

Mini DLS System

On-line particle size analyzer for nanoparticles and emulsions from 20 nm to 2 μ m

INTRODUCTION

Nanoparticles are now standard materials for many industrial products. Like most other processes, the production of nanoparticles needs to be measured in order to be controlled. Dynamic light scattering (DLS) measurements have traditionally performed using laboratory instruments like the Entegris Nicomp system, but the Entegris particle sizing team also has many years of online experience. The Mini DLS builds on these years of experience to develop a flexible and elegant solution that can be adapted to a wide range of nanoparticle manufacturing processes including milling, homogenizing, or self-assembly. A pressurized stream of suspension product connects to the Mini DLS system. This sample is then automatically diluted to achieve an appropriate light scattering intensity for the measurement. The particle size distribution is determined and the system is automatically flushed and cleaned before the measurement sequence is repeated.



APPLICATIONS

- Downstream of a homogenizer or microfluidizer to control pressure or number of passes to achieve the desired particle size
- The manufacture of liposomes and other lipid nanoparticles
- The manufacture of other nanoparticles for drug delivery
- The manufacture of abrasives used for CMP slurries
- Other nanoparticle suspensions or emulsions

FEATURES & BENEFITS

- Flexible protocols to optimize measurement to application
- Automated sampling and measurement to eliminate operator and lab resources
- Continuous data to track changes in particle size distribution
- Easy data transfer to process monitoring control software

SPECIFICATIONS

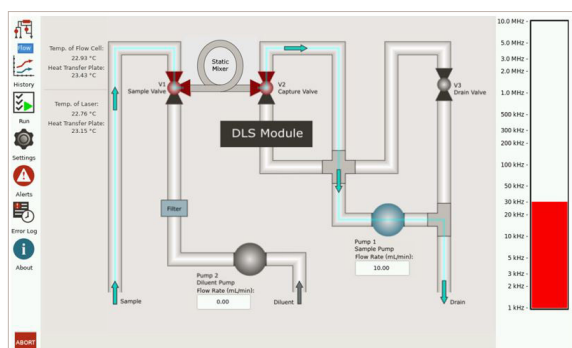
Size range	20 nm – 2 µm (sample dependent)
Concentration	0.1 to 10 wt%
Principle	Dynamic light scattering (DLS)
Measurement angle	90 degrees
Sample volume	2 mL minimum
Accuracy	±10% of certified mean for 90 nm PSL
Laser	35 mW, 639 nm
Detector	PMT
Dimensions	26 L x 10 W x 39 H in (66 x 25.5 x 99 cm)
Weight	88 lb (~40 kg) not including fluid
Case	Stainless steel
Sample temperature*	4° – 40°C (39° – 104°F) internal control range External coolant pump required
Diluent pressure	20 psi max
Diluent filter	0.22 µm
Flow rate	0.1 – 35 mL/min
Control	Touchscreen
External trigger	3.3 V input
Output	RS-232 JSON Intensity weighted gaussian mean Standard deviation Chi squared Distribution by channel number Error status and description, if applicable
Safety	Leak detector
Environmental	Indoor use only
Environmental conditions	Temperature: 4° – 27°C (40° – 80°F) Humidity: 0 – 80% Altitude: 0 – 2000 m

*To get to lowest temperature external cooling and nitrogen purge may be needed

OPERATION

The screen below shows the parameters used to create a measurement protocol. This screen provides the ability to easily adjust system and measurement parameters to optimize the test conditions. The sample loop volume and diluent flow rate control the sample dilution ratio to achieve the chosen intensity test point – typically near 300 kHz. Measurement duration, time between replicates, and flushing sequences optimize the balance between result generation and data quality. A test is started either on the touchscreen or through an external trigger.

The flow screen shown below provides a graphical user interface to monitor the operation. Critical test conditions are displayed in real time. This screen can also be used to manually control operations such as pumps and valves to flush the system.

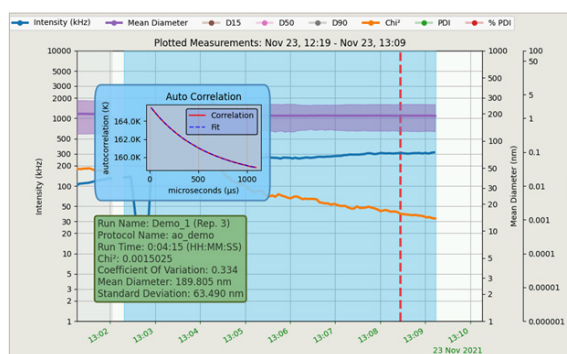


RESULTS

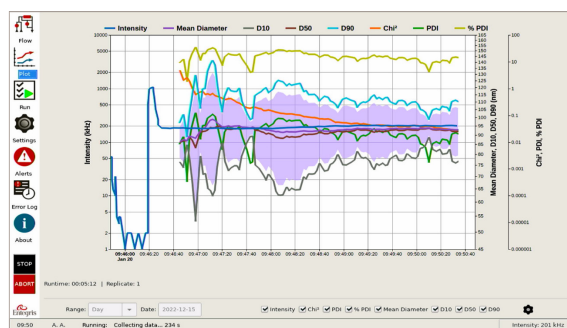
The following parameters can be plotted vs. time during the measurement:

- Intensity
- Mean diameter
- D10, D50, D90
- Chi squared
- PDI and PDI%

A separate chart displays the data correlation function and fit. The screen shown below displays both results vs. time and data for a specific measurement where the mean is 189.8 nm and the standard deviation is 63.5 nm.



Results from a test run can be displayed showing multiple result parameters as a function of time as shown below.



CONNECTIONS

Sample and diluent line:

- Fittings: Swagelok SS-200-61
- Up to 7 meters from process line to instrument
- 5 – 20 psig

Sample: 0.1 – 35 mL/min

Diluent: Max 10 mL/min during autodilution

Nitrogen purge to reduce the effects of humid air inside the enclosure (optional)

Coolant in/out such as chilled water to regulate the temperature of the thermoelectric cooler

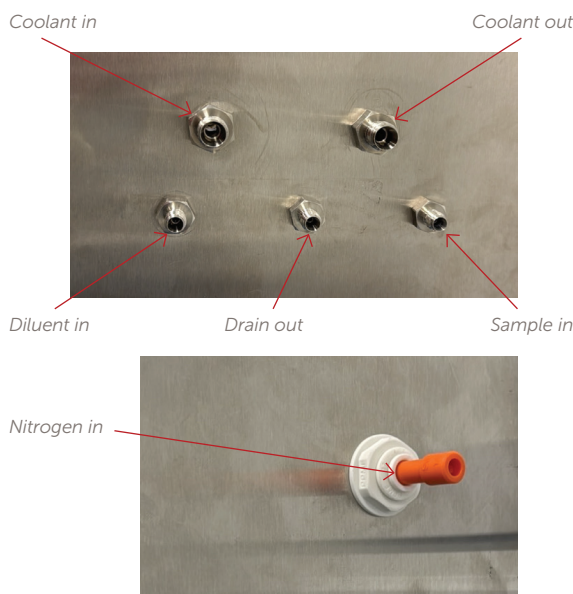
Note: Coolant must be pumped independently from the Mini DLS.

Minimum coolant flow rate = 250 mL/min
(external coolant pump required)

Fittings: Swagelok SS-400-61

Coolant temperature = 4° – 40°C (39° – 104°F)
(to get to lowest temperature external cooling and nitrogen purge may be needed)

Mounting: requires four 3/8" bolts, up to 3.5 ft above diluent source (recommended)



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