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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  $\Leftrightarrow$  TEM
- Extremely sensitive for very low number concentrations
- Very little sample preparation or sample disturbance

### Cons:

- Assume a spherical shape
- Poor size limits for certain nanoparticles (e.g.  $\text{SiO}_2$ )
- 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**

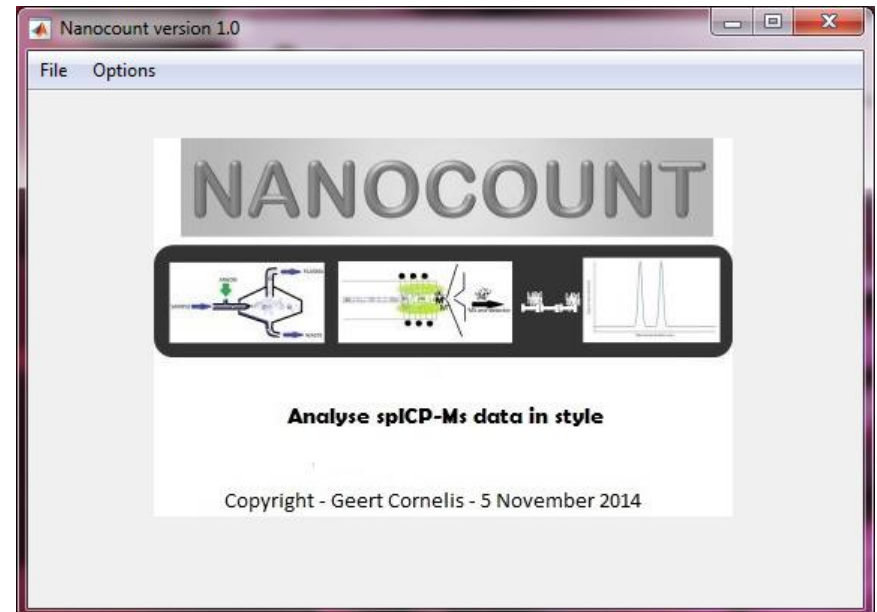


## Basic data interpretation steps

- 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



# Additional data interpretation

The screenshot displays a software window titled "Data treatment" for the element "Au - 5 ms". The interface is divided into several sections:

- Left Panel:** A list of data objects and components. The "Components" list includes: b1-Au, b2-Au, b3-Au, b4-Au, b5-Au, Au1ppt, Au10ppt, Au5ppt, Au30ppt, Au100ppt, Au1000ppt, b1-Ag, and b2-Ag.
- Control Panel:** Includes "Edit data" options, "Intensity anchors" (both set to "None"), and checkboxes for "Drift correction" (checked) and "Remove outliers" (checked). Below these are sliders for "p = 1.5e-07" and "n = 10.3", along with "Add cut" and "Delete cut" buttons.
- Top Graph:** A plot of "Signal intensity (counts)" vs "Time (s)" from 0 to 10000. It shows "Raw data" as a noisy black line and a green trend line representing the drift correction.
- Bottom Graph:** A plot of "Signal intensity (counts)" vs "Time (s)" from 0 to 8000. It shows "Corrected data" as a noisy black line with the drift removed.
- Right Panel:** Contains "Exit" and "Apply to all" buttons, a vertical scrollbar, and radio buttons for "Data" (selected) and "Histogram".



