Particle size and zeta potential analysis via Dynamic Light Scattering (DLS) / Elelctrophoretic Light Scattering (ELS)

Date
28.05.2016

Version
1.1 English

Content

1 Scope

This Standard Operating Procedure (SOP) describes the size and zeta potential analysis for the investigated nanoparticle suspensions within the nanOxiMet project.
Note: The SOP should be taken as guideline and informative advice for DLS analysis and have to be adapted specifically for other DLS instruments.

2 Basics

The aim of this Standard Operating Procedure is the description of the handling and procedure parameters to be used for the DLS comparison measurement for particle size and zeta potential.

3 Materials & Instruments

3.1 Materials

The following materials and chemicals are required:

- HPLC Grade water
- Nanoparticle suspension
- Cuvette (Disposable)
- Pipette

3.2 Instruments

The following instruments are required:

- Dynamic light scattering instrument
- Liquid handling apparatus / Pipette

Note: At the IUTA the dynamic light scattering instrument Delsa-Nano C - Beckman Coulter, Krefeld (Germany) is used to measure particle size and zeta potential of the different nanomaterials in aqueous suspensions. The usage and maintenance of the instruments will be not described in this SOP. Please refer to the manual.

4 Experimental procedure

4.1 Suspension preparation

The suspension preparation has been described in nanOxiMet SOP_- Dispersion protocol_sonication_cup horn_1.1

4.2 Measurement and Instrument settings

For analysis, a minimum of 2 mL of the prior prepared suspension has to be transferred (pipette) into a 4.5 mL disposable plastic cuvette, placed in the analysis device and subsequent analysed for size analysis. The zeta potential is measured as a function of the pH using an automatic titration regime that adjusts the pH of the sample to pre-defined values by adding 0.1 M HCl or 0.1M NaOH. For the titrator a volume of 20 mL suspension is necessary.

Instrument settings / measurement parameters / general conditions

Beckman Coulter (Delsa-Nano C - Beckmann Coulter):

Size measurement

File Save:	Auto
Repetition:	3 (recommended 10)
Manuel Temperature Setting:	No
Auto Print:	Manuel
Equilibrating:	Yes
Statistical Summary	Yes
Equilibration:	15 min
Waiting time:	2 sec
Size Measurements	
Dust Limit	5
Upper Dust Limit	10
Lower dust Limit	100
Minimum Intensity	3000
Pinhole (µm)	50
Analysis Parameter	
General	
Analysis method:	CONTIN
Side cut left	0
Side cut right	0
Display	
Graph x Axis:	Manual 4000 nm
Graph Y Axis	AUTO
Others	
Fitting range	G2(τ)
G2(τ)max	2
G2(τ)min	1.003
Noise cut level (%)	0.3
Molecular weight Analysis	Const
Molecular weight	NO
Cell Parameter	
General	

Measurement item	Size
Measurement type	Type 2
Cell name	Disposable Cuvette
Details	
Correlation type	Log
Size Measurements	
Accumulation Time	70
Dilutent properties	
Dilutent name	Water
- Properties	
RI	1.33
Viscosity	0.89
Dielectrical constant	78.3

Zeta potential measurement

Measurement Parameter:

Auto save:	
File save	auto
Manual Temperature	No
Equilibration	Yes
Statistical summary	No
Zeta measurement:	
Repetition	1
Auto Print	Manual
Equilibration (sec)	30
Wait time (sec)	0
Others:	
Pinhole	50

Analysis Parameter:

Smoluchowski	
Lorentzian Fit	1 Peak
Conversion Equation	Smoluchowski

Cell Parameter:

General:	Flow Cell
Measurement Item	Zeta Potential
Measurement Type	Type 4
Cell Name	Flow Cell
Details:	
Correlator Type	Linear

Zeta Measurement:	
Accumulation times	10
Cell Position	0.7/0.35/0.00/-0.35/-0.7
Zeta Measurement (detail):	
Applied Voltage	Fixed
Selected Voltage (v)	60
Polarity	auto
Maximum Current (mA)	51
Titrator:	
Titration Mode	pH Titration
pH Titration :	
pH Table	pH 7, 9, 7, 4
pH Tolerance	0.1

Dilutent properties

Dilutent name	Water
Properties:	
RI	1.33
Viscosity	0.89
Dielectrical constant	78.3

5 Data Evaluation / Reporting

Light scattering instruments are largely affected by the particle size. For particles < 100 nm the intensity of the scattered light is proportional to the particle size by the exponent of six. In consequence, larger particles might be over-represented, due to their higher scattered intensity, which can overlap the signal of smaller particles in the DLS size measurement. Therefore the data interpretation of polydisperse samples can be difficult The analysis results can be presented as intensities, number and volume concentrations of the particles. However, the intensity data is the basic information obtained from the instrument. The further results number and volume are calculated based on the intensity results. Therefore, the intensity data are used. The cumulate analysis gives two values, the mean size of the particles (x_{cum} or z-average) and a polydispersity index (PDI).

Test report according to ISO 22412:2008 containing the following information:

a. Average particle size, z.average being the mean and standard deviation of at least three repeated measurements*

b. The PDI, being the mean and standard deviation of at least three repeated measurements

c. If the mean values of z.average and PDI are concentration dependent, this value extrapolated to infinite dilution or the value obtained at the lowest concentration

d. All the information required for the complete identification of the sample, including details of particle shape and homogeneity
e. The sampling method used, if known
f. The test method used, together with reference to this International Standard
g. The instrument type and number
h. The dispersion conditions
    1. Dispersing liquid and its cleaning procedure
    2. Concentration of particulate material
    3. Dispersing agents and their concentration
    4. Dispersing procedure
    5. Sonication conditions: frequency and applied power (if necessary)
i. The measurement conditions
    1. Actual concentration investigated
    2. Viscosity and refractive index of the dispersion liquid
    3. Temperature of the sample
j. Analyst identification
    1 Name and place of laboratory
    2 Operator´s name and initials
    3 Date
k. All operation details not specified in this International Standard, or regarded as optional, together with details of any incident that may have influenced the result(s).

* ISO 13321:1996 specifies six repeated measurements. Experience with the method specified in this International Standard indicated that three measurements are sufficient.

6 Quality control

The results are presented as intensity weighted z-average in nm.

7 Safety precautions

In general when handling the nanomaterials, protective clothing and suitable gloves have to be worn at any time and the working area as well as the used materials and instruments have to be labelled. Please follow the safety information of the instrument manufacturer and material provider.

8 Waste disposal

Please follow the disposal advice of the material provider, if available.

Title: Particle size and zeta potential analysis via DLS/ELS

This SOP is valid from: 28.05.2016

This SOP replaces the SOP from: 28.01.2014 (1.0)

Responsible for the implementation of this SOP: Measurement personal

Previous changes: pH titration steps (1.0)

Signature/Date

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