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Interactive spICPMS data treatment using Nanocount

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spICP-MS: pros and cons

Pros:

- Determines
  - polydisperse sizes
  - particle number concentration
  - dissolved concentrations vs. particulates
- uses an existing machine to calculate size
- It can do small sizes fast ⇔ TEM
- Extremely sensitive for very low number concentrations
- Very little sampel preparation or sample disturbance

Cons:

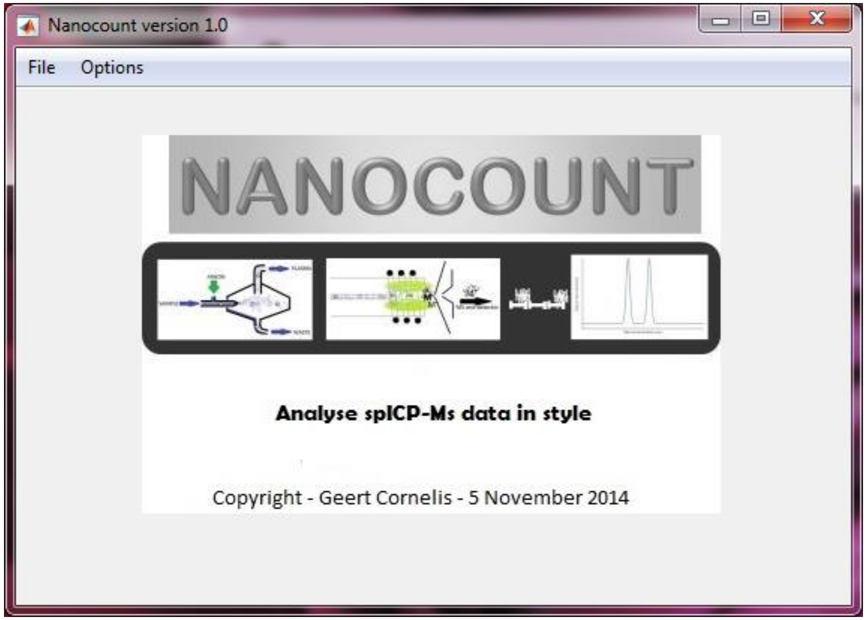
- Assume a spherical shape
- Poor size limits for certain nanoparticles (e.g. SiO_2)
- Works only for inorganic particles and only ”sees” the inorganic part
- Only one element at the time (maybe TOF-spICPMS in the future)
- Method optimization (dilution, dwell time)
- **Data treatment**

Basic data interpretation steps

- Export data from ICP-MS and import in your tool (e.g. excel)
- Obtain calibration curve
- Calculate histograms from raw data
- Determine dissolved/particulate level and remove dissolved data
- Calculate nebulisation efficiency
- Calculate diameters from signal intensities
- Calculate number concentrations from frequencies

Additional data interpretation steps

- Drift correction
- Signal discrimination
- Nebulisation efficiency determination
- Particle size distribution editing