# Nebulisation efficiency

Fit  $\eta_e$  so that calculated size = known size

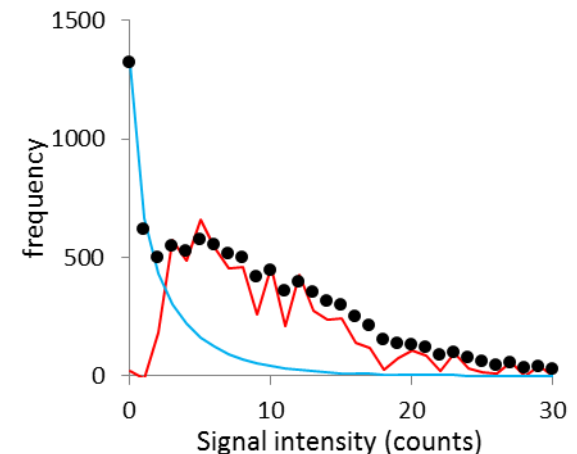
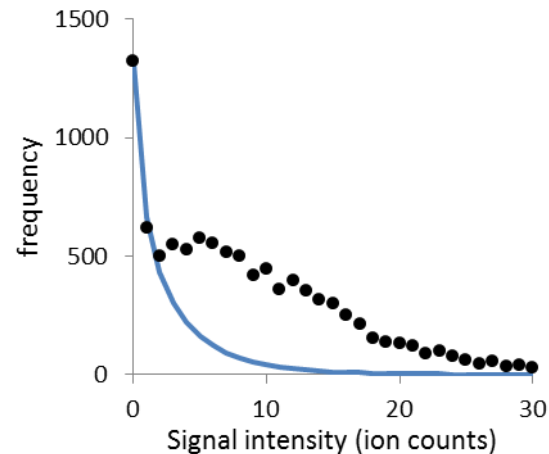
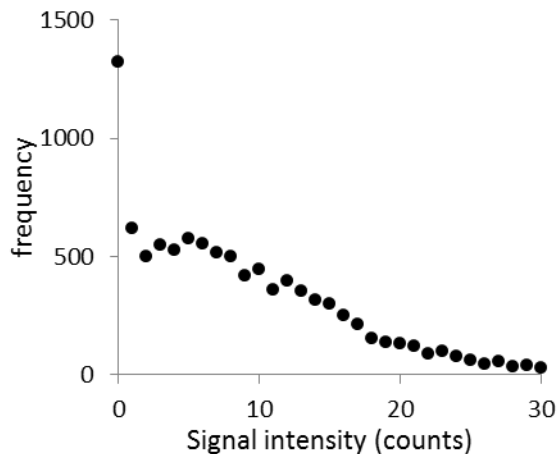
Measure flow

Calibrate

Measure particle with known size (NIST 60 nm)

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

