

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

Date: 2021-04-14

Tags: *PSD DLS Synthesis Optimisation*

Created by: James Bird

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

Procedure :

## Sample preparation

- MXene product suspensions, diluted to concentrations on the order of  $1 \times 10^{-2}$  and  $1 \times 10^{-3}$  wt% from select syntheses in [\[Experiment\] Optimisation of MXene Synthesis - Execution of Plackett-Burman Screening Design](#)
- Sample preparation protocol described in synthesis experiment file and generalised in [\[Experiment\] Dynamic Light Scattering \(DLS\) for nanoparticle size-distribution acquisition](#)
- Target 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,  $\text{RI} = 1.330$ )
- Use dispersant viscosity as sample viscosity
- Temperature =  $25^\circ\text{C}$  with 120 s equilibration time
- ZEN0040 disposable cuvettes or DTS1070 folded capillary cell
- $173^\circ$  backscatter measurement angle
- Automatic measurement duration
- Three measurements per sample
- Automatic attenuation selection and positioning method seeking optimum
- General purpose (normal resolution) analysis model *only*

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## Results :

All sample details and measurement parameters are described in the table below. All individual measurements returned a polydispersity index (PDI) > 0.1, which defines the threshold below which outputs can only be compared quantitatively. Despite this, the combination of three measurements for one sample did meet the data quality criteria defined in the analysis program (Zetasizer) - highlighted in green in the table below - suggesting a Z-average of roughly 1  $\mu\text{m}$  in hydrodynamic diameter. Three outputs returned a PDI of 1 across each of the three sub-measurements - these are highlighted in red. The suspension height in the ZEN0040 disposable cuvettes was made to sit between 10 and 15 mm height as recommended.

Run / #	Concentration / wt%	Record N° / #	Z-average / d.nm	Z-average std / d.nm	Fit model	Cell	PDI / dimensionless	PDI std
1	$1 \times 10^{-2}$	1-3	4430	1693	Normal resolution	ZEN0040	1	0
1	$1 \times 10^{-3}$	4-6	779.1	64.24	Normal resolution	ZEN0040	0.867	0.13
16	$1 \times 10^{-2}$	7-9	2214	256.5	Normal resolution	ZEN0040	0.899	0.145
16	$1 \times 10^{-3}$	10-12	2855	1113	Normal resolution	ZEN0040	0.989	0.02
13	$1 \times 10^{-2}$	13-15	1062	68.83	Normal resolution	ZEN0040	0.330	0.252
13	$1 \times 10^{-3}$	16-18	2448	303.5	Normal resolution	ZEN0040	1	0
12	$1 \times 10^{-2}$	19-21	2270	249	Normal resolution	ZEN0040	0.826	0.132
12	$1 \times 10^{-3}$	22-24	2139	462.7	Normal resolution	ZEN0040	1	0
1	$1 \times 10^{-2}$	34-36	1870	708.3	Normal resolution	DTS1070	0.891	0.184

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.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:

Measurements taken of a product  $\text{Ti}_3\text{C}_2$  suspension from the same run number should show equivalent particle size distributions (PSDs) regardless of concentration or cell type - this was not the case for any of the four synthesis products measured. For two of the four synthesis products measured, the Z-average and associated error overlapped (Run #12 & 16), although the unity or near-unity values of PDI in each case leave doubt as to the validity of the result. The one potentially trustworthy result (with PDI = 0.33) was recorded at the same concentration as half of the other measurements from different synthesis products, hence concentration optimisation for one product may not be applicable across all. A thorough investigation on the effect of concentration on DLS measurement is required, although it may simply be the case that these suspensions are too polydisperse for the algorithm to reliably fit the data.

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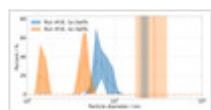
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## Attached files

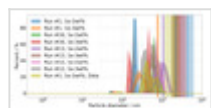
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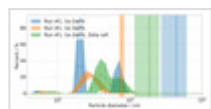
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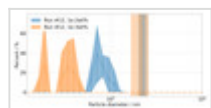
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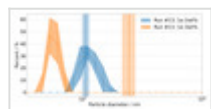
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DLS\_2021-04-14\_Run13.png

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2021-04-14.dts

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2021-04-14.csv

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Unique eLabID: 20221107-18fcd9f0cde9abd0ed7821ec247356935a2d0268

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=67>