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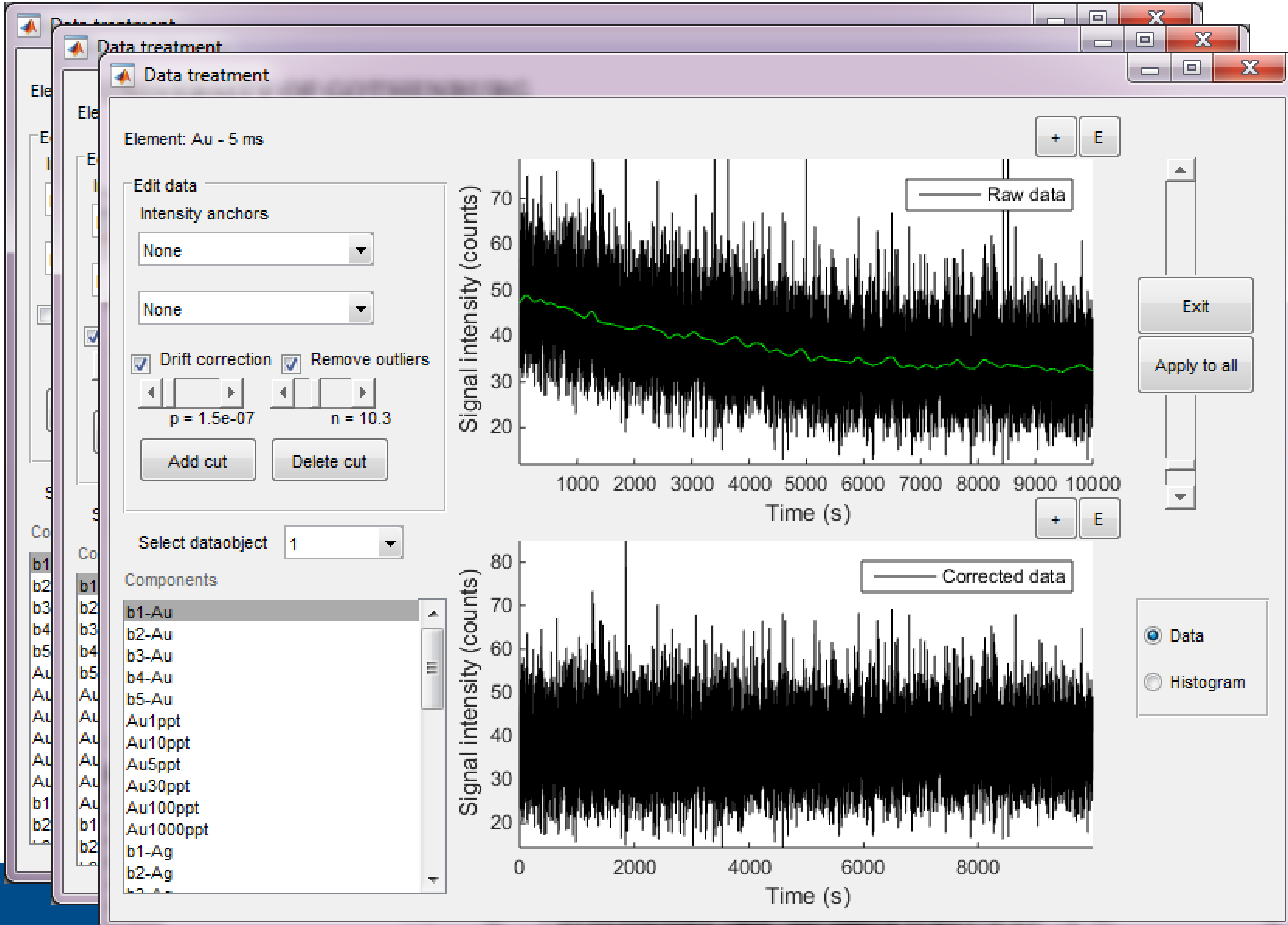
Additional data interpretation