## 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

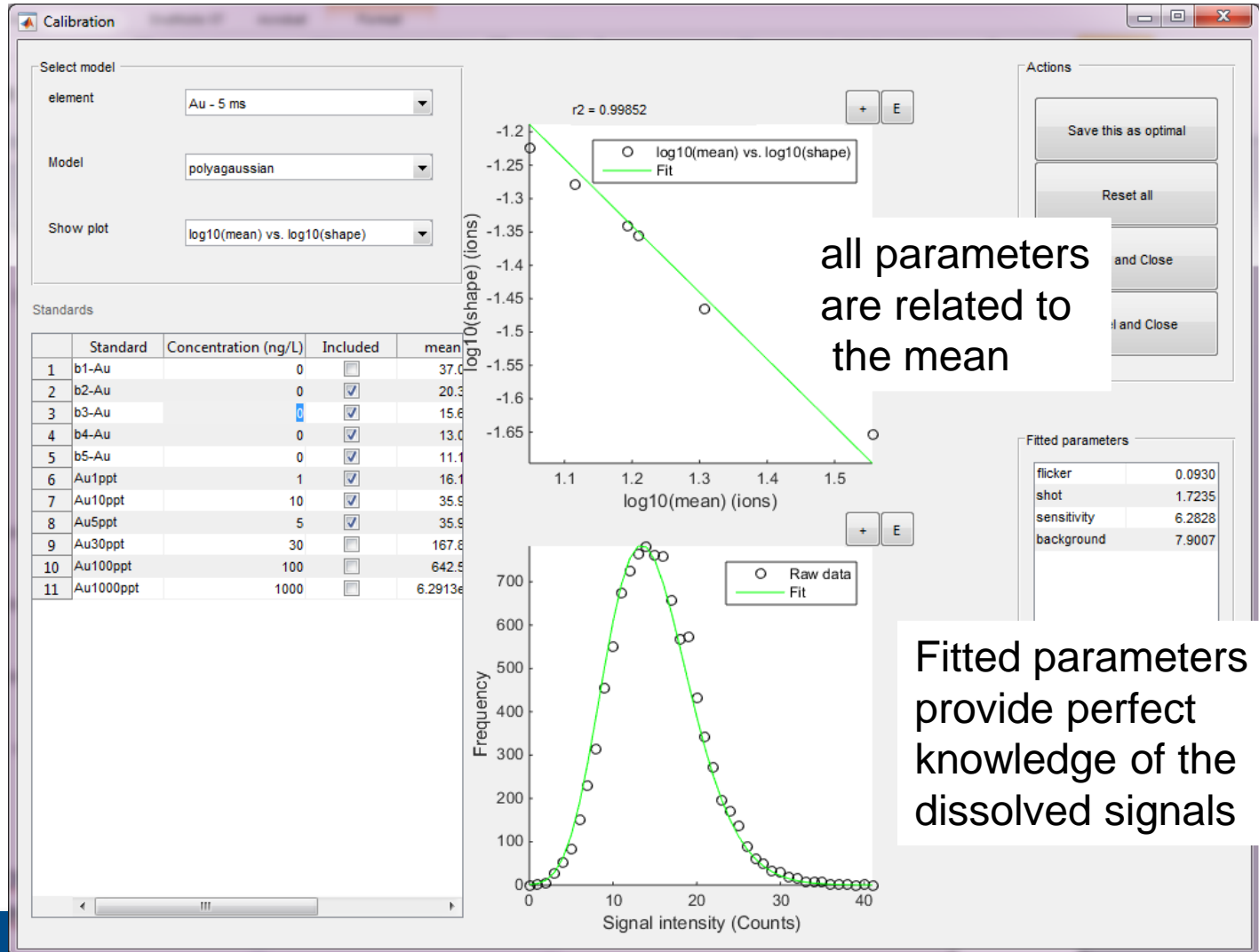
Cornelis, G.; Hasselov, M., A signal deconvolution method to discriminate smaller nanoparticles in single particle ICP-MS. *Journal of Analytical Atomic Spectrometry* **2014**, 29 (1), 134-144.

