Retraction

Retraction: Determination of colloidal nanoparticle parameters (*J. Phys.: Conf. Ser* **1420** 012011)

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Determination of colloidal nanoparticle parameters

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Abstract. The paper describes number concentration of colloidal nanoparticles measurement methods and principles. Results additional measurements of particle sizes and zeta potential are presented. Conclusions are drawn about the possibility of using this method for characterization of the reference materials.

1. Introduction
Nanotechnology is actively used to solve scientific and industrial problems. Measurements of the nanoparticles concentration in colloidal suspensions are of great interest for a wide range of industries, including pharmaceuticals, cosmetology, food and microelectronic industries.

However, to date, the methods used to determine the concentration of nanoparticles in liquids have not been standardized yet.

2. Measurand
The sample is colloidal spherical gold nanoparticles (stabilized by citrate buffer) with an average diameter of approximately 30 nm suspended in water. The nominal number of particles is in the range 1-2.5E11 NPs/g.

3. Measurement methods used by VNIIFTRI
During discussion of spICPMS measurements it was noted that this is a very new method and a key aspect of measurements is to gain experience of the measurements and investigate consistency between different instruments.

For spICPMS measurements, dwell time resulting in baseline separation between background signal and particles population are recommended.

Transport efficiency should be determined following the procedure specified in PD ISO/TS 19590:2017, but it is recommended to assess transport efficiency at the start, middle and at the end of the measurement batch to be able to assess the within batch variability of this parameter.

We used two methods to measure the concentration of gold nanoparticles:
— The method of dynamic light scattering (DLS);
— Inductively coupled plasma mass spectrometry (sp-ICP-MS).

3.1 Dynamic light scattering
Dynamic light scattering method [1] is used to measure the size of nanoparticles. In this method, the diffusion coefficient of dispersed particles in a liquid is determined by analyzing the characteristic time of fluctuations measured in the intensity of scattered light. The diffusion coefficient is used to
calculate the hydrodynamic radius of nanoparticles. Thermal (Brownian) motion of particles causes
fluctuations of their local concentration. In turn, these fluctuations lead to local inhomogeneities of the
refractive index of the medium. When a laser beam passes through such a medium, part of the light is
scattered on these inhomogeneities. The fluctuations of the scattered light intensity correspond to the
fluctuations of the local concentration of dispersed particles. Information on the particle diffusion
coefficient is contained in the time-dependent correlation function of the intensity fluctuations.

Particle size measurements are made according to the Stokes-Einstein equation (1):

\[ d = \frac{kT}{3\pi \eta D} \]  

Where:
- \( k \) = Boltzmann constant;
- \( T \) = Absolute temperature;
- \( \eta \) = Solvent viscosity;
- \( D \) = Diffusion coefficient.

To calculate the concentration of particles, data on their size, optical properties (imaginary and real
parts of the refractive index), polydispersity index and count rate (number of photons per second) are
used.

3.2 Inductively coupled plasma mass spectrometry

Inductively coupled plasma mass spectrometry (ICP-MS) [2] is a type of mass spectrometry that uses
an inductively coupled plasma to ionize the sample. The sample, an aqueous suspension, is introduced
continuously into a standard ICP-MS system that is set to acquire data with a high time resolution (i.e.
a short dwell time is used). Following nebulization, a fraction of the nanoparticles enter the plasma
where they are atomized and the individual atoms ionized resulting in a cloud of ions. This cloud of
ions is sampled by the mass spectrometer and since the ion density in this cloud is high, the signal
pulse is high compared to the background signal if a high time resolution is used.

The number of pulses detected per second is a directly proportional to the number concentration of
nanoparticles in the aqueous suspension that is being measured. To calculate concentrations, the
nebulization efficiency has to be determined first using a reference particle.

Particle concentration measurements are made according to the equation (2):

\[ C_p = \frac{C_m}{m_p} \]  

Where:
- \( C_p \) = particle number concentration;
- \( C_m \) = mass concentration of the particle suspension;
- \( m_p \) = mass per particle.

Individual particle mass is calculated according to the equation (3):

\[ m_p = \frac{I_p t_d \cdot V \eta_n \cdot M_p}{RF_{ion} \cdot 60 \cdot M_a} \]  

Where:
- \( m_p \) = particle mass;
- \( I_p \) = particle signal intensity in the sample;
- \( RF_{ion} \) = ICP-MS response for ion standard;
- \( t_d \) = dwell time;
- \( V \) = sample flow rate;
- \( \eta_n \) = nebulization efficiency;
- \( M_p \) = molar mass nanoparticle material;
- \( M_a \) = molar mass analyte measured.
4. Results
Number concentration that was measured by DLS and ICP-MS methods is shown below:
DLS: $1.668 \cdot 10^{11}$ NPs/g;
ICP-MS: $1.616 \cdot 10^{11}$ NPs/g.

However, we have carried out additional measurements of the size and zeta potential of the particles, as the values of the concentration varied from vial to vial within 15%.

According to the results of particle size measurements, the average particle diameter was 30 nm, and the polydispersity reached 21% (fig. 3).

![Figure 3. Gold nanoparticles size and polydispersity index](image)

According to the results of measurements of the Zeta potential of the particles, the average value was about 50 mV, and the deviation reached 27 mV (fig. 4).

![Figure 4. Gold nanoparticles zeta potential](image)

Summing up all the obtained measurement results (size, counting concentration, zeta potential), the methods are acceptable to use for characterization of the reference materials.

5. References.
[1] ГОСТ Р 8.774-2011 "ГСИ. Дисперсный состав жидких сред. Определение размеров частиц по динамическому рассеянию света”.
[2] H.E. Pace, N.J. Rogers, C. Jarolimek, V.A. Coleman, C.P. Higgins and J.F. Ranville 2011 Determining transport efficiency for the purpose of counting and sizing nanoparticles via single particle inductively coupled plasma mass spectrometry. Anal. Chem. 9361–9369.