Nebulisation efficiency

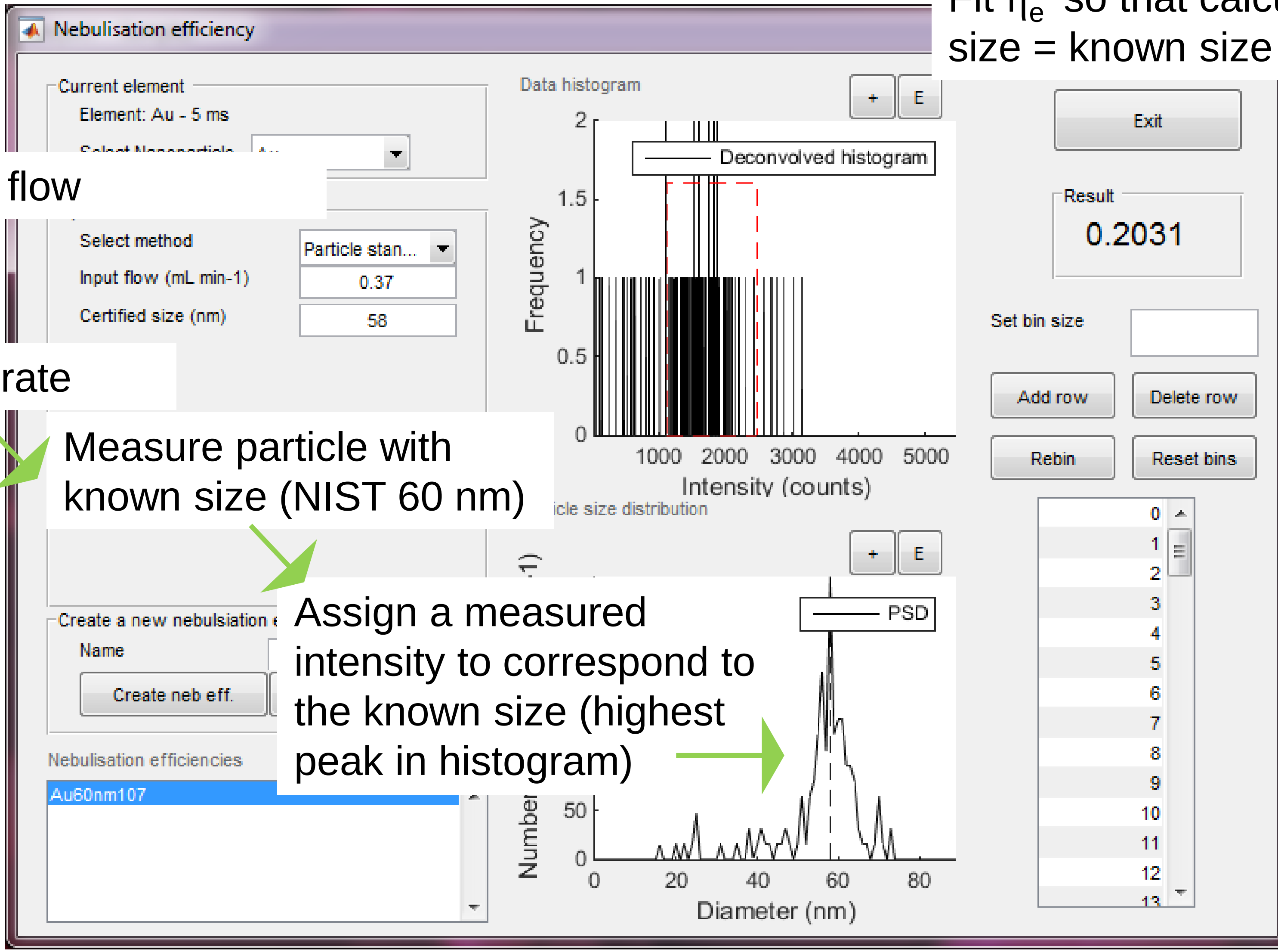
Fit η_e so that calculated size = known size

Measure flow

Calibrate

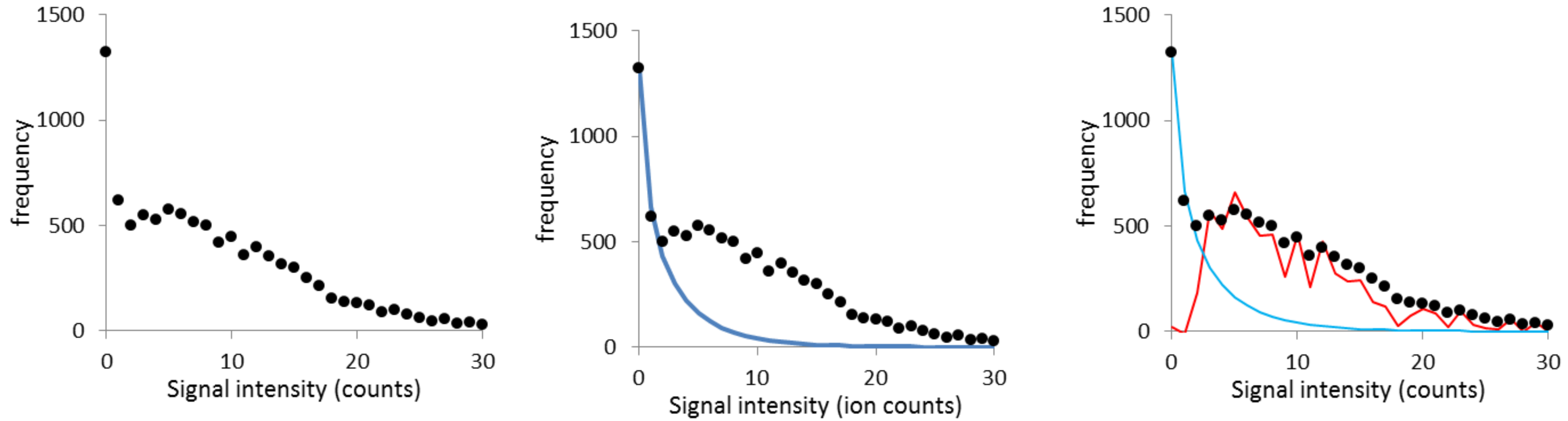
Measure particle with known size (NIST 60 nm)