# 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



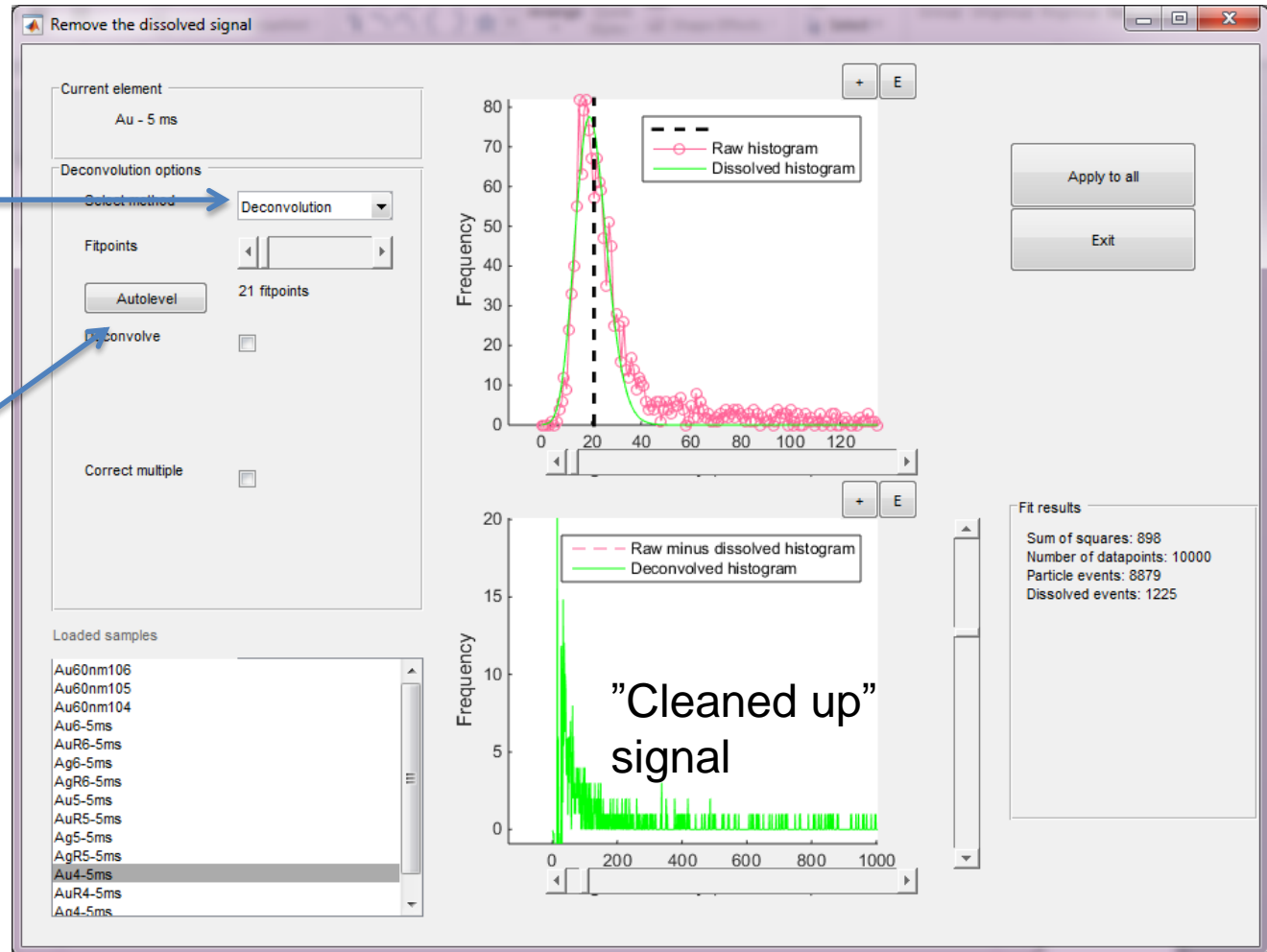
# Dissolved signal removal

Several methods:

- "None"
- Outlier analysis
- Deconvolution
- K-means

Choice of number of fitpoints

- manual
- Do a sweep



# PSD editing

Current element: Au - 1 ms  
Select Nanoparticle: Au

PSD options  
Flow (mL min<sup>-1</sup>): 0.4  
Neb eff 1: Au60nm1  
Neb eff 2: none selected  
PSD based on: Number  
Smooth: [slider]  
Only > 0:   
X-axis trend: log(expected...  
Y-axis trend: log(measure...  
[Rebin PSD] [Reset bin]

Loaded samples

Name	Expected Value
11 12	0.0014
12 13	7.8000e-04
13 14	4.9600e-04
14 15	2.8300e-04
15 16	1.5600e-04

Particle size distribution

Particle number concentration (mL<sup>-1</sup>) × 10<sup>4</sup> vs Size (nm)

Sample trend

log(measured/expected) number (mL<sup>-1</sup>) vs. log(epsilon)

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

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

Particle size distribution

Current element: Au - 1 ms  
 Select Nanoparticle: Au

PSD options  
 Flow (mL min<sup>-1</sup>): 0.4  
 Neb eff 1: Au60nm1  
 Neb eff 2: none selected  
 PSD based on: Number  
 Smooth: [slider]  
 Only > 0:   
 X-axis trend: log(expected...)  
 Y-axis trend: log(measure...)

Buttons: Rebin PSD, Reset bin

Loaded samples

Name	Expected Value
11 12	0.0014
12 13	7.8000e-04
13 14	4.9600e-04
14 15	2.8300e-04
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Particle size distribution graph: Particle number concentration (mL<sup>-1</sup>) × 10<sup>4</sup> vs Size (nm). Legend: 1-16.

Sample trend graph: d/expected number (mL<sup>-1</sup>) vs log(expected number). Legend: log(measured/expected) number (mL<sup>-1</sup>) vs. log(ε).

Buttons: Exit, Add row, Delete row

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.

