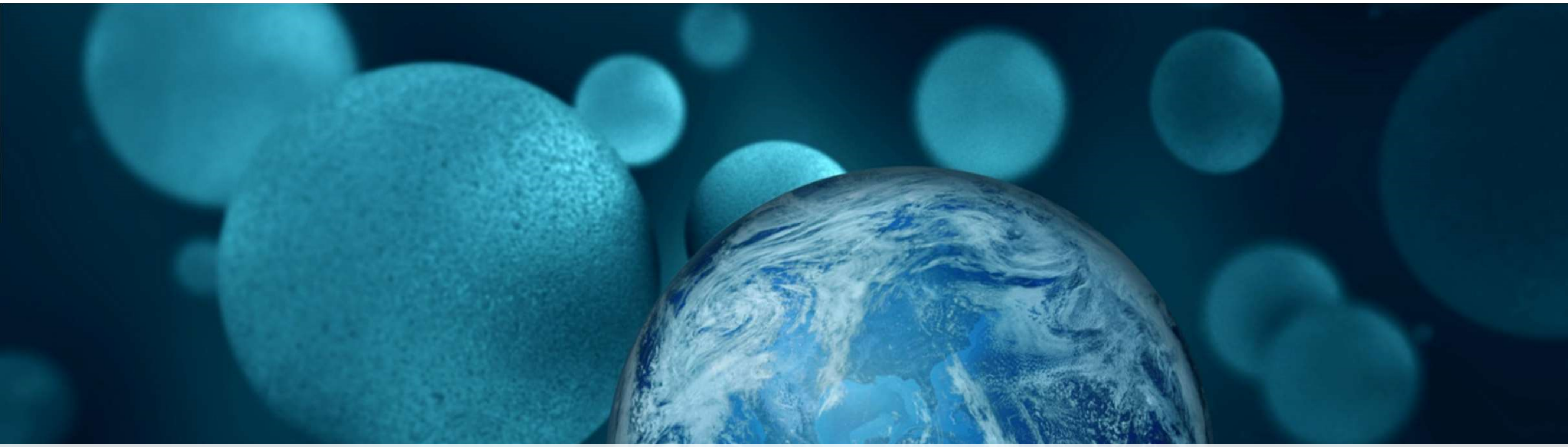

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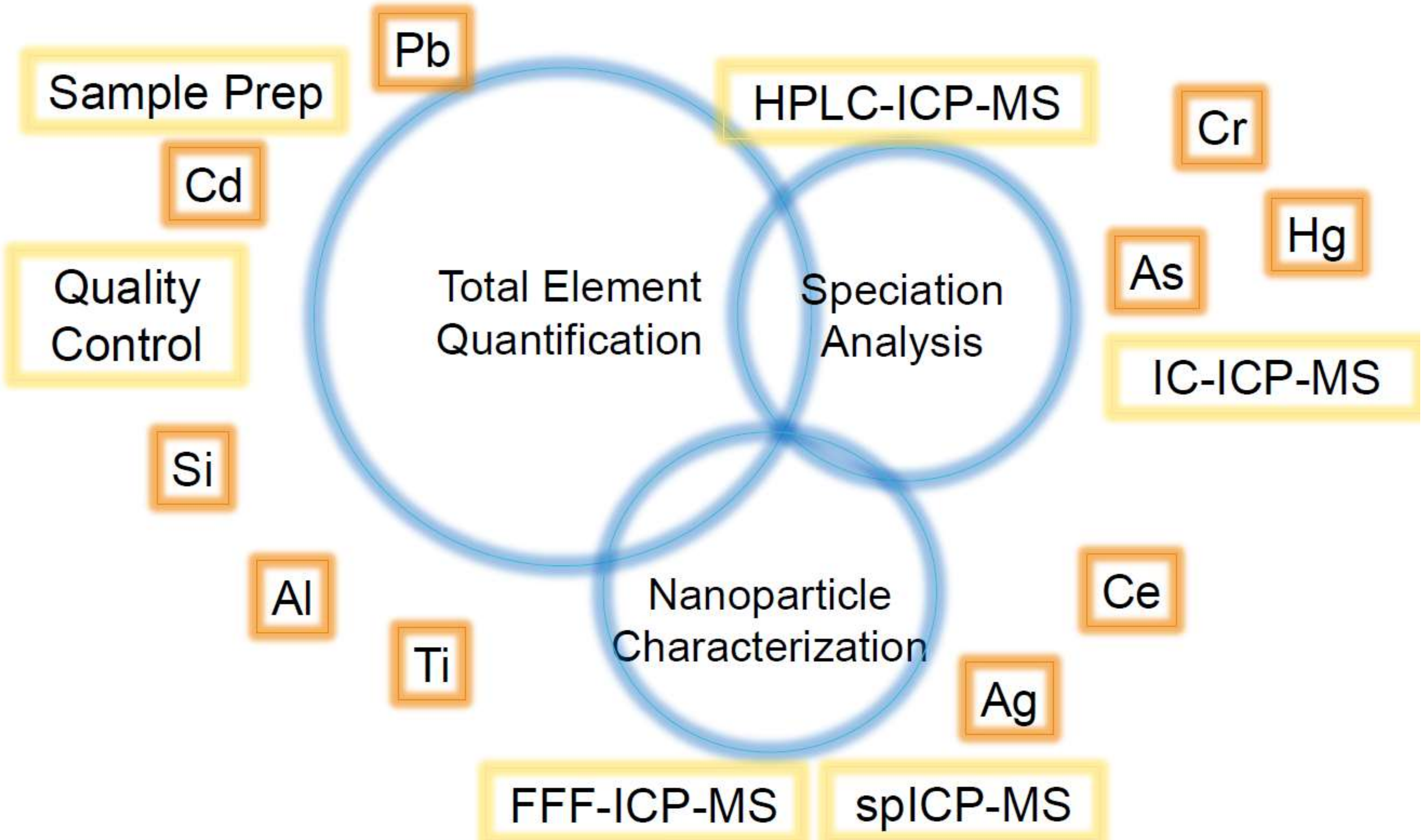
Nanoparticle Analysis: An Upcoming Challenge for Environmental Analysis

John Schmelzel, Application Scientist
Thermo Fisher Scientific

Nanoparticle Analysis – A Task for a Modern ICP-MS Lab



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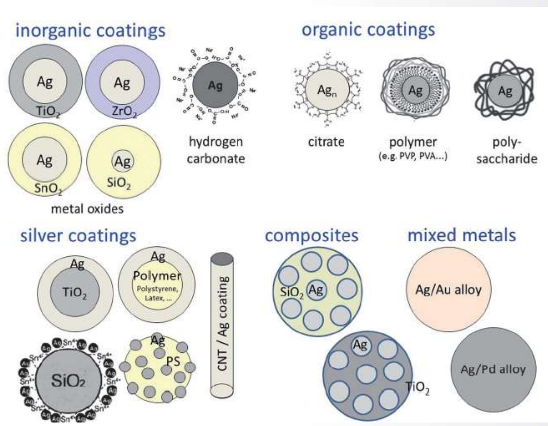
Nanoparticle Analysis – Single Particle ICP-MS

- Definition of "Nanomaterial" – 2011/696/EU Commission Recommendation
  - Particles measuring 1-100 nm, where >50% of the particles in the sample are in this size range
- What does this mean for your analysis requirements?
  - Rapid Analysis
    - Thousands of particles can be present in samples, all of which pass through the instrument in 1-2 minutes
    - Rapid analysis (short dwell times) needed to ensure all particles are measured
  - High Sensitivity
    - High sensitivity and superior detection limits needed to detect low particle concentrations and single particle events
  - Robustness
    - Rugged technique (such as an ICP-MS) is required to reduce the sample manipulation before analysis

spICP-MS: An appealing alternative to existing techniques!

Nanoparticles

- Natural or engineered nanoparticles (ENPs)
- Exceptional properties - high surface to mass ratio
- Added in Foods, packaging, hygiene products, clothes etc...
- Widespread industrial uses



A. Ulrich and co. *JAAS* 2012, **27**, 1120

- Publication: New Scientist, 14 Aug 2010

Environmental impact clouds benefits of bug-killing silver

ANTIBACTERIAL nanoparticles may have more of an impact on the environment than we thought, including potentially raising levels of greenhouse gases.

