ADVANCED PURITY SOLUTIONS | MANUAL

# Mini DLS

*User manual*

## NOTICES



EtQ Document Number

25560

Software

2.2.2.1

Technology Licenses

The hardware and/or software described in this document are furnished under a license and may be used or copied only in accordance with the terms of such license.

**WARNING: Do not use the Mini DLS without having fully read and understood this manual Improper handling can potentially lead to personal injury or damage to the instrument.**

## TABLE OF CONTENTS

# SECTION 1: INTRODUCTION

## 1.1 ABOUT THE MINI DLS

The Mini Dynamic Light Scattering (DLS) system measures the hydrodynamic size of particles ranging from 2 to 2000 nm in diameter. Autodilution in the Mini DLS eliminates the need for manual dilution of concentrated samples, allowing for automated sampling from a process line.

## 1.2 THE MINI DLS USER MANUAL

This manual describes the procedures for installing, operating, and maintaining the Mini DLS system.

**NOTE: This manual is subject to the same end-user license agreement shown in the About screen of the software. A copy of this agreement is provided in the appendix of this manual.**

## 1.3 TECHNICAL SUPPORT

Before contacting Entegris for technical support, be sure to perform the following primary troubleshooting steps:

1. Check that the unit is powered on.
2. Check the software for error messages. Take a picture of any error messages that appear in the Error Log for the relevant date range; sharing these pictures will greatly facilitate technical support.
3. Ensure that all cables are properly connected from the electronics and fluidics to the controlling computer.
4. Check incoming sample and diluent lines. Be sure that tubing is properly connected and inspect connections for leakage or air gaps.

Additional troubleshooting steps can be found in Section 7 of this manual.

Contact Us:

If technical issues persist, contact Entegris at 727 846 0866, Entegris, Inc., 7225 W Oakland Street, Chandler, AZ 85226, USA, or visit the following webpage:

Expedited Instrumentation Technical Assistance | Entegris

Please note the following information for your system to facilitate your service request. This information can be found on the About screen of the touchscreen software:

- Serial number
- Model number
- Software version
- Firmware version

# SECTION 2: SAFETY

This section highlights the key safety issues related to using the Mini DLS.

**WARNING: If equipment is used in a manner not specified by Entegris, the protection provided by the equipment may be impaired.**

## 2.1 CE MARK

The CE mark is a mandatory European marking for certain product groups to indicate conformity with the essential health and safety requirements set out in European Directives. To permit the use of a CE mark on a product, proof that the item meets the relevant requirements must be documented.

The following label is affixed on the Mini DLS to indicate that the instrument has passed CE mark testing and conforms to the European Union Directives for Electromagnetic Compatibility (EU EMC).

CE

## 2.2 RISK ANALYSIS

Using the Mini DLS has inherent safety risks, including fluidics hazards and electrical hazards. Chemical hazards may be present depending on the end user's application of the instrument.

## 2.3 SAFETY RELATED POLICY

Entegris will provide end user information about any safety related upgrades or newly identified hazards with the Mini DLS should it become necessary.

The end user and their subcontractors who work with the Mini DLS must ensure that their respective employees are provided with material safety data sheets from their Environmental Health and Safety (EHS) department for all chemicals that pass through the Mini DLS. The end-user is responsible for checking that the instrument components of the Mini DLS are compatible with their process before use; see the Appendix for more information on instrument specifications.

It is imperative that when working on any piece of equipment, the service technician follows all policies, practices, and procedures established by the end users' EHS group.

