Verification of the Calculation Procedures in the npQuant Evaluation Module for Qtegra Intelligent Scientific Data Solution Software

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Application Brief 43330

Key Words
ICP-MS, Nanoparticles, Single Particle, spICP-MS, Verification

Goal

This note compares the results obtained for spICP-MS determinations with the npQuant plug-in for Qtegra ISDS against a well-established spreadsheet calculation. The verification covers the automated determination of transport efficiency as well as the determination of particle size- and number concentration.

Application Brief

The analysis of nanoparticles using inductively coupled plasma-mass spectrometry (ICP-MS) operated in the so-called single particle (sp) mode has gained increasing attention during recent years. In comparison to other common analytical methods (e.g. TEM, DLS etc.) it has several advantages that explain the growing role of this technique in the common method portfolio for nanoparticle analysis[1].

Above all, spICP-MS is experimentally simple to perform, since no special peripheral devices are required. Furthermore, it allows a sufficiently high number of particles to be probed in a short period of time (e.g. 500 particles in 60s). Last but not least, spICP-MS works at a low particle concentration range (2- to 5 × 10^4 particles mL^{-1}), so that often, no pre-concentration steps are required for real samples, e.g. from environmental studies.

For correct results, however, it is necessary to determine special instrumental parameters, such as sample flow and transport efficiency to the plasma. For data evaluation, conversion of raw data into a particle size, distribution and number concentration can been accomplished through spreadsheet calculations, however, detailed knowledge on the different calculation steps is required to obtain accurate results.

In order to facilitate the use of spICP-MS in routine analysis, a dedicated software solution, npQuant, has been developed as an additional plug-in to the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) software[2]. This note highlights the correct calculation of results through verification against a well established spreadsheet calculation tool, namely the Single Particle Calculation Tool[3] (SPC) created by the experts at RIKILT, Wageningen UR, The Netherlands. This spreadsheet is available and down-loadable for free via the internet[4] and is a widely accepted tool for performing spICP-MS calculations.

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Method

A typical sample analysis workflow for the analysis of nanoparticles using a single quadrupole ICP-MS (Thermo Scientific™ iCAP™ Qc ICP-MS) operated in the single particle mode (spICP-MS) was performed. A well characterized, spherical, monodisperse certified reference material based on citrate stabilized gold nanoparticles with a nominal diameter of 60 nm (NIST 8013) was used for sample preparation. In brief, the reference particles were sonicated for 10 min in an ultrasonic bath and then diluted to obtain a final concentration of 50 ng L^{-1}.

The acquired dataset was evaluated using both the npQuant evaluation module for Qtegra ISDS and the validated RIKILT Single Particle Calculation Tool (SPC). Relevant parameters potentially affecting the calculation (including sample flow, detection sensitivity and threshold values) were taken into account in order to evaluate the data under identical conditions. The calculations covered in this note included the determination of the transport efficiency and the calculation of particle size and number concentration. The transport efficiency parameter is especially crucial for the correct calculation of any results and needs to be determined carefully. As in the SPC tool, the npQuant plug-in uses an automated procedure based on the measurement of a particle containing standard solution.

Results

In order to obtain correct results using spICP-MS, it is crucial to determine the transport efficiency of the ICP-MS system. Briefly, this parameter reflects the fraction of sample that actually reaches the plasma with respect to the amount delivered to the nebulizer. Both, the npQuant plug-in and the SPC allow the determination of the transport efficiency based on a particle measurement, if a suitable standard with known particle size and number concentration is available. Transport efficiency can be assessed either through comparison of the expected and observed number of particles in a run, or the expected and observed particle size or mass. In a larger batch (50 unknown samples plus the required standards, 59 samples in total), 10 independent determinations of this parameter were performed overall. The transport efficiency determined through both of the above mentioned strategies was compared to the result obtained with the SPC tool. The results are summarized in Table 1.

As can be seen from the results, both ways of calculating the transport efficiency with the npQuant module agree with each other and furthermore, as expected, the transport efficiency also agrees well with the value determined using the SPC as a reference calculation tool. It is also evident that the assessment of the transport efficiency using the particle mass shows less variations in a larger batch as it is not as dependent on the conditions of the sample solution (particles may agglomerate over time).

Table 1. Comparison of transport efficiency determined using the npQuant plug-in evaluation module and the SPC (N=10)

	npQuant – Particle number	npQuant – Particle mass	SPC
Average [%]	4.1	4.3	4.6
SD	0.3	0.04	0.3
RSD [%]	7.3	0.9	6.5

Particle Determination

In a similar way, the particle size and number determination was verified against the SPC. The reference particles were analyzed (6 repetitions), and the data was processed using both the npQuant module and the SPC. The average of the calculated particle size and the detected number of particle signals (which is subsequently converted into the number concentration in the sample) are shown in Table 2. Please note that the number of detected particles is slightly lower for the npQuant module, as, in contrast to the SPC, an upper threshold is also applied in addition to the lower threshold in order to discriminate a given signal range for evaluation.

The particle solution was analyzed under optimized concentration conditions (approx. 50 ng L^{-1} for NIST 8013).

Both particle size and particle number concentration determined, using either the npQuant module or the SPC, did not show any significant variation when a t-test was applied ($P>0.05$). The values determined are virtually identical taking into account the aforementioned difference in data collection.

Conclusion

The performance of the npQuant Plug-in for Qtegra ISDS software was successfully evaluated against an external and validated calculation spreadsheet for spICP-MS. The verification procedures comprised both the automated determination of the transport efficiency input parameter, and the correct calculation of the particle size and determined number of particle derived signals in a data set.

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References

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2. Technical Note 43279, Thermo Fisher Scientific
3. Peters, R. et al.; *Journal of Anal. At. Spectrom.* **2015**, 30, 1274-1285
4. https://www.wageningenur.nl/en/show/Single-Particle-Calculation-tool.htm

Table 2. Comparison of particle size and detected particle number

Repetition		SPC	npQuant
1	Size [nm]	52	53
	# · mL^{-1}	21.800	21.000
2	Size [nm]	53	54
	# · mL^{-1}	23.200	22.900
3	Size [nm]	54	54
	# · mL^{-1}	24.300	23.500
4	Size [nm]	54	54
	# · mL^{-1}	24.000	23.300
5	Size [nm]	53	54
	# · mL^{-1}	26.200	25.800
6	Size [nm]	53	54
	# · mL^{-1}	21.700	21.900

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