Silver nanoparticles are used as an antibacterial agent in a wide range of products, from odour-free socks to wound-healing bandages (see diagram, below). They can find their way into waste water, and have been shown to reduce the activity of bacteria used to remove ammonia when the water is treated.

So far most of the research on the environmental impact of nanoparticles has been carried out on single nanoparticles get into the real environment," says Colman. "These particles are developed with the express purpose of killing things."

Two months on, the microbial population in the outdoor tub containing silver had significantly declined relative to the lab sample measured after one week. What's more, the activity of the enzymes they produce to break down organic matter was 34 per cent lower in the tub that had been dosed with nanoparticles than in the tub to which only sludge had been added.

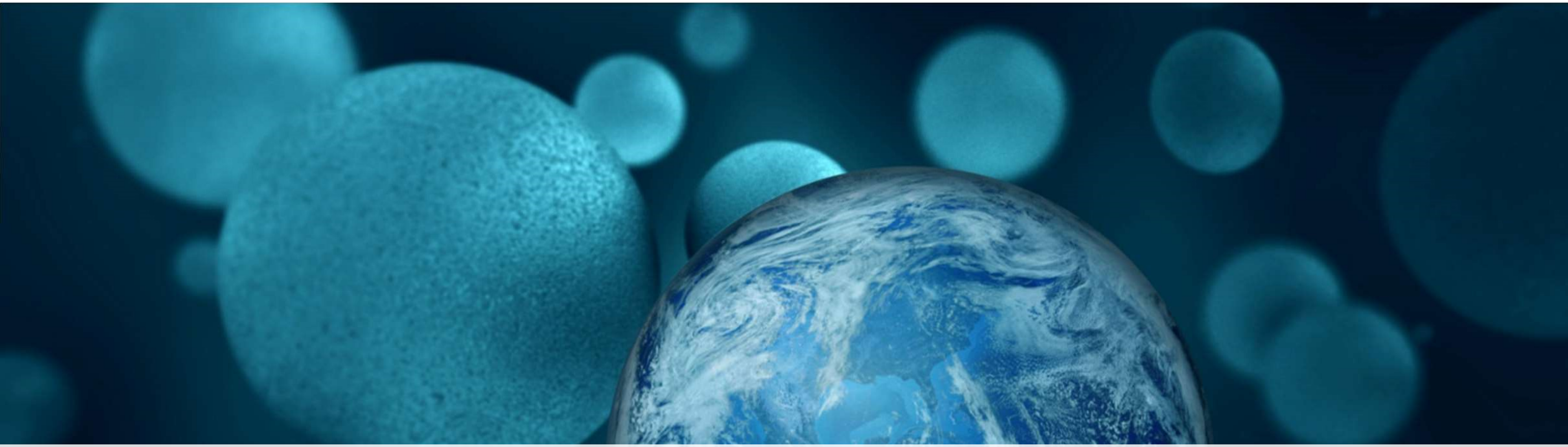
Given that the outdoor tub containing nanoparticles had a much

Potential environmental effects are not clear!

The Nanodefine EU Project

- Scientific project financed by the European Comission with the objective to develop analytical tools to verify “Nano-content“ in different sample types (consumer products, environmental etc.)
- Goal: development of methods that reliably identify, characterize and measure nanomaterials (NM)
  - Close international cooperation and networking between academia, concerned industries and standardization bodies
- Thermo Fisher Scientific is one of the 28 partners
  - The **ONLY** ICP-MS manufacturer participant
- Deliverable from Thermo Fisher Scientific:
  - sp-ICP-MS software development (npQuant)

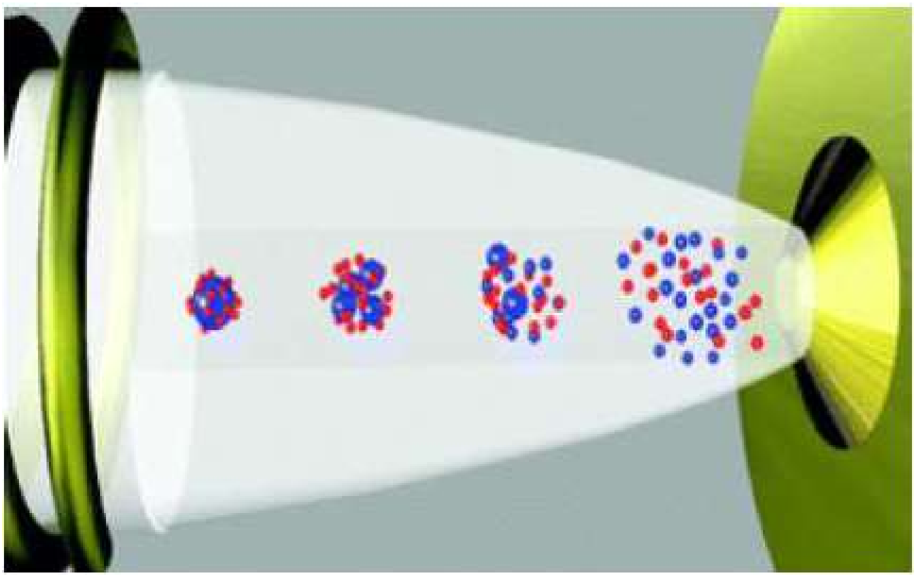
NanoDefine
www.nanodefine.eu

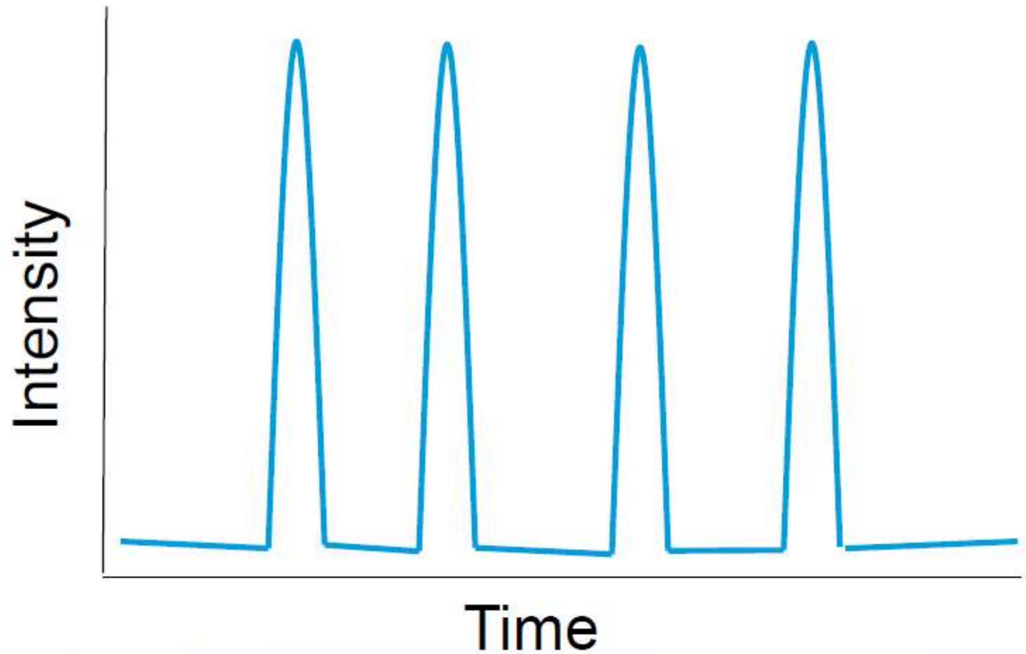


Nanoparticle Characterization via Single Particle Event Analysis

Single Particle ICP-MS for Nanoparticle Characterization

- Single nanoparticles produce ion plumes in the plasma
- These single particle events (SPEs) last ~300 µs each