## 2.4 SIGNAL WORD DEFINITIONS

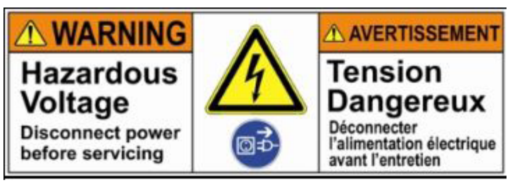
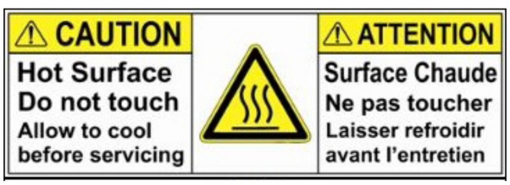
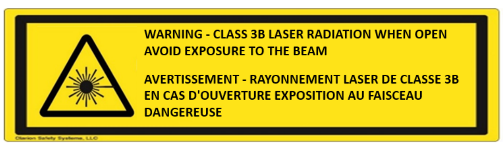
The following symbology is used throughout this user manual next to hazard information:

**WARNING: Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.**

**CAUTION: Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. It may also be used to alert against unsafe practices.**

**NOTICE: Indicates a statement of policy directly or indirectly related to personnel safety or property protection.**

The following symbols are found on the equipment to designate hazards. Operators must consult the user manual when servicing any part of the equipment where these symbols are found.

| LABEL | DESCRIPTION |
|---|---|
|   | Do not reach into the upper chamber without first powering the unit off AND disconnecting the power cable. Failure to adhere to this requirement may lead to death or serious injury. |
|   | The DLS module contains the Peltier system and can get hot. The pumps may get hot during and after operation. Do not touch these surfaces. If service is necessary, power off the system and allow surfaces to cool first. |
|     | The Mini DLS is a class 1 laser product. The DLS module encloses the class 3B laser, resulting in negligible exposure in normal use. not to open the laser module containing the class 3B laser. Any repairs related to the laser module needs to be performed by the manufacturer (Entegris). Please note that staring or even in contact with class 3B laser will cause serious burns. |

## 2.5 ELECTRICAL SAFETY

Access to the electrical connections within the instrument requires a flat head key tool to open the upper cabinet door.

The Mini DLS is designed with a removable power cord. Entegris provides the power cord suitably rated for the Mini DLS with purchase.

**WARNING: Do not replace the power cord with an inadequately rated power cord. Ensure that the wall power connection receptacle is adequately grounded.**

## 2.6 EMISSIONS

The Mini DLS contains a 639 nm, 35 mW laser diode module. This laser is a Class 3B laser, fully enclosed within the lower chamber of the Mini DLS and is not easily accessible due to its containment within the DLS module.

The Mini DLS itself is a Class 1 product, resulting in exposure to negligible levels of non-ionizing laser radiation during normal operation, maintenance, or service.

**WARNING: Do not open the DLS module that encloses the Class 3B laser. Do not stare directly into the laser beam. Doing so can lead to serious injury.**

## 2.7 ERGONOMICS

Care should be exercised when lifting and installing the Mini DLS. See the Appendix for size and weight information.

- Lifting requires two people—one person at each end. Before carrying, disconnect any outside tubing and close/lock both cabinet doors.
- Follow proper lifting technique to avoid injury.

The Mini DLS should be mounted at a comfortable level for interfacing with the touchscreen.

## 2.8 POTENTIAL FAILURE

Potential points of concern can include but are not limited to the following:

- Chemical exposure due to leaking fittings
- Chemical exposure due to maintenance activities
- Potential electric shock from servicing electrical components

Operators should avoid potential hazards by wearing proper personal protective equipment and following on-site procedures for handling the chemicals to be analyzed. Operators should not service electrical components without first powering down the instrument and unplugging the power cable.

## 2.9 FUNCTIONAL INTERLOCKS

The Mini DLS has been designed with functional interlocks that shut off the pumps when the following events occur:

- water block temperature exceeds the acceptable range set in the software
- a leak is detected in the lower chamber

These interlocks are not safety interlocks and are only meant to prevent damage to the equipment.

## 2.10 POTENTIALLY HAZARDOUS MAINTENANCE

Operators should not need to access the upper chamber in normal operation. This chamber houses electronics and should remain closed. If Entegris service personnel need to access the upper chamber electronics, they should power down the system first to prevent electrical shock.

Replacing the filter and performing tubing checks requires access to the lower chamber, which houses the fluidics system. In these situations, the user will not be required to access the laser in excess of class 1.