Assign a measured intensity to correspond to the known size (highest peak in histogram)



Pace, H. E.; Rogers, N. J.; Jarolimek, C.; Coleman, V. A.; Gray, E. P.; Higgins, C. P.; Ranville, J. F., Single Particle Inductively Coupled Plasma-Mass Spectrometry: A Performance Evaluation and Method Comparison in the Determination of Nanoparticle Size. *Environmental Science & Technology* **2012,** *46* (22), 12272-12280.

Signal discrimination: Deconvolution



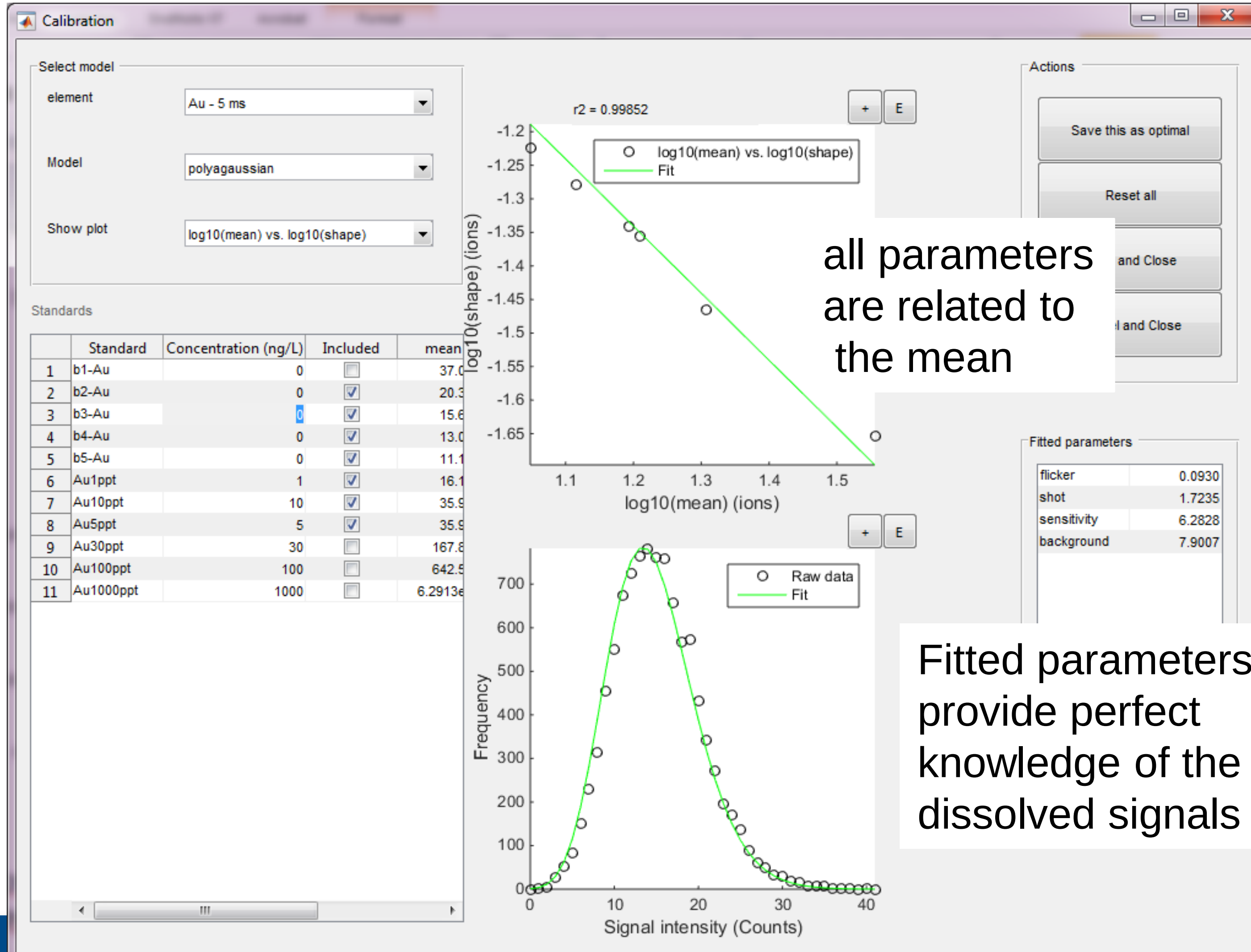
If one has perfect knowledge how dissolved signals look like in histograms they could be subtracted to provide a histogram free of dissolved signals