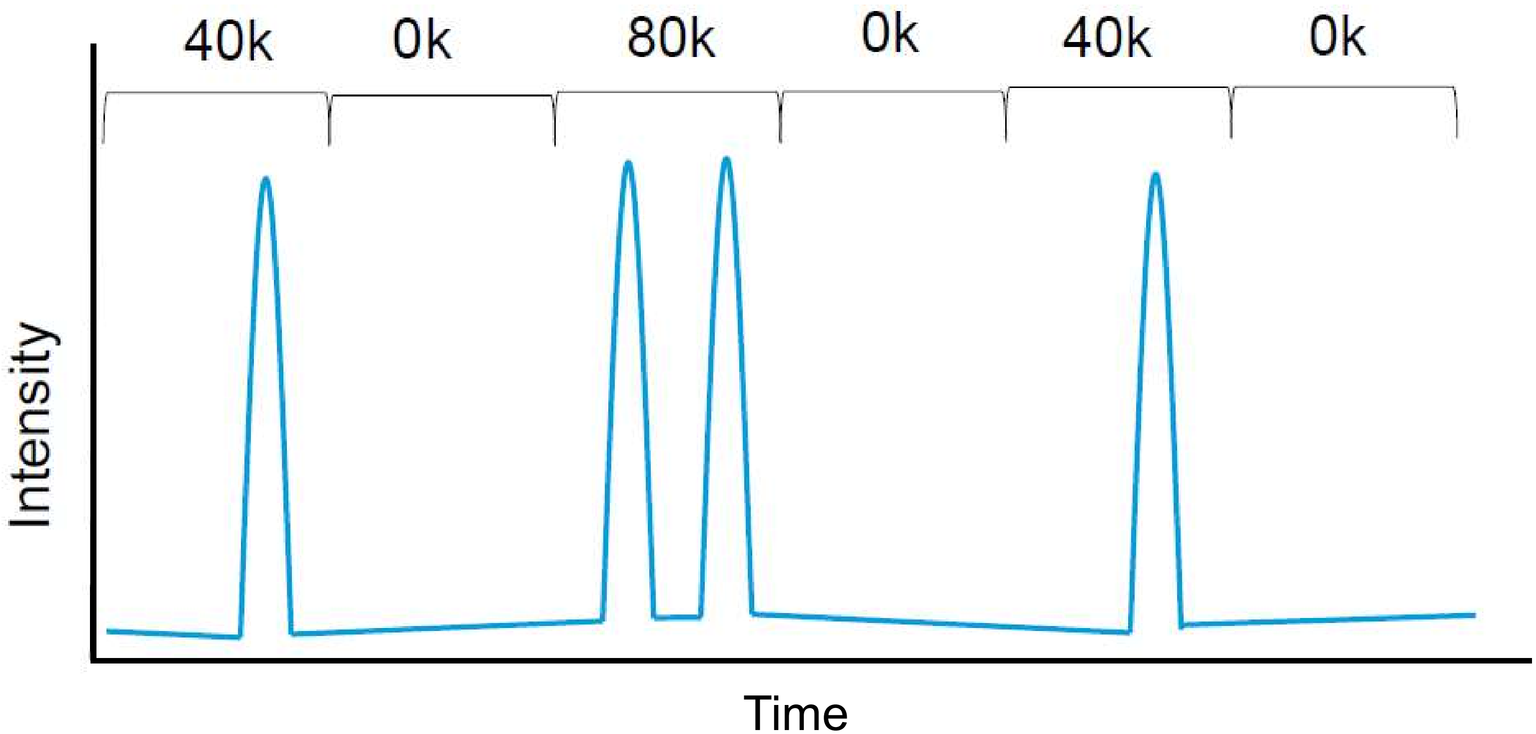




Single Particle ICP-MS for Nanoparticle Characterization

Particle Size α Signal Intensity

- Fast scanning of single isotope
  - Scan envelopes whole SPE (3 ms duration)



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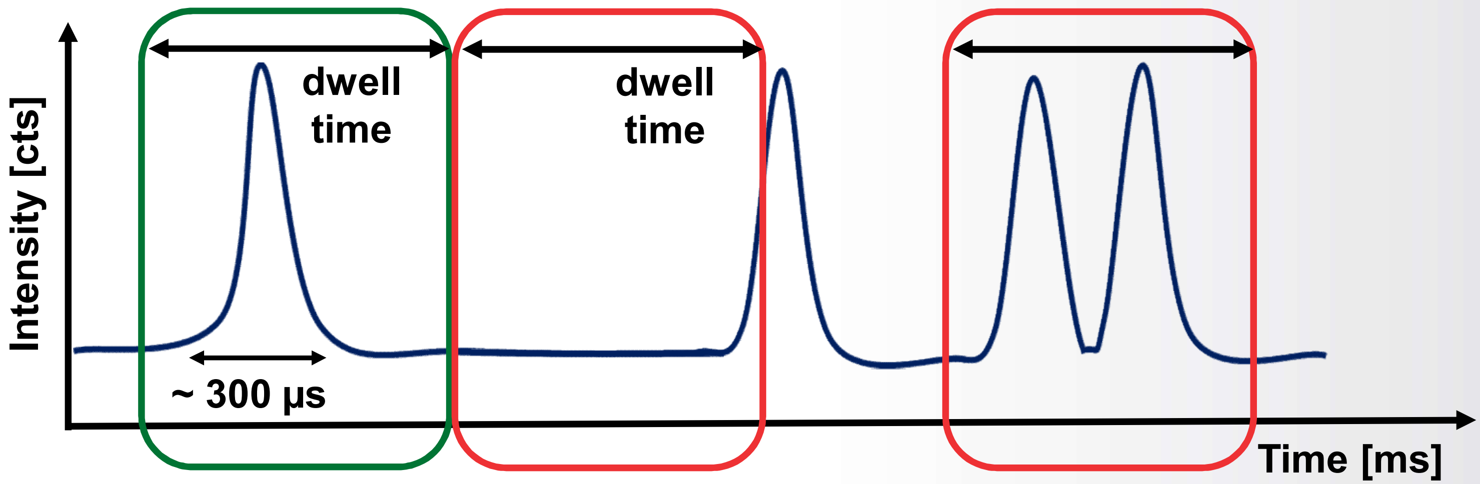
Single Particle ICP-MS for Nanoparticle Characterization

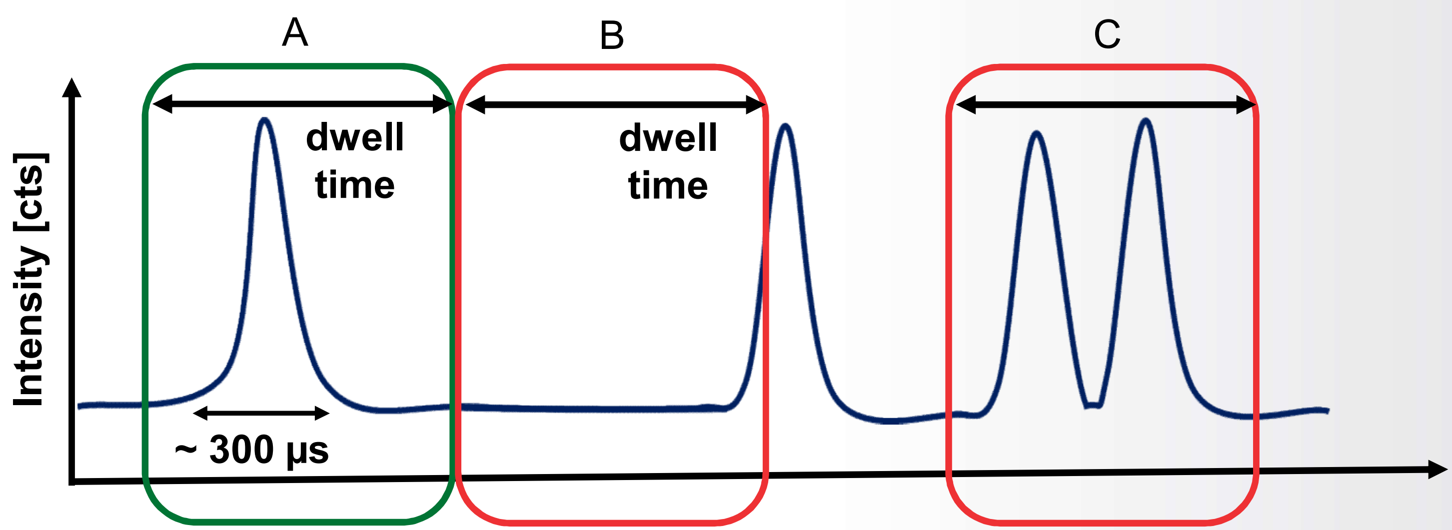
- For the discrimination of various particles short dwell times (~ 3-10 ms) are applied
- Chosen dwell time is a critical parameter in spICP-MS



Artifacts During Data Collection in spICP-MS

- Split Particle Events (A,B): A nanoparticle signal is observed in two adjacent measurement slots. The extent of split particle events depends on the nanoparticle pulse duration and the applied dwell time, and can be reduced by using longer dwell times.

