



PRECISELY MEASURING THE SIZE OF SILICA PARTICLES ACROSS A WIDE RANGE OF CONCENTRATIONS

Context

Silica nanoparticles exhibit versatility across numerous domains, such as chromatography, ceramics, catalysis, drug delivery, pigments, therapeutic systems, semiconductors, and sensors. Accurate size measurement of silica nanoparticles is crucial in all these cases, as the properties and functionalities of nanoparticles are intricately tied to their size. For instance, the semiconductor industry utilizes silica-based Chemical Mechanical Polishing (CMP) slurries with concentrations ranging from 5% to 30% by weight for polishing silica wafers.

Similarly, in pharmaceutical and research settings, silica nanoparticles of varying sizes and lower concentrations are required for targeted drug delivery and imaging. Therefore, it is essential to accurately measure silica nanoparticles of different sizes over a range of concentrations.

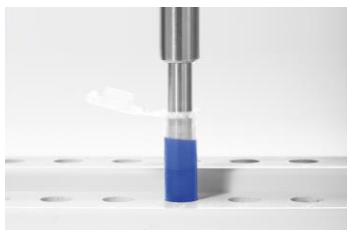


Fig.1: The probe is placed into the sample

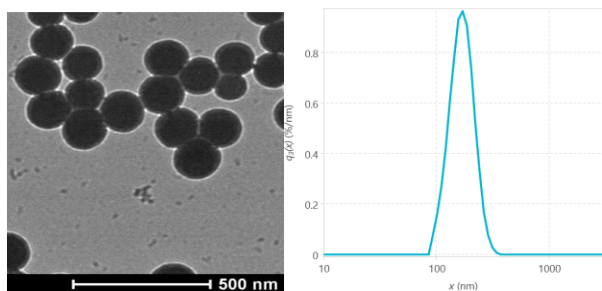
Silica scientists frequently depend on Transmission Electron Microscopy (TEM) due to its reliability in measuring nanoparticle sizes in dry states. However, attaining this level of precision proves challenging under both very high and very low concentrations. In this app note, we demonstrate that the NANOTRAC Flex not only matches TEM's accuracy in nanoparticle size measurement but also excels in analyzing particle size across a broad concentration range.

Sample Preparation

In this study, we used the Stöber process to synthesize silica nanoparticles. One advantage of silica nanoparticles is that their hydrodynamic radii remain consistent even in dry state. We measured their size at various time points during synthesis and made the TEM grid at that time to compare the data obtained from NANOTRAC Flex with TEM. Additionally, we investigated the lowest and highest concentrations at which Flex can accurately measure silica nanoparticle size.

Results

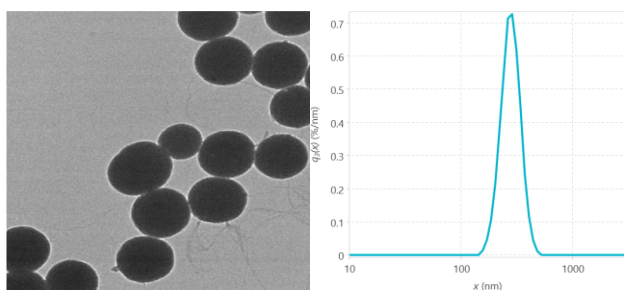
The graphs below depict precise measurements of various sizes of silica nanoparticles, validating the accuracy of NANOTRAC Flex dynamic light scattering (DLS) by comparison with Transmission Electron Microscope (TEM) data. In the DLS method, samples were measured conventionally by immersing the probe into the sample.



Summary		Percentiles	
Data	Value	10%	123.9 nm
MI(nm)	178.6 nm	20%	138.8 nm
MN(nm)	174.4 nm	30%	150.4 nm
MA(nm)	165 nm	40%	160.8 nm
SD	39.6 nm	50%	171.2 nm
PDI	0.05	60%	182.1 nm
		70%	194.1 nm
		80%	209.1 nm
		90%	231.2 nm
		95%	250.6 nm

Peak Summary		
x	Q3(x)	Width
171.2 nm	1	83.3 nm

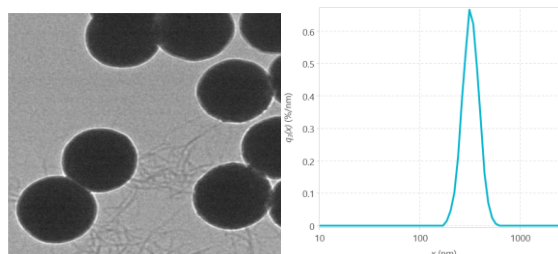
After 20 minutes from the onset of the reaction, the size of silica nanoparticles (measured via Dynamic Light Scattering, DLS) was determined using NANOTRAC Flex, revealing a mean intensity diameter of 178nm, consistent with the TEM data displayed on the left.