## 2.11 ENVIRONMENTAL ISSUES AND WASTE TREATMENT

Environmental issues include consideration of sample volumes and wastewater effluent. The Mini DLS drain lines should be connected to an appropriate waste collection system for the customer's sample. Depending on the chemistry of the sample and the parameters used for each measurement, 1 to 10 mL of concentrated sample may be consumed per measurement and collected with wastewater.

## 2.12 DECONTAMINATION AND DISPOSAL

To clean the Mini DLS fluidics system, flush DI water through each flow path using Flow screen controls. If hazardous chemicals were used in the system, use proper decontamination procedures.

Do not dispose of the Mini DLS or its components in the trash. Properly dispose of the unit with an electronics waste management system according to local policies. Before disposing the unit, contact Entegris for instructions on disabling the unit from being powered on again.

## 2.13 SAMPLE HANDLING WARNINGS

- Always handle all substances in accordance with the COSHH regulations (UK) or any local regulations concerning sample handling safety. The Material Safety Data Sheets (MSDS) must be obtained for all substances used in the system and safety precautions and control measures used accordingly.
- Use the instrument in a well-ventilated room if noxious samples or dispersants are to be analyzed.
- Wear a protective respiratory mask if noxious samples or dispersants are being handled, particularly in their dry state during sample preparation.
- Wear protective gloves when handling hazardous materials, or those that cause skin infections or irritations. Chemical protection gloves are suitable, which are tested according to EN 374. At least 4 mil thick. Type of material PVC: polyvinyl chloride, PE: polyethylene, NR: natural rubber, latex, CR: chloroprene (chlorobutadiene) rubber, NBR: acrylonitrile-butadiene rubber, IIR: isobutene-isoprene (butyl) rubber, FKM: fluoro-elastomer, PVA: polyvinyl alcohol, Nitrile. Gloves should be used in accordance with the instructions provided by the PPE supplier, except where additional instructions by the equipment supplier are required.
- Always test a sample for chemical compatibility before using in the instrument.
- After measuring a hazardous sample scrupulously clean the instrument to remove any contaminants before making another measurement.
- Always label samples for analysis using industry standard labeling, particularly if they are handled by a number of staff or stored for long periods. Clearly mark any operator hazard and associated safety precautions that are required for the handling of dangerous materials.
- Always adopt responsible procedures for the disposal of waste samples. Many chemicals are forbidden by law to be disposed of in such a manner as to allow their entry into the water system. The user is advised to seek local advice as to the means available for disposal of chemical wastes in the area of use. Recommendations can be found in the Safety Data Sheets.
- The surfaces of the instrument may be permanently damaged if samples are spilt onto them. If a spill occurs, then the instrument should be disconnected from the power supply before cleaning.

## 2.14 REAGENTS USED

The user is responsible for the chemicals put through the system that come from their own process. Entegris may supply a polystyrene latex (PSL) standard with the system for verifying particle size accuracy performance:

Thermo Scienific Polymer Microsphere Suspension which is defined as non-hazardous based on its SDS. It contains the following chemistries:

a. Polystyrene 1% (CAS no. 9003-53-6) or Polystyrene divinylbenzene 1% (CAS no. 9003-70-7)
b. Sodium azide <0.09% (CAS no. 26628-22-8)

### 2.15 SHUTDOWN AND TAGOUT PROCEDURES

In nonemergency situations shut down the system following these procedures:

1. Notify the appropriate personnel.
2. Press the red Abort icon in the lower left of the control screen as seen in Figure 7, page 10.
3. Turn the power button on the left panel to OFF. The main power cord must be pulled out of its socket, with a lockout-tagout applied to the plug.
4. Shut off DI Water supply to system and affix isolation equipment in accordance with on-site Safety Engineers. Perform Lockout Tagout at the isolation equipment. End user needs to provide main supply disconnect with lockout tagout function to the DI water supply.
5. Shut off Sample In supply to system and affix isolation equipment in accordance with on-site Safety Engineers. Perform Lockout Tagout at the isolation equipment. End user needs to provide main supply disconnect with lockout tagout function to the sample supply.
6. Shut off Coolant In supply to the system (if used) and affix isolation equipment in accordance with on-site Safety Engineers.