- Double or Multiple Particle Events (C): Two or more particles are observed in one measurement slot, leading to an overestimation of the particle size. The occurrence of such events can be estimated using Poisson statistics and can be reduced by sample dilution.



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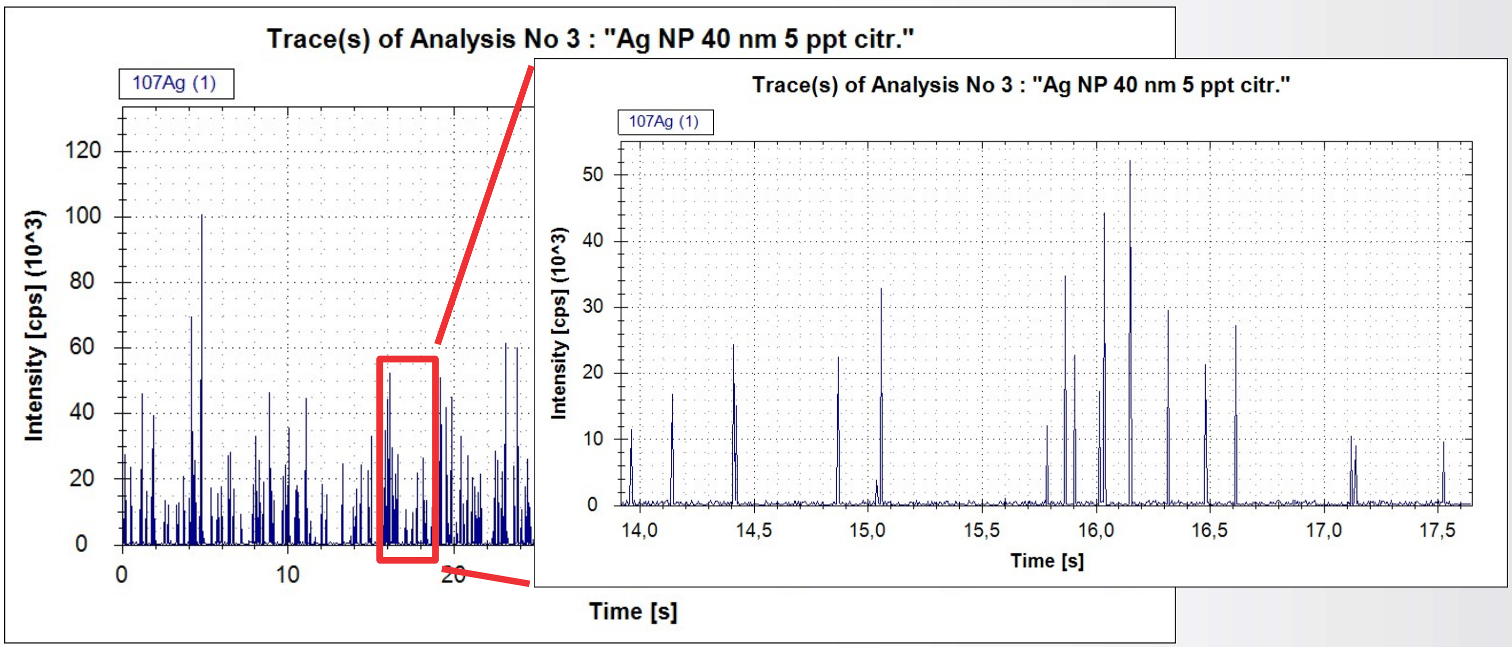
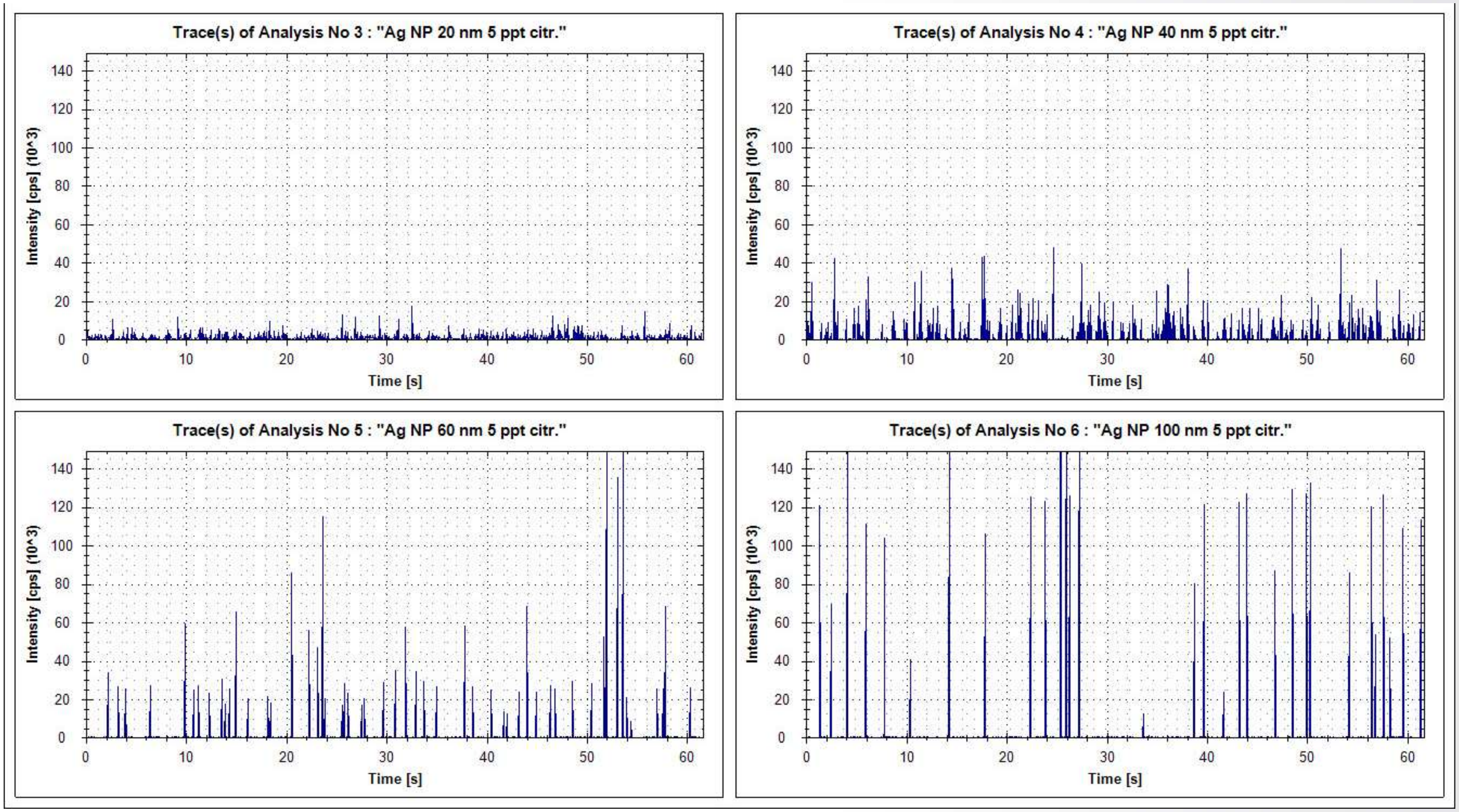
Characterization of Ag nanoparticles

- Background signal ~ concentration of dissolved species
- Number of events ~ particle number concentration
- Signal intensity of events ~ particle mass



Single Particle ICP-MS for Nanoparticle Characterization

- Nanoparticles of different size at the same mass concentration of Ag (5 ppt)