Cornelis, G.; Hassellov, M., A signal deconvolution method to discriminate smaller nanoparticles in single particle ICP-MS. *Journal of Analytical Atomic Spectrometry* **2014,** *29* (1), 134-144.

Calibration in the deconvolution method

Different models
- Basic
- Normal
- Polyagaussian
- Poissongaussian

Model parameters are fitted to several dissolved standards



all parameters are related to the mean

Fitted parameters provide perfect knowledge of the dissolved signals

Dissolved signal removal

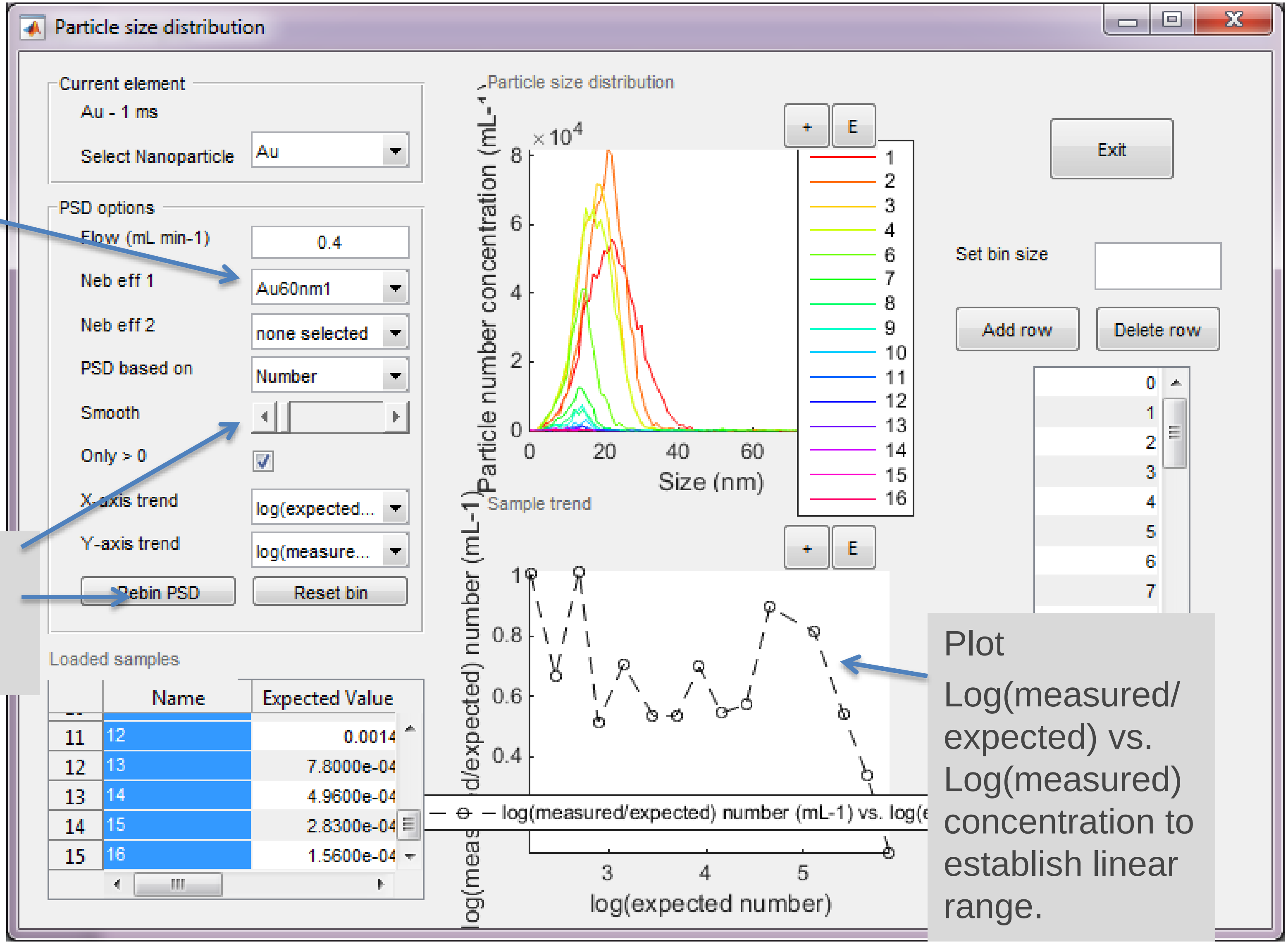
Several methods:

- ”None”
- Outlier analysis
- Deconvolution
- K-means

Choice of number of fitpoints
- manual
- Do a sweep

PSD editing

Use calculated nebulisation efficiency to calculate diameters and number concentrations



Edit PSD by

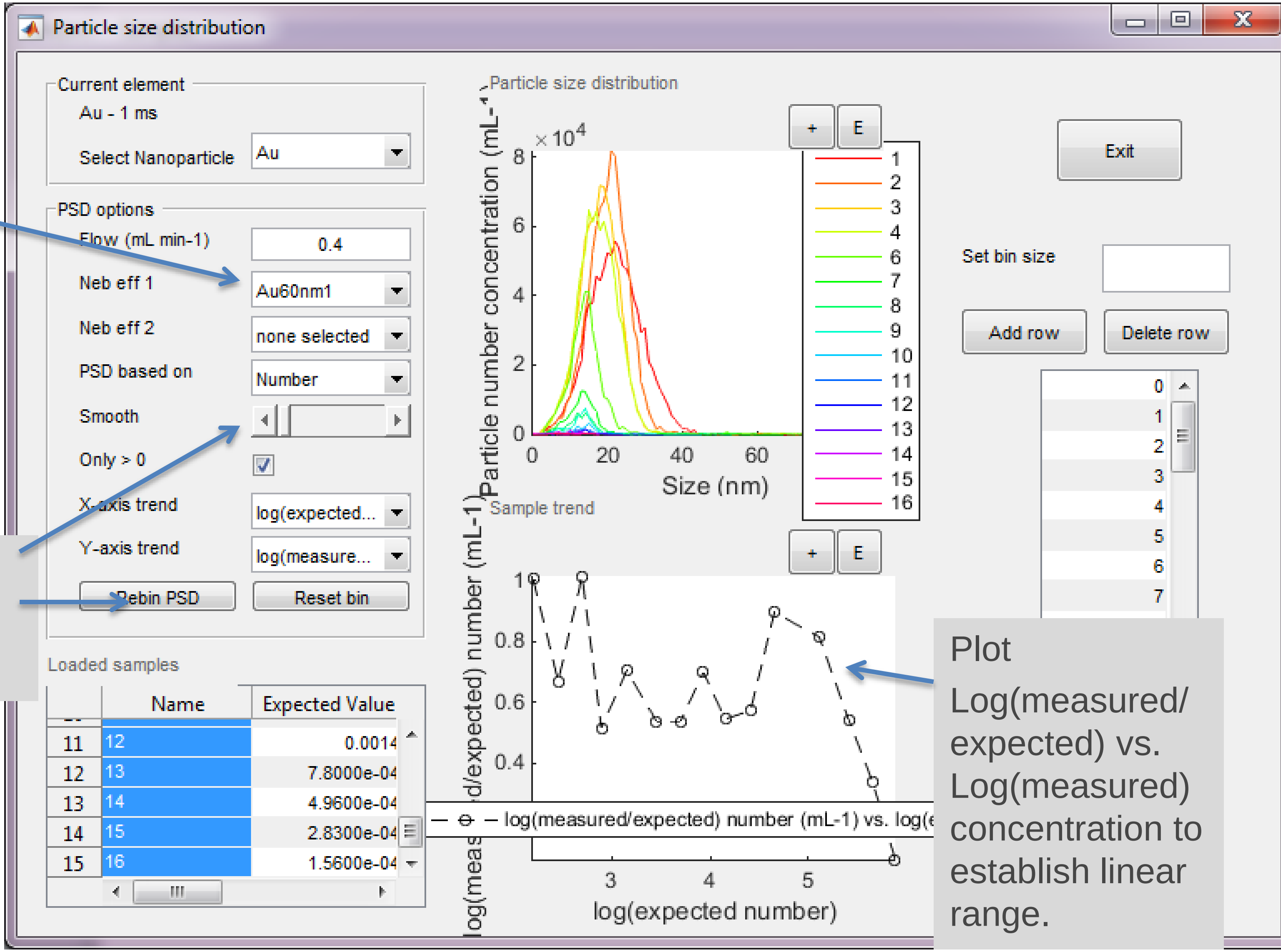
- Smoothing
- Rebinning

Plot Log(measured/ expected) vs. Log(measured) concentration to establish linear range.

PSD calculation

Use calculated nebulisation efficiency to calculate diameters and number concentrations

Edit PSD by
- Smoothing
- Rebinning



Plot Log(measured/ expected) vs. Log(measured) concentration to establish linear range.