In emergency situations shut down the system following these procedures:

1. Notify the appropriate personnel.
2. Press the red Abort icon in the lower left of the control screen as seen in Figure 7, page 10.
3. Turn the power button on the left panel to OFF. Main power cord need is pulled out and apply lockout tagout for the plug.
4. Shut off DI Water supply to system and affix isolation equipment in accordance with on-site Safety Engineers. Perform Lockout Tagout at the isolation equipment. End user needs to provide main supply disconnect with lockout tagout function to the DI water supply.
5. Shut off Sample In supply to system and affix isolation equipment in accordance with on-site Safety Engineers. Perform Lockout Tagout at the isolation equipment. End user needs to provide main supply disconnect with lockout tagout function to the sample supply.
6. Shut off Coolant In supply to the system (if used) and affix isolation equipment in accordance with on-site Safety Engineers.
7. To relieve hazardous energies bleed sample in, diluent in, and coolant in lines until empty.

## SECTION 3: DESCRIPTION

### 3.1 OVERVIEW

The Mini DLS consists of the following major features, shown in Figure 1:

- Upper Chamber: contains circuitry
- Lower Chamber: contains fluidics
- Touchscreen Panel: contains the software interface for operating the Mini DLS

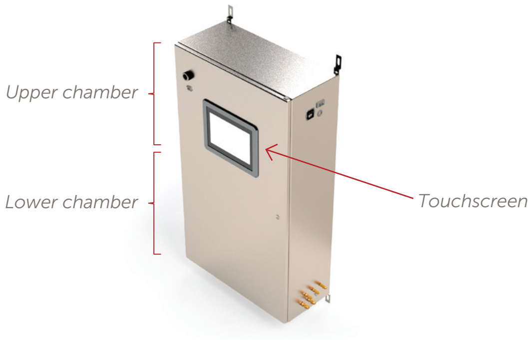



*Figure 1. Mini DLS.*

### 3.2 UPPER CHAMBER FEATURES

The upper chamber houses the major electrical components of the Mini DLS.

The simplified model in Figure 2 shows these components without their wiring connections:

1. Temperature controllers
2. CPU board and correlator board
3. Control board
4. PMT module
5. Power supply module
6. USB hub

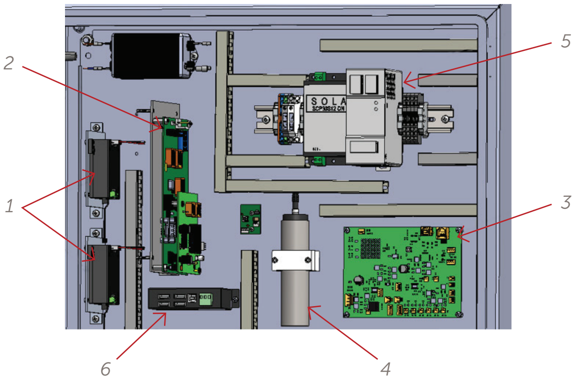



*Figure 2. Upper chamber.*

### 3.3 LOWER CHAMBER FEATURES

The lower chamber houses the fluidics system of the Mini DLS.

The simplified model shows the major components of the fluidics system without their tubing and wiring connections:

1. Valve 1 (V1)
2. Valve 2 (V2)
3. Valve 3 (V3)
4. DLS Module
5. Pump 1 (Sample Pump)
6. Pump 2 (Diluent Pump)
7. Filter Assembly

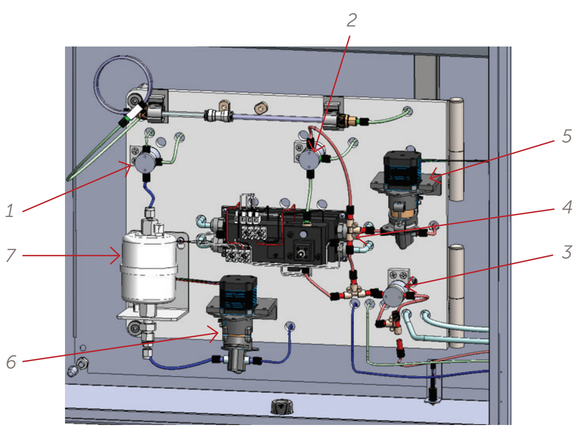



*Figure 3. Lower chamber.*

#### 3.3.1 Internal Connections

The lower chamber components receive power from wiring connections through the middle panel. Fluid flows between each component through PFA tubing. Figure 4 shows the lower chamber components when tubing and cables are connected. These connections are pre-installed with the system. Only trained Entegris personnel should disconnect or reconnect if needed.