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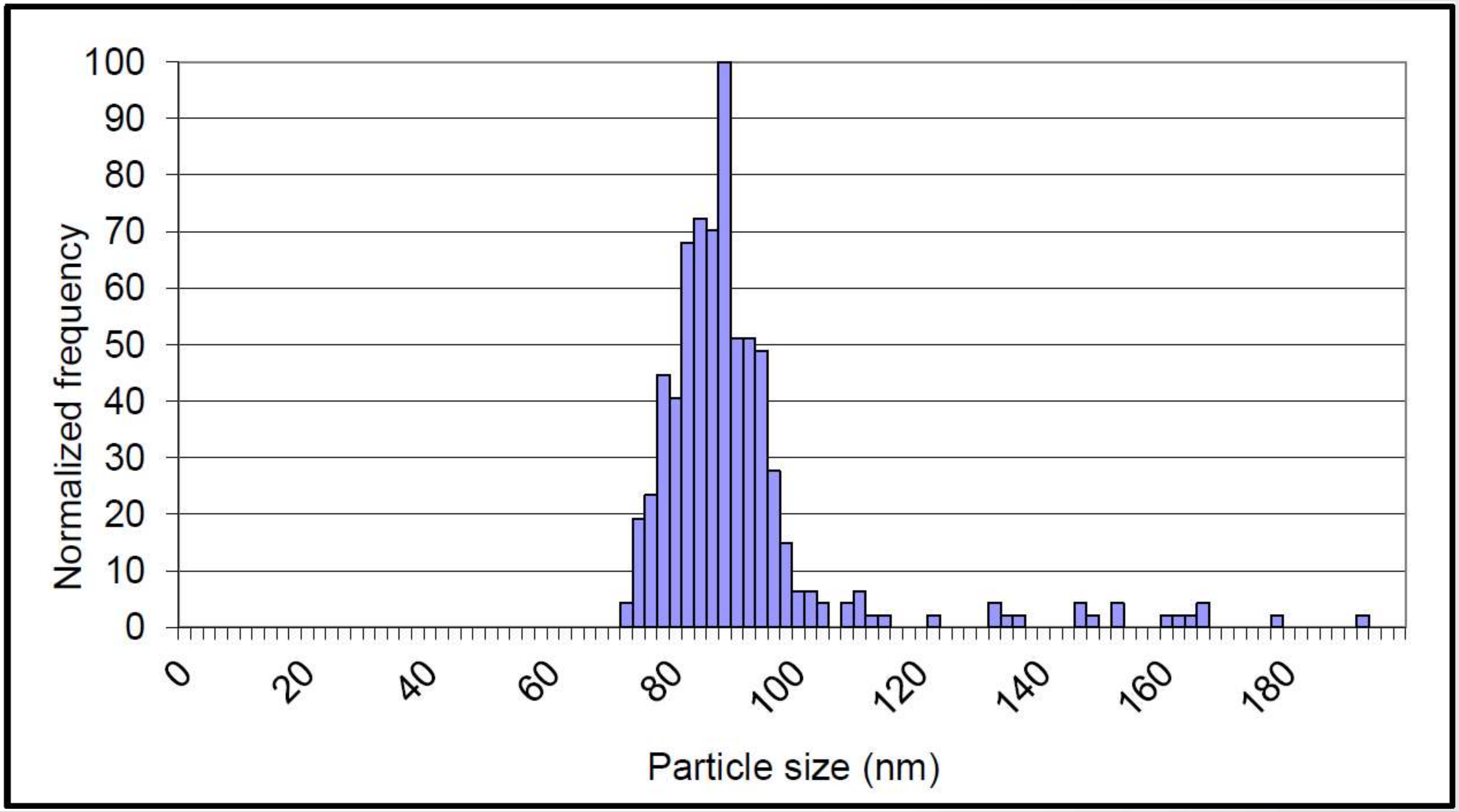
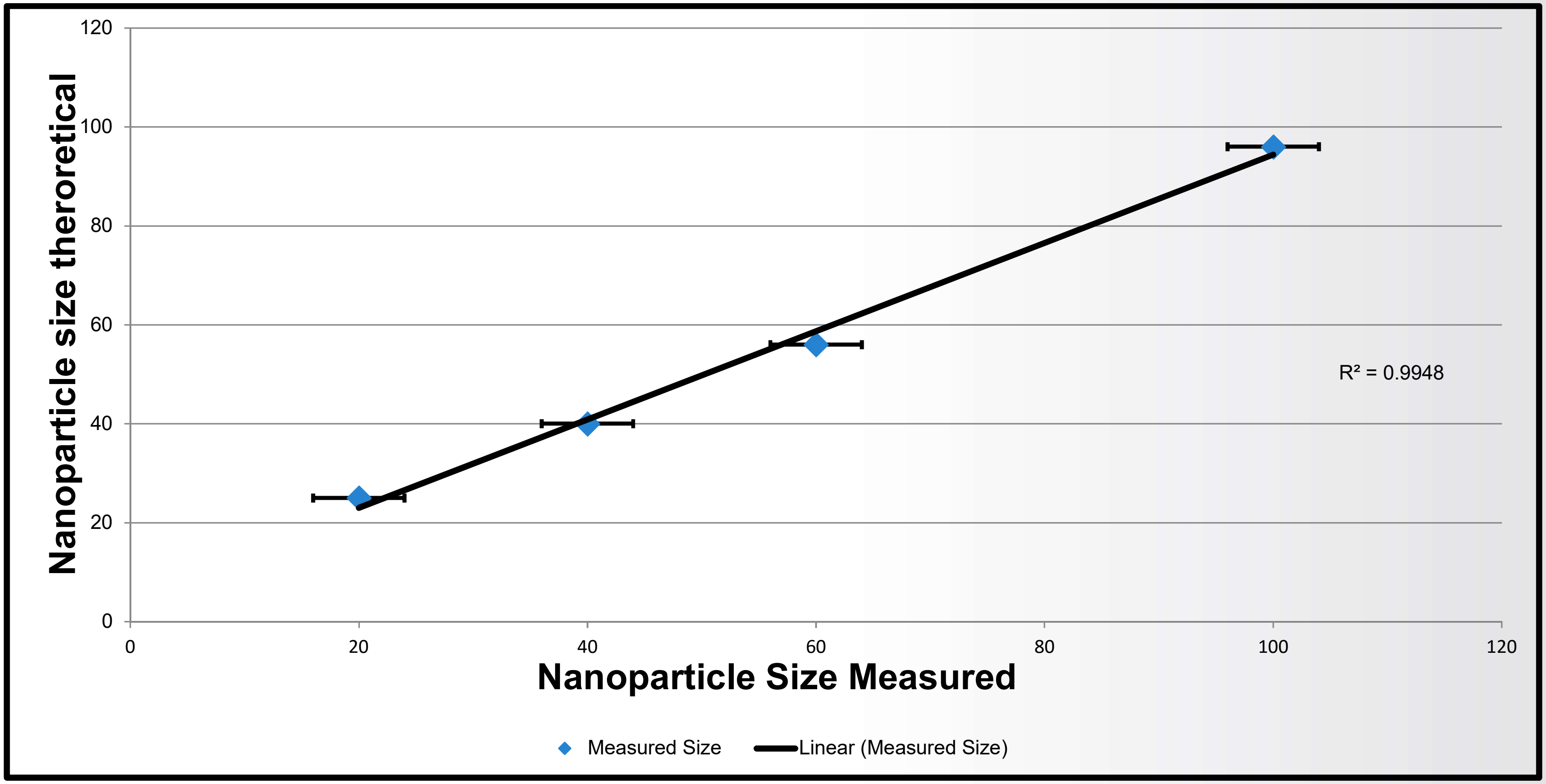
Single Particle ICP-MS for Nanoparticle Characterization

- Particle size distribution



Single Particle ICP-MS for Nanoparticle Characterization



Particle size LOD dependant on detection sensitivity!

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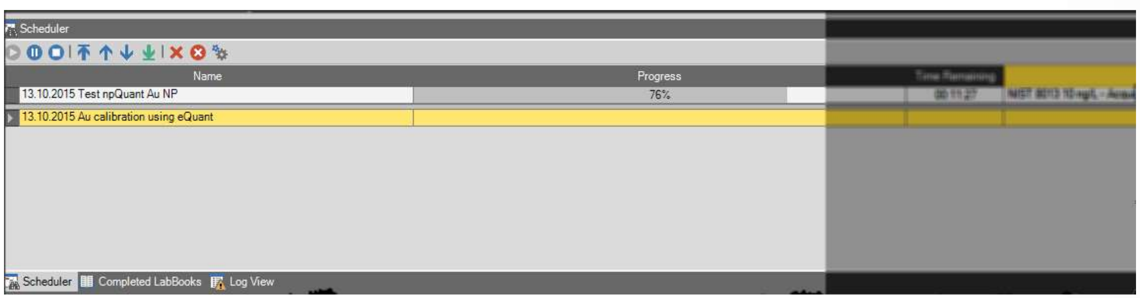
Single Particle Analysis Using npQuant – Now Available!