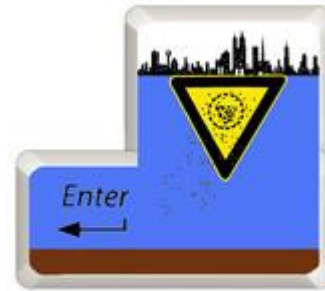


## Why ?

- 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 disturbs 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



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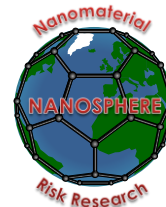


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Thank you

Contact:

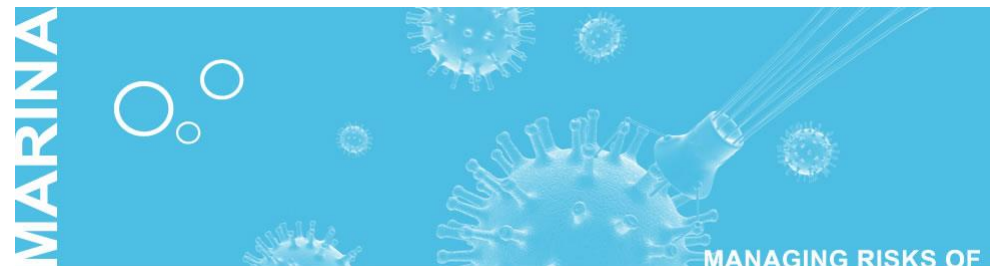
[Geert.Cornelis@chem.gu.se](mailto:Geert.Cornelis@chem.gu.se)



GUIDE *nano* ▶▶▶▶▶



Vetenskapsrådet

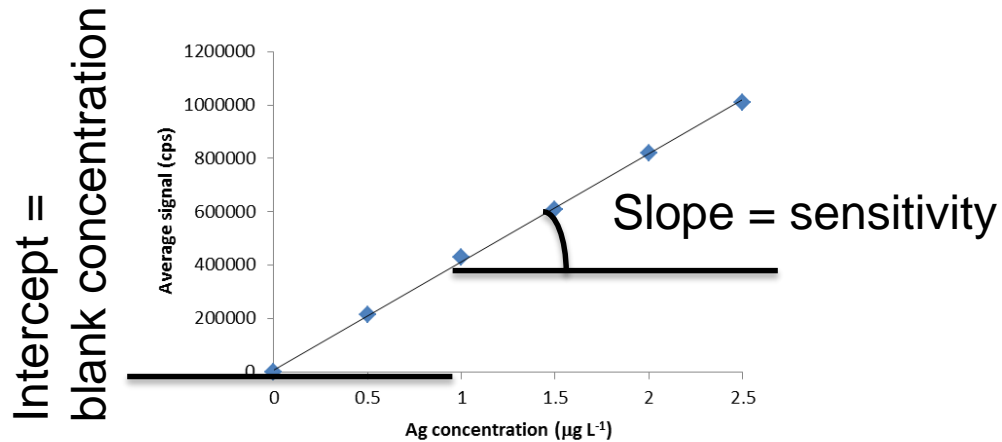


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MANAGING RISKS OF  
NANOMATERIALS