Why ?

- spICP-MS is very promising
- Probably the only technique that can
  - Monitor (inorganic) NMs in complex environments
  - Measure realistically low concentrations
  - Quantify number concentrations
  - Hardly disturbes the sample
- ICP-MS is readily available in many labs
- Data treatment theory is available but will be developed further and is impossible to handle in a spreadsheet format

Thank you

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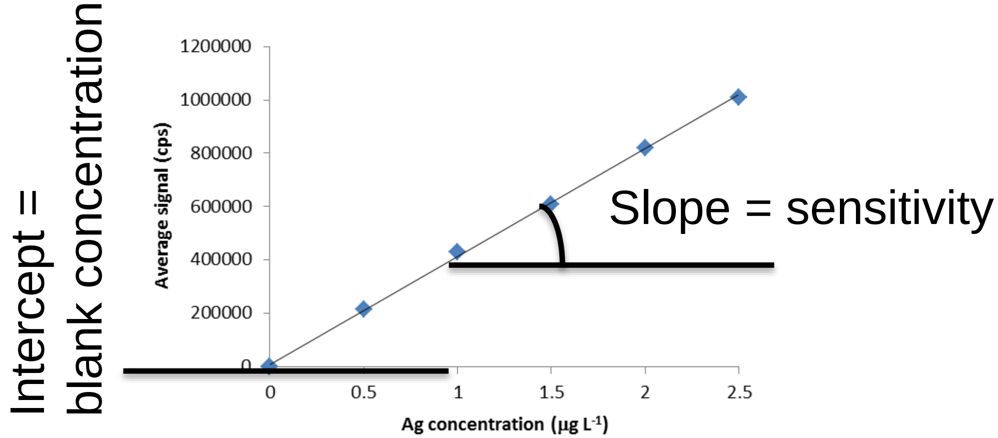
- Calibration curve