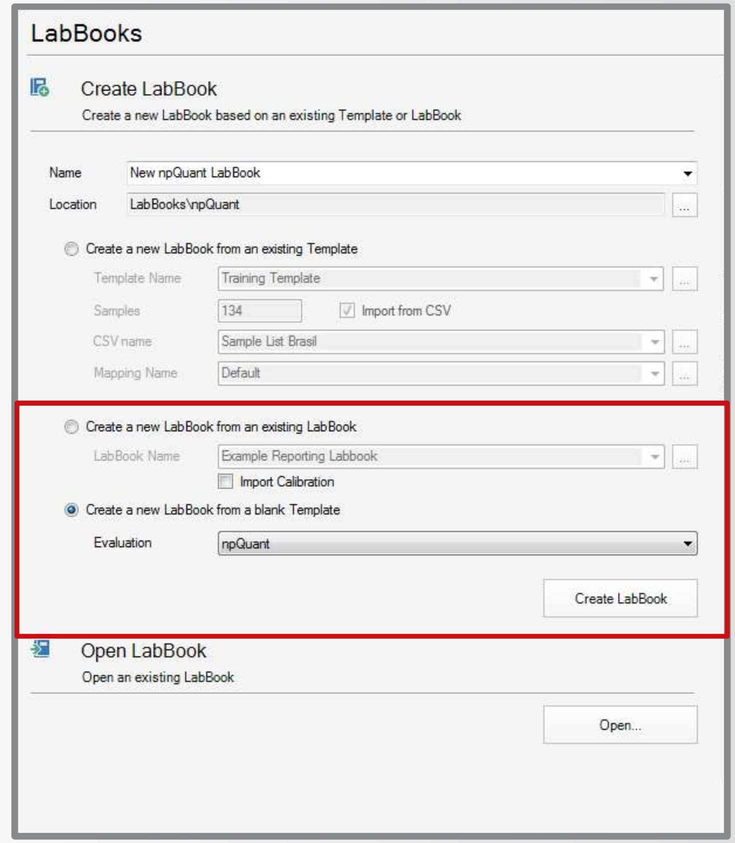
- Thermo Scientific™ Qtegra™ ISDS™ 2.6 SP1 – now available!
- This version supports single particle ICP-MS data collection (npQuant)
- npQuant provides the software tools for simple and accurate single particle nanoparticle characterization
  - Easy experimental setup, short analysis time per sample
  - Higher sensitivity is the key to detecting nanoparticles of smaller and smaller diameters
- The npQuant evaluation module guides the user through method set-up and aids in data evaluation
  - Automated determination of key parameters
  - Statistical evaluation of data sets

Routine characterization of nanomaterials has never been easier!

npQuant – A Total Solution for Nanoparticle Analysis

- npQuant Evaluation Module
  - Dedicated solution for spICP-MS data acquisition and evaluation
  - Now available as an add-on to Qtegra ISDS 2.6 SP1
- Create templates and LabBooks for data collection
- Uses Qtegra platform architecture to control peripheral devices
- LabBooks using different evaluation modules can easily be mixed





*Same hardware configuration needs to be used

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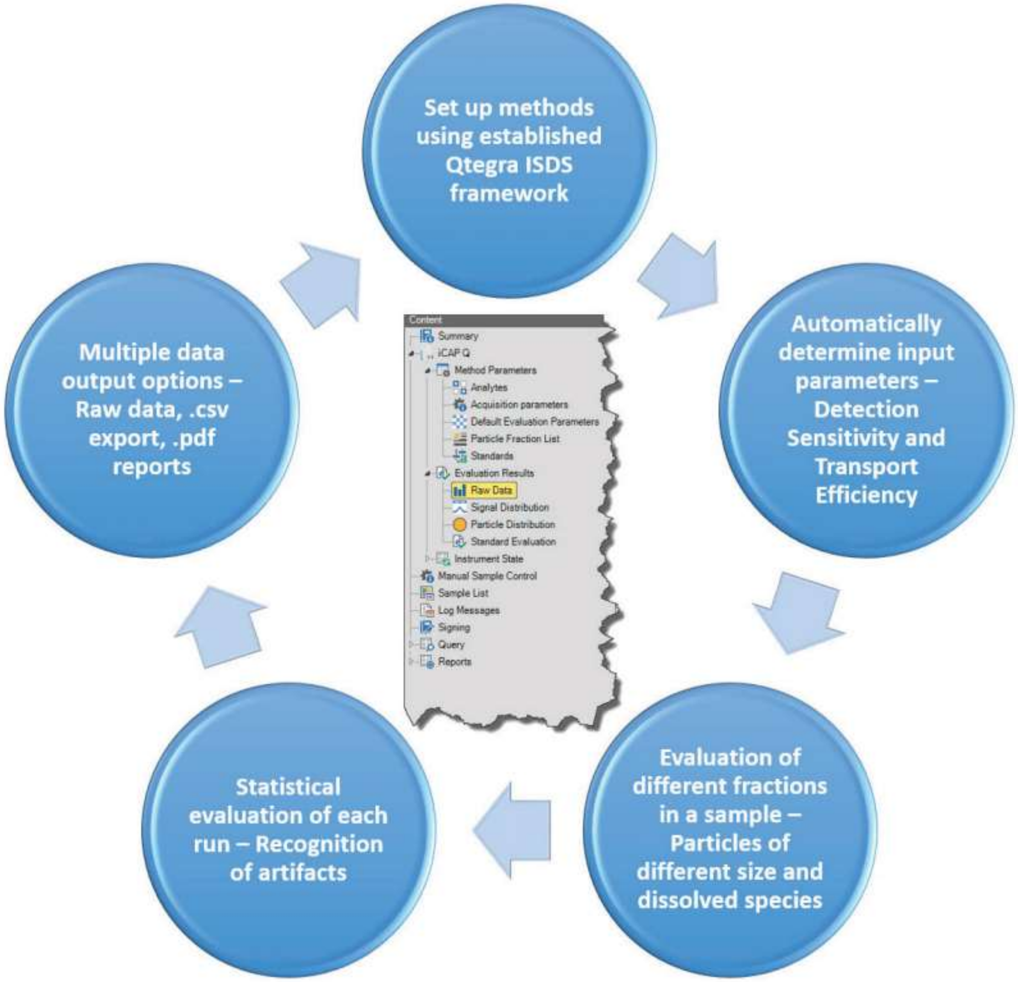
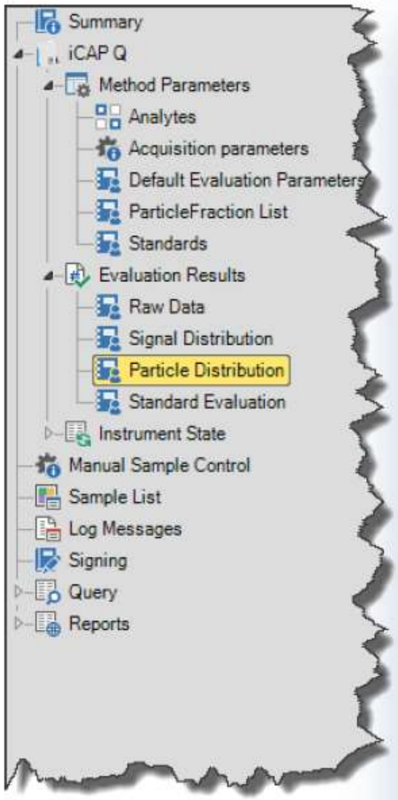
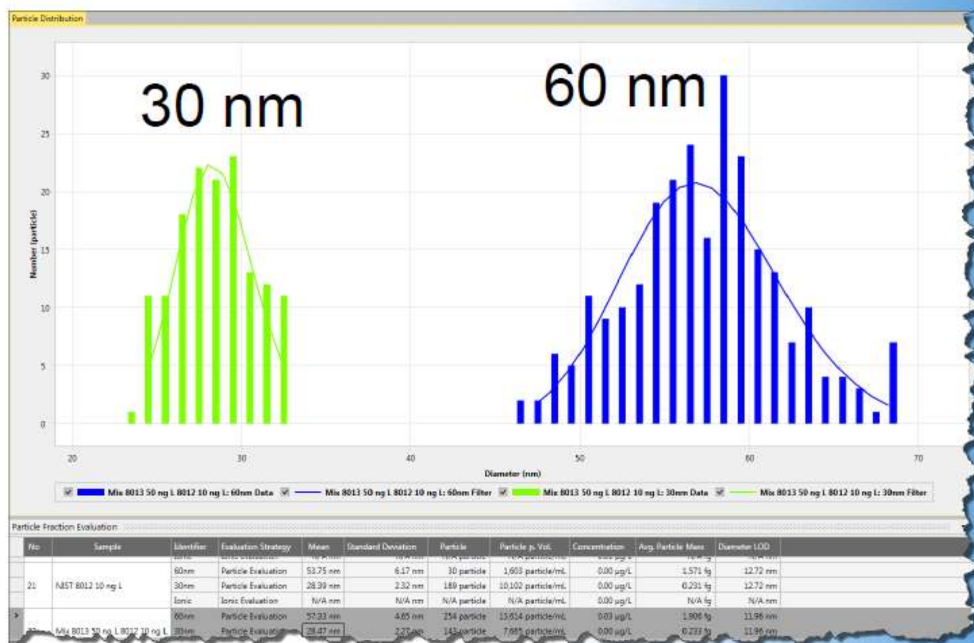
npQuant – An Overview