Summary		Percentiles	
Data	Value	10%	215.2 nm
MI(nm)	283.7 nm	20%	235.8 nm
MN(nm)	283.1 nm	30%	251.4 nm
MA(nm)	272.5 nm	40%	265.1 nm
SD	54.8 nm	50%	278.7 nm
PDI	0.04	60%	293.1 nm
		70%	309 nm
		80%	328 nm
		90%	356 nm
		95%	382 nm

Peak Summary		
x	Q3(x)	Width
278.7 nm	100%	109.5 nm

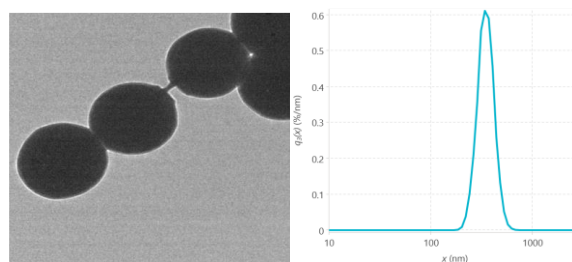
After 60 minutes from the onset of the reaction, the size of silica nanoparticles (measured via Dynamic Light Scattering, DLS) was determined using NANOTRAC Flex, revealing a mean intensity diameter of 284nm, consistent with the TEM data displayed on the left.



Summary		Percentiles	
Data	Value	10%	251.2 nm
MI(nm)	327 nm	20%	273.8 nm
MN(nm)	327 nm	30%	291.4 nm
MA(nm)	315 nm	40%	307 nm
SD	61.3 nm	50%	321 nm
PDI	0.03	60%	337 nm
		70%	356 nm
		80%	378 nm
		90%	409 nm
		95%	439 nm

Peak Summary		
x	Q3(x)	Width
321 nm	100%	122.7 nm

After 120 minutes from the onset of the reaction, the size of silica nanoparticles (measured via Dynamic Light Scattering, DLS) was determined using NANOTRAC Flex, revealing a mean intensity diameter of 327nm, consistent with the TEM data displayed on the left.

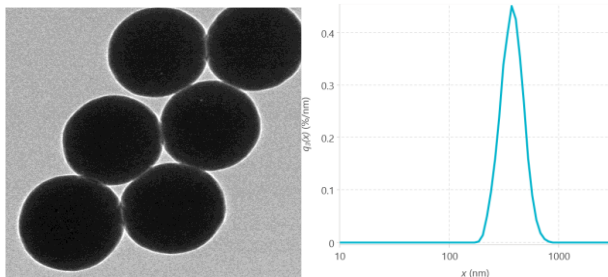


Summary		Percentiles	
Data	Value	10%	273.3 nm
MI(nm)	353 nm	20%	296.9 nm
MN(nm)	352 nm	30%	314 nm
MA(nm)	341 nm	40%	331 nm
SD	63.4 nm	50%	347 nm
PDI	0.03	60%	364 nm
		70%	382 nm
		80%	404 nm
		90%	439 nm
		95%	470 nm

Peak Summary		
x	Q3(x)	Width
347 nm	100%	126.8 nm

After 240 minutes from the onset of the reaction, the size of silica nanoparticles (measured via Dynamic Light Scattering, DLS) was determined

using NANOTRAC Flex, revealing a mean intensity diameter of 353nm, consistent with the TEM data displayed on the left.



Summary	
Data	Value
MI(nm)	391 nm
MN(nm)	392 nm
MA(nm)	371 nm
SD	90.9 nm
PDI	0.05

Percentiles	
10%	279.8 nm
20%	312 nm
30%	337 nm
40%	360 nm
50%	382 nm
60%	406 nm
70%	433 nm
80%	465 nm
90%	517 nm
95%	563 nm

Peak Summary		
x	Q3(x)	Width
382 nm	100%	181.8 nm

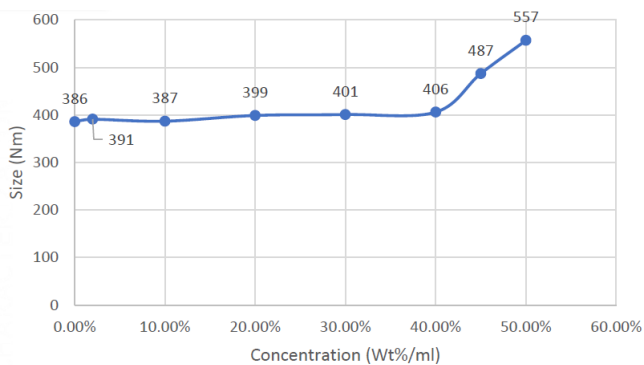


Fig.2: Silica nanoparticles dispersed in ethanol with varying weight percentages.

Summary

The MICROTRAC DLS analyzers are equipped with the remarkable capability to precisely measure samples, not just in terms of their actual size, but also across an impressive range of concentrations. From as low as 0.01 wt% to a substantial 40 wt% of silica nanoparticles, these analyzers can excel in providing accurate measurements across diverse sample conditions.

After 12 hours of the reaction, the size of silicananoparticles (measured via Dynamic Light Scattering, DLS) was determined using NANOTRAC Flex, revealing a mean intensity diameter of 391nm, consistent with the TEM data displayed on the left.



We conducted size measurements of silica nanoparticles of our final product across concentrations ranging from 0.01 wt% to 50 wt% using NANOTRAC Flex. The instrument reliably determines nanoparticle sizes within the range of 0.01 wt% to 40 wt%. Beyond this range, particle sizes tend to appear larger due to increased aggregation.

