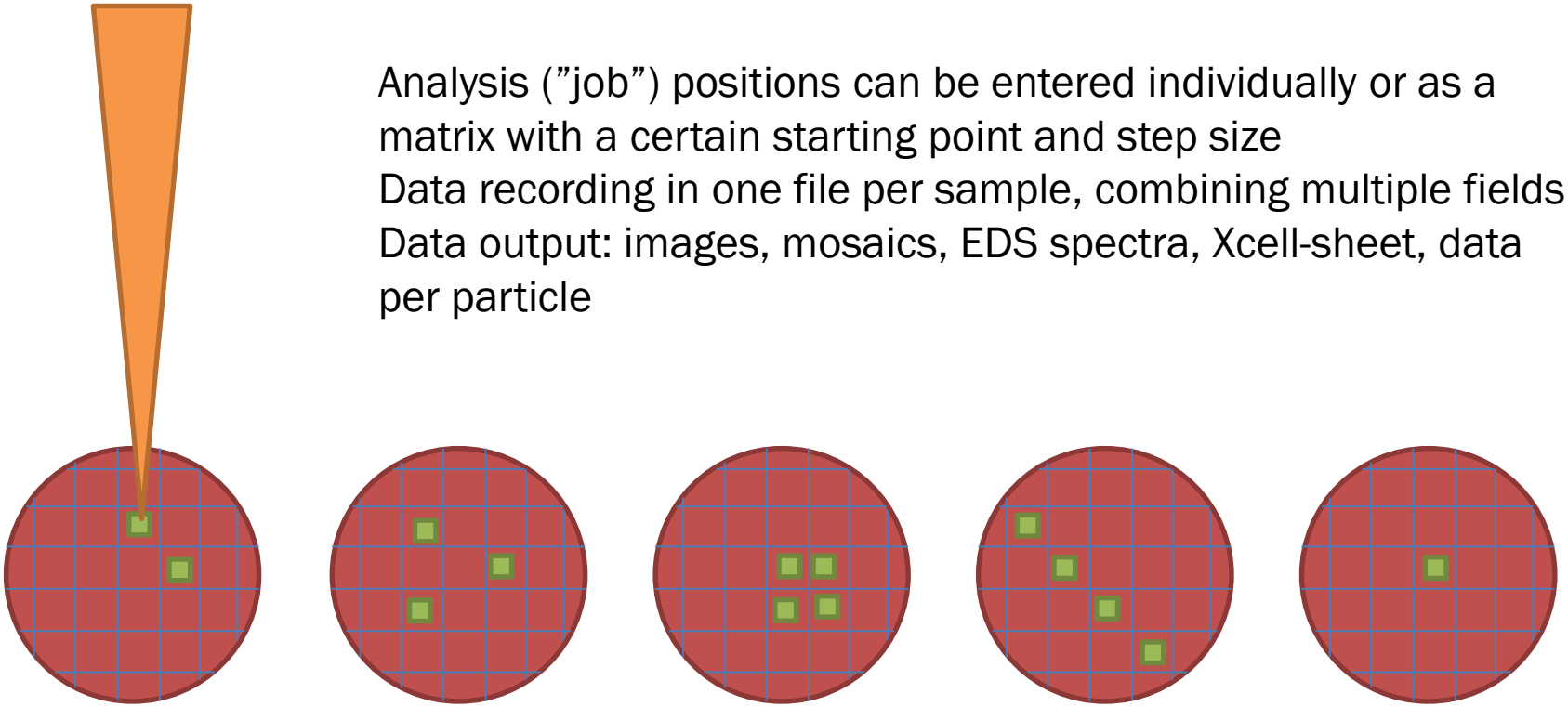


NEW

job and stage setup

Beam freely movable over wide range
Sample should have similar Z-height/working distance
Recording software is reading and controlling stage positions

Analysis (”job”) positions can be entered individually or as a matrix with a certain starting point and step size
Data recording in one file per sample, combining multiple fields
Data output: images, mosaics, EDS spectra, Xcell-sheet, data per particle