## Basic data interpretation steps

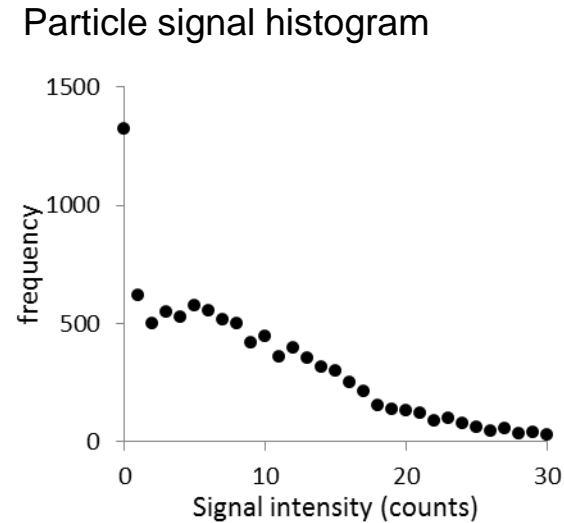
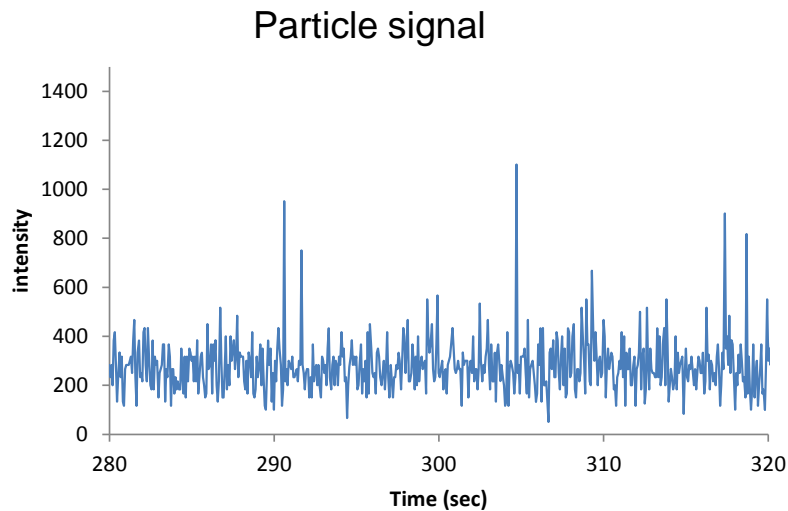
- Export data from ICP-MS and import in your tool (e.g. excel)
- Calibration curve





## Basic data interpretation steps

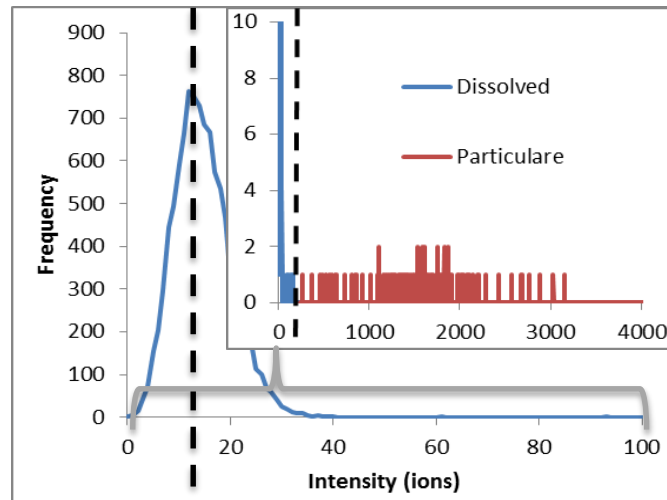
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- Obtain calibration curve
- Calculate histograms from raw data



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- Determine dissolved/particulate level and remove dissolved data

e.g. 60 nm Au NPs:



Average dissolved intensity

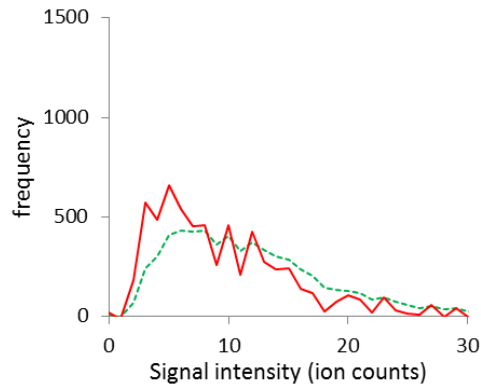


## Basic data interpretation steps

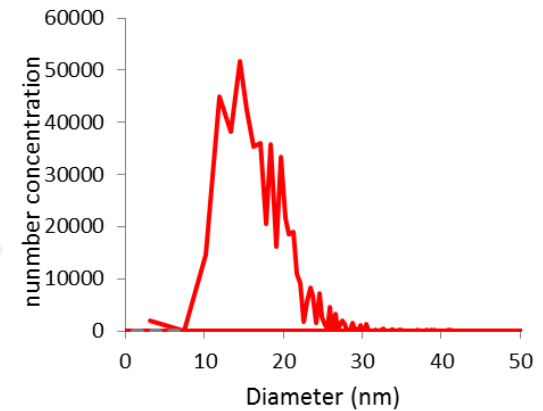
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## Basic data interpretation steps

Particle signal histogram



Particle size distribution calculation



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



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