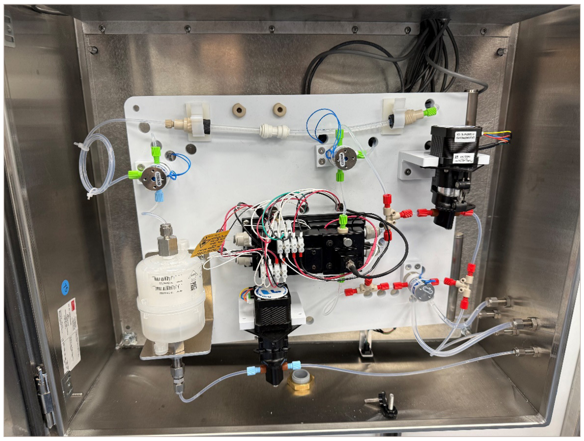


*Figure 4. Internal connections.*

#### 3.3.2 External Connections

Input and output tubing connects to the right side of the enclosure, labeled below in Figure 5. (See Appendix A-1 for specifications.)

- **Diluent:** Clean water enters here. The Mini DLS uses this source to flush the system clean and to automatically dilute the sample during a run.
- **Sample:** Concentrated sample enters here.
- **Drain:** Liquid drains out of the system here.
- **Coolant:** The coolant lines are connected to a water block for the laser and flow cell. The Mini DLS uses an alert system to abort operation when the heat transfer plates are too hot or too cold (limits defined in Settings).
- **Nitrogen:** Nitrogen gas can be injected here to reduce the effects of humid air inside the enclosure, if needed. The gas will flow back out through small air gaps around the door.

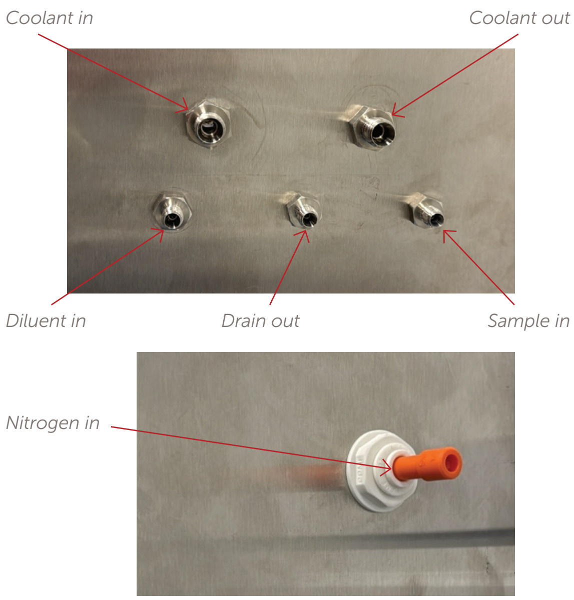



*Figure 5. External connections.*

## 3.4 TOP PANEL FEATURES

The top panel contains the following electrical ports, shown in Figure 6.