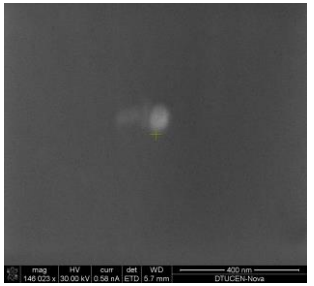


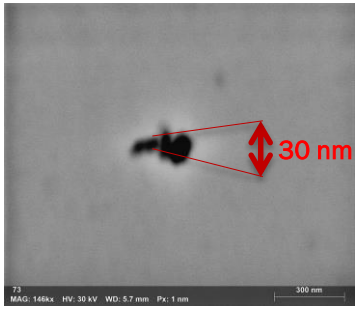
data processing

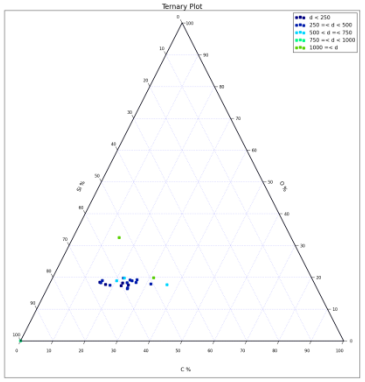
- Xcel file and images can be used for processing
- Our code is written in Python 2.0 (free ware!)
- Classifying upon chemical composition on mineral base.
- Morphology and size parameters used for binning.
- Easily adjustable for statistical correlation.
- Atributes added such as material density per class based on mineralogy and mixing rules.
- Additional parameters as fractal dimension can be calculated from the existing morphology data or an additional image processing code can be applied and combined with the particle table (numbering of particles line by line from right to left and top to bottom)
- Finally, added atributes and additional parameters can be combined, e.g. specific density or aerodynamic size can be calculated.

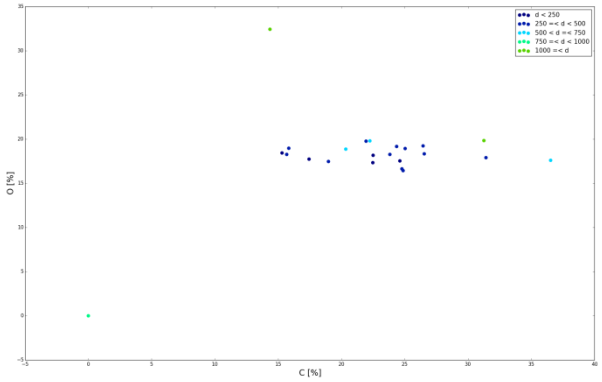
Results

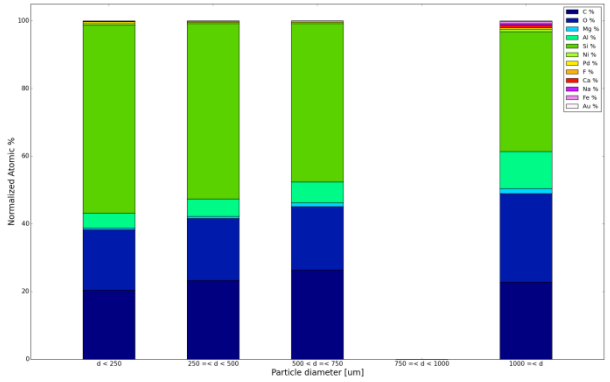
- More level of detail BF compared to SE leads to an at least qualitative description of low contrast material, which could actually mean we detect non-volatile organics.
- Classes and size resolved statistics
- …











Prerequesits

- The limitation of your SEM is the user. We recomend a well-aligned conventional FEG-SEM in combination with a skilled microscopist.
- Retractable TKD detector, mountable BSE detector or STEM detector, where the samples are freely movable in X, Y and Z relative to the detector and beam.
- Free hanging grid holder or clamp, e.g. L-shaped or pretilted holder on tilted stage.
- EDS software with ”particle” or ”feature” option and stage control enabled. EDAX Genesis particles and BRUKER Esprit features were succesfully tested, and provide the right data format. OXFORD Inca feature provides automated detection as well, but was not tested. Other suppliers were not reviewed.
- Multipe channel input into the software is required.

Take home message

- Improved sampling and analysis for workplace measurements
- Nano is In!
- Time for analysis reduced
- Data output improved for exposure modeling

Method will be published and the script will be available fro download!

Thanks and acknowledgements…

Thank you for your attention!

- joint project work with the Danish Center for Nanosafety, WP 1.4 and PhD student Anders B. Bluhme at Technical University of Denmark (DTU), Department for Nanotechnology.
- We acknowledge access to the microscopes at DTU Center for Electron Microscopy. Technical support by Labmanager Adam Fuller contributed to succesfully installing the methods at a FEI NOVA NanoSEM.
- Online support and collaboration by Bruker is gratefully acknowledged.