

# Dynamic Light Scattering (DLS) for nanoparticle size-distribution acquisition

Date: 2020-03-16

Tags: Training PSD 11/03/2020 Synth DLS

Created by: James Bird

1 / 4

**Goal : Use DLS to obtain particle size distributions (PSDs) of  $\text{Ti}_3\text{C}_2$  MXene nanoparticles in aqueous suspension**

Procedure :

## Sample preparation

- Dilute MXene from bulk suspension (direct from synthesis - see [\[Experiment MXene synthesis V\]](#)) by creating a single daughter suspension (~ 20 mL) from which to produce multiple, more dilute suspensions (~ 30 mL)
- Dilution takes place by first pipetting a small quantity of one suspension using a disposable 1 mL Pasteur pipette into a glass vial, recording the mass of suspension added, then recording the mass of deionised water added to make up the total mass
- Suspensions are homogenised throughout with vortex mixing
- Final concentration suspensions transferred to cuvette with Pasteur pipette - cuvette exterior gently dried if necessary

## DLS operation

Standard Operating Procedure (SOP) settings (size measurement type):

- Narrow band filter fitted
- Water dispersant ( $\eta = 0.8872$  cP,  $RI = 1.330$ )
- Use dispersant viscosity as sample viscosity
- Temperature = 25 °C with 120 s equilibration time
- ZEN0040 disposable cuvettes
- 173 ° backscatter measurement angle
- Automatic measurement duration
- Three measurements per sample
- Automatic attenuation selection and positioning method seeking optimum
- Analysis model either: General purpose (normal resolution) or multiple narrow modes (high resolution)

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2 / 4

## Results :

Concentrations in wt% calculated using formula:  $w/w_f = (w/w_i \times m_{\text{susp},i}) / m_{\text{susp},f}$  where  $w/w_i$  and  $w/w_f$  are the initial and final concentrations, respectively, and  $m_{\text{susp},i}$  and  $m_{\text{susp},f}$  are initial and final suspension masses, respectively. Bulk suspension direct from the synthesis has concentration  $7.01 \pm 0.12$  wt%.

Sample N°/Name / #	Mass of initial suspension added ( $m_{\text{susp},i}$ ) / g	Total mass of final suspension ( $m_{\text{susp},f}$ ) / g	Final concentration ( $w/w_f$ ) / wt%
Daughter	0.1766	19.5953	$6.32 \times 10^{-2}$
1	0.8447	31.0482	$1.72 \times 10^{-3}$
2	0.4784	31.6336	$9.56 \times 10^{-4}$
3	0.2228	31.5563	$4.46 \times 10^{-4}$
4	0.1265	33.0358	$2.42 \times 10^{-4}$
5	0.0634	33.5099	$1.20 \times 10^{-4}$

Only used Sample #5 at  $1.2 \times 10^{-4}$  wt% and changed measurement parameters (see table below). All samples returned a polydispersity index (PDI)  $> 0.1$ , which defines the threshold below which outputs can only be compared quantitatively. The height of the suspension in the cuvette in Run 4 was made to sit between 10 and 15 mm height (as recommended), whilst the prior runs had the cuvette filled to the triangular marking.

Run N° / #	Record N° / #	Z-average / d.nm	Z-average std / d.nm	Fit model	PDI / dimensionless	PDI std
1	1-3	1496	39.85	Normal resolution	0.897	0.095

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3 / 4

2	4-6	1158	73.43	Normal resolution	0.719	0.122
3	7-9	1388	150	High resolution	0.727	0.087
4	10-12	1503	392.9	High resolution	0.843	0.162

.dts is the raw datafile which can be read into the Zetasizer software program, .csv is an exported, comma-separated summary of the .dts datafile and .png is the plotted data; particle diameter (on a log scale) is plotted against the mean percentage of that diameter interval contributing to the intensity-based particle size distribution. Vertical lines spanning the whole plot height are mean Z-average values (quoted above), where the regions of matching colour spanning left and right of this value correspond to its standard deviation.

## Conclusions:

No confidence is held in any of the values reported here due to the PDI of all samples. The sample type is definitively unsuitable in this condition. A good starting point would be gauging whether finding the optimal concentration can reduce the PDI to return values that can be used quantitatively.

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4 / 4

## Attached files

2020-03-16.dts

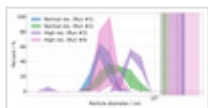
sha256: c864d85ecb5c9989538d5e92bdb911d0aa98d466ac6c12cbdcc830de4dad303f

2020-03-16.csv

sha256: 72928cb48f424d2189f0d057c4209093b41c614d223475b739be9b5e46752256

DLS\_2020-03-16.png

sha256: 1d89445014144067e1af976de6b07b0cbd4935033184639196c6dd5ab322a5ad



Unique eLabID: 20221021-ec88f6f3208dcd8c42ee89004fe8e54bed63e063

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=57>