Particle Size and Concentration Evaluation

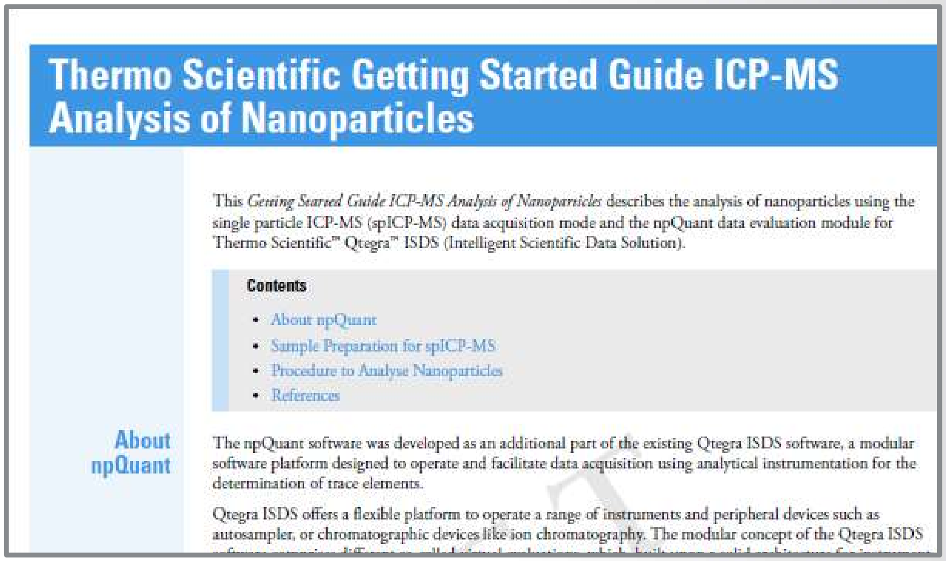


- Distribution of nanoparticles
- E.g. Measure NPs (Au/Ag) in range 10 to 100 nm



Nanoparticles – Documentation Available

- Getting Started Guide
  - Provides background information on the spICP-MS technique
  - Covers best practices



Thermo Scientific Getting Started Guide ICP-MS Analysis of Nanoparticles

This *Getting Started Guide ICP-MS Analysis of Nanoparticles* describes the analysis of nanoparticles using the single particle ICP-MS (spICP-MS) data acquisition mode and the npQuant data evaluation module for Thermo Scientific™ Qtegra™ ISDS (Intelligent Scientific Data Solution).

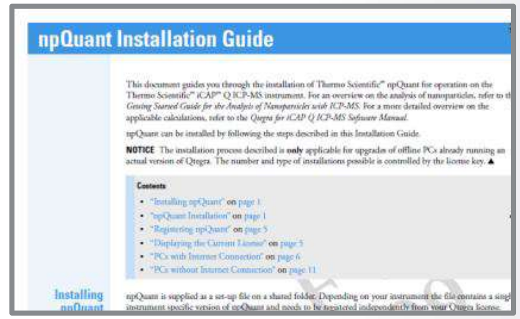
Contents

About npQuant

The npQuant software was developed as an additional part of the existing Qtegra ISDS software, a modular software platform designed to operate and facilitate data acquisition using analytical instrumentation for the determination of trace elements.

Qtegra ISDS offers a flexible platform to operate a range of instruments and peripheral devices such as autosampler, or chromatographic devices like ion chromatography. The modular concept of the Qtegra ISDS

- Technical Documentation
  - Installation Guide



npQuant Installation Guide

This document guides you through the installation of Thermo Scientific™ npQuant for operation on the Thermo Scientific™ iCAP™ Q ICP-MS instrument. For an overview on the analysis of nanoparticles, refer to th *Getting Started Guide for the Analysis of Nanoparticles with ICP-MS*. For a more detailed overview on the applicable calculations, refer to the *Qtegra for iCAP Q ICP-MS Software Manual*.

npQuant can be installed by following the steps described in this Installation Guide.

NOTICE The installation process described is **only** applicable for upgrades of offline PCs already running an actual version of Qtegra. The number and type of installations possible is controlled by the license key. ▲

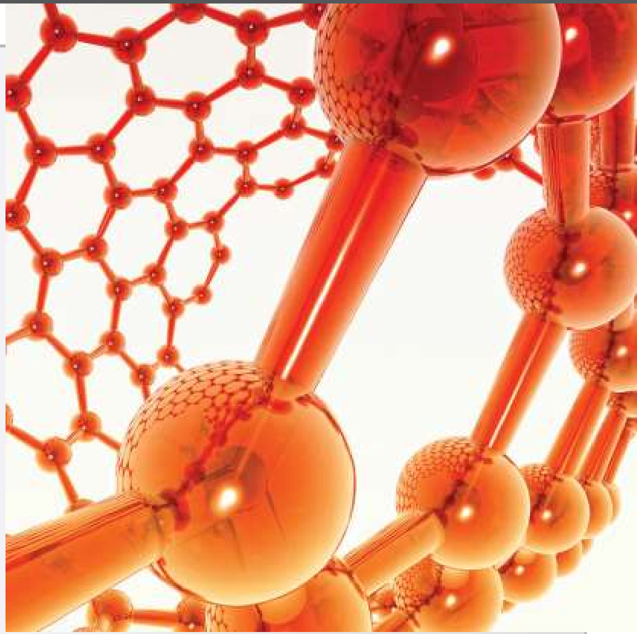
Contents

Installing npQuant

npQuant is supplied as a set-up file on a shared folder. Depending on your instrument the file contains a singl instrument specific version of npQuant and needs to be registered independently from your Qtegra license.

- Software Manual
  - Example LabBook with detailed explanation

Nanoparticles – Additional Information

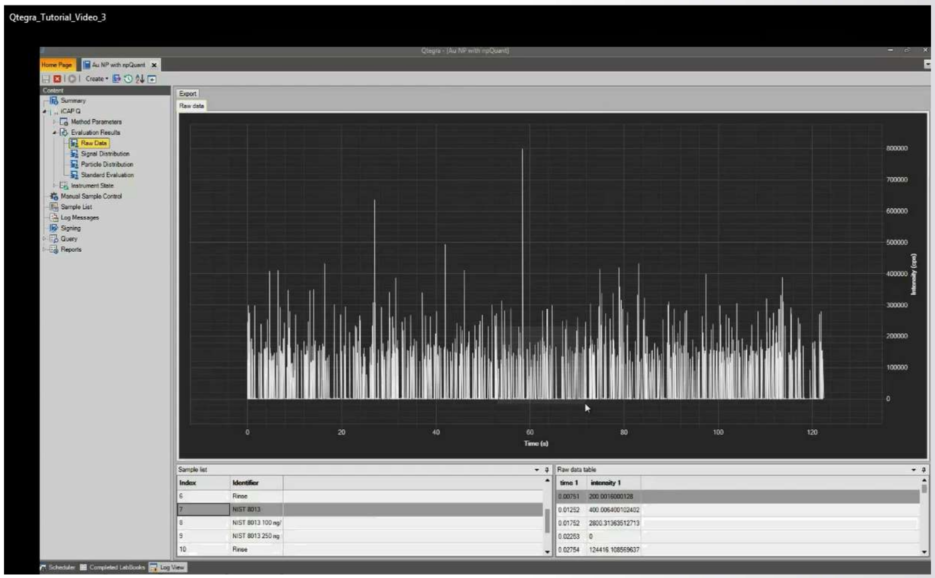


Technical Note (TN43279)