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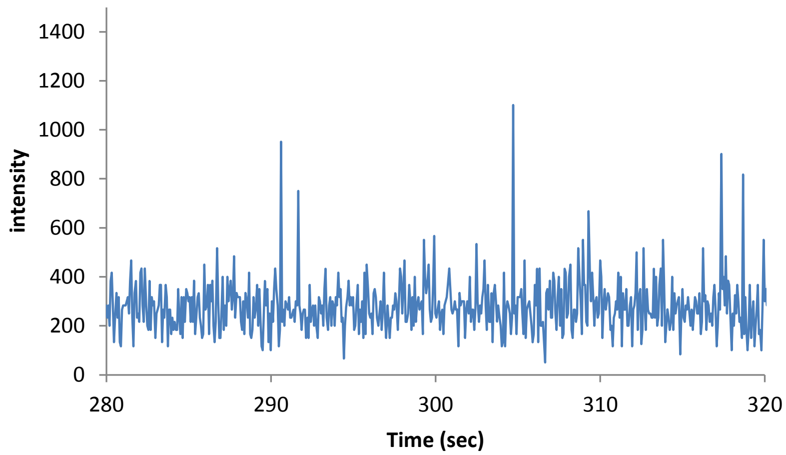
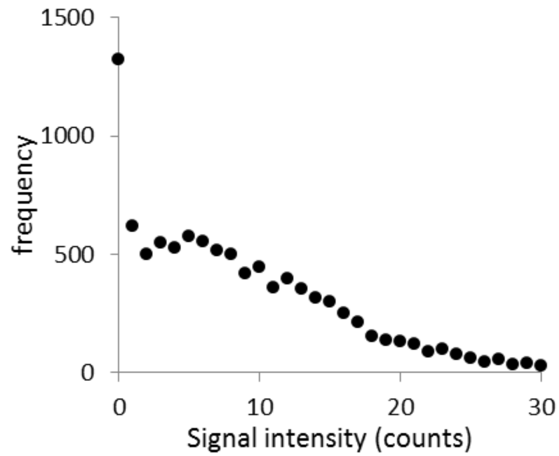
Particle signal



Particle signal histogram



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Basic data interpretation steps

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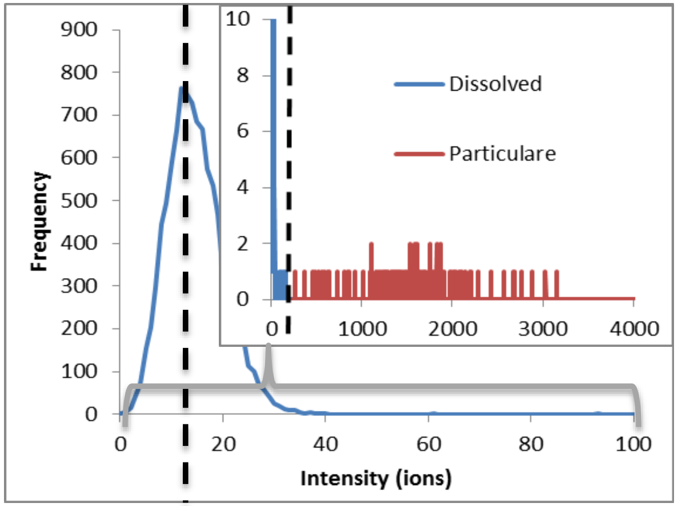
- Determine dissolved/particulate level and remove dissolved data

e.g. 60 nm Au NPs:



Average dissolved intensity

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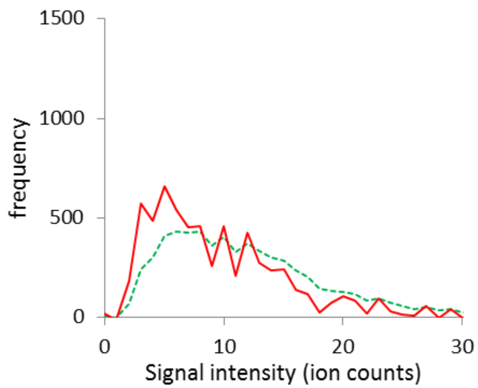
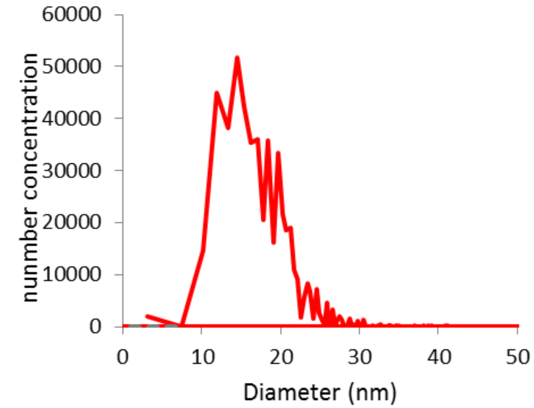
Basic data interpretation steps

Particle signal histogram



$$N_i = \frac{f(P_i)}{\eta_n q\ Dt_d}$$

Particle size distribution calculation



$$d = \sqrt[3]{\frac{(I - I_d - I_{bkg})6\rho q \eta_e M_w}{\pi m t_d}}$$