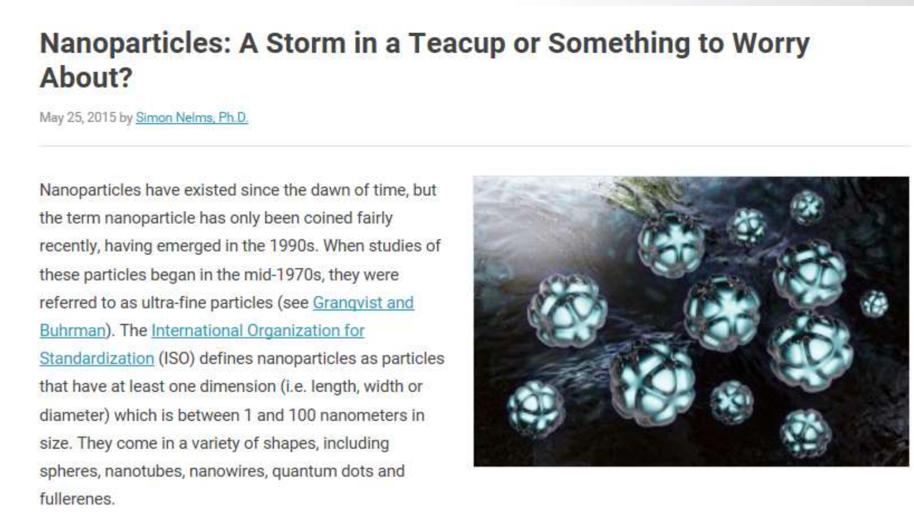
- Contains a brief description of spICP-MS, along with:
  - Method setup
  - Instrumentation Used
  - Evaluation Mode Overview:
    - Particle Evaluation: determine particle-size and number distribution
    - Ionic Evaluation: signals are averaged and evaluated against a calibration curve to determine the nanoparticle concentration in solution
  - Results:
    - Au nanoparticle analysis in certified reference materials (NIST 8012 and NIST 8013)
    - Au nanoparticles in a matrix containing dissolved gold
    - A solution containing Ag nanoparticles with 20, 40 and 60 nm diameters
  - Overview of Data Handling and Calculations

Nanoparticles – Additional Information

- 5-Minute video tutorial on the operation of the npQuant plug-in:
- http://analyteguru.com/videos/npquant-plugin-qtegra-isds-tutorial-video/



- A topical blog written by one of our Regional Marketing Managers for elemental analysis:
- http://analyteguru.com/nanoparticles-a-storm-in-a-teacup-or-something-to-worry-about/



Nanoparticles: A Storm in a Teacup or Something to Worry About?

May 25, 2015 by Simon Nelms, Ph.D.

Nanoparticles have existed since the dawn of time, but the term nanoparticle has only been coined fairly recently, having emerged in the 1990s. When studies of these particles began in the mid-1970s, they were referred to as ultra-fine particles (see Granqvist and Buhrman). The International Organization for Standardization (ISO) defines nanoparticles as particles that have at least one dimension (i.e. length, width or diameter) which is between 1 and 100 nanometers in size. They come in a variety of shapes, including spheres, nanotubes, nanowires, quantum dots and fullerenes.

Single Particle ICP-MS

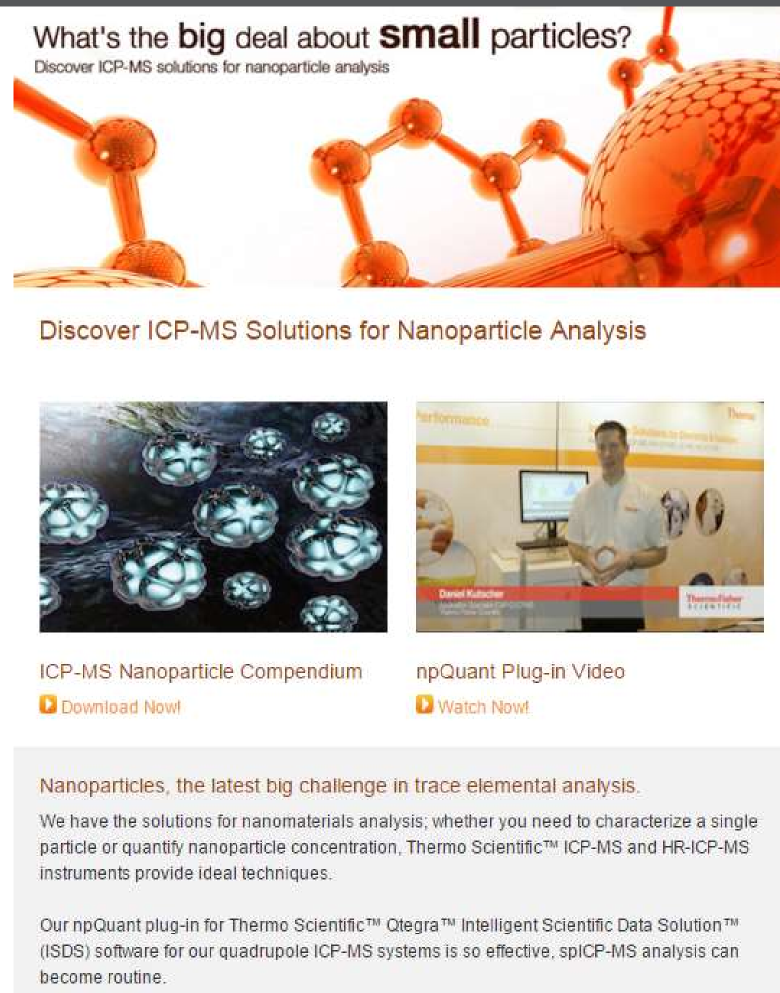
Summary

- Simple setup, no separation unit
- High throughput analysis
- Information about low-molecular metal species, number of particles, metal mass in a particular particle, particle size
- Dwell times in the low ms range are sufficient for monoelemental nanoparticles
    - Faster scanning is required for multielement/isotopic information
- Obtainable limit of detection (LOD), approximately 20 nm

Resources on Nanoparticles

- Additional nanoparticle resources available
  - Visit www.analyteguru.com

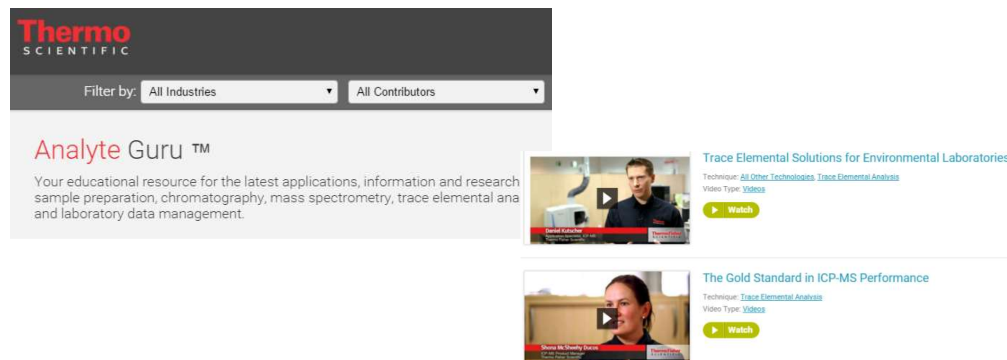
1. Nanoparticle Compendium, 30-page interactive .pdf which covers:
   - Characterization of NPs using ICP-MS
   - Characterization of NPs using spICP-MS
   - The calculations performed in npQuant
   - spICP-MS analysis using HR-ICP-MS
2. npQuant plug-in video
   - Recorded live demonstration of npQuant software with ICP-MS application specialist)
3. Scientific poster
   - Application data comparing nanoparticle characterization via FFF-ICP-MS and spICP-MS



Thank You For Your Attention

Check out our resources on www.thermofisher.com

- Social media:
  - Facebook (Chromatography Solutions)
  - Twitter (www.twitter.com/ChromSolutions)
  - YouTube (www.youtube.com/ChromSolutions)
- Resource Libraries
- Learning Centers
- Our blog site: analyteguru.com
- Videos and Resources (links in Analyte Guru)
- Community pages:
  http://www.thermofisher.com/us/en/home/applications-techniques.html
- Subscribe to our blog and community pages to receive information updates that are relevant to your application!



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