1. **Remote trigger:** used for connection to remote trigger for beginning runs remotely, if needed
2. **Power entry:** connects the Mini DLS to a power source; use the power switch to turn the Mini DLS on or off.
3. **DB9:** used for serial connection (RS232 output), if needed
4. **USB:** used for database backup or firmware upgrade
5. **Power On Button:** used to power the unit on

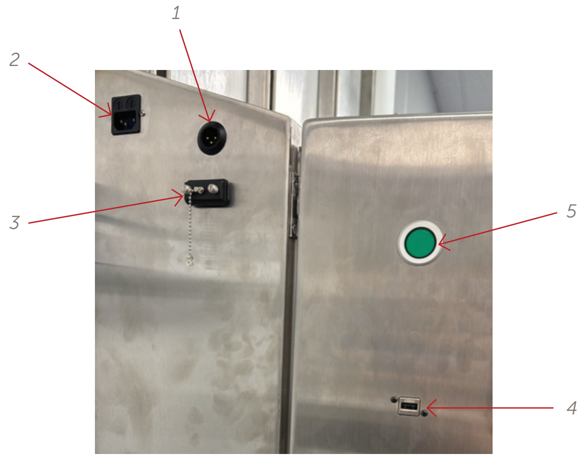



*Figure 6. Top panel.*

## 3.5 TOUCHSCREEN SOFTWARE FEATURES

Users can operate the Mini DLS using the touchscreen software shown below (Figure 7). Touch the screen with a single finger or stylus to control the software.

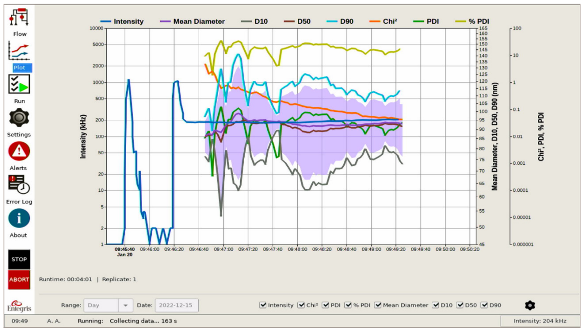
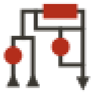


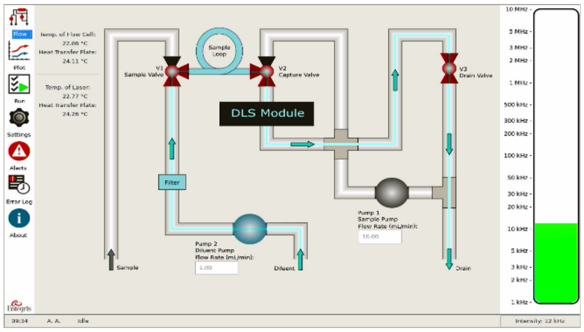

*Figure 7. Touchscreen software.*

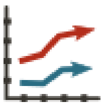
Tap the shortcut icons on the left side of the interface to navigate to each screen. A description is given below for each screen:



The Flow screen contains manual controls for the pumps and valves.

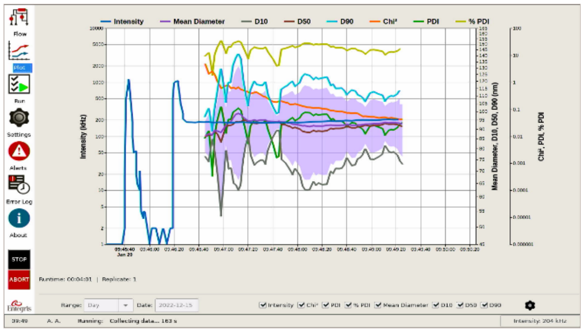
To energize a pump or valve, tap its icon on the screen.

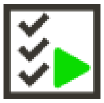


The Plot screen displays the live data collection and contains tools for viewing previously collected data in the database.

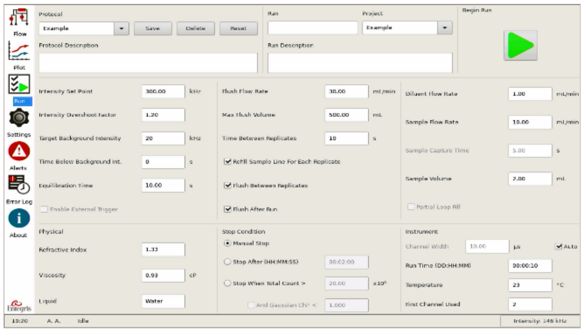
See Section 5.5.1 (page 25) for more information on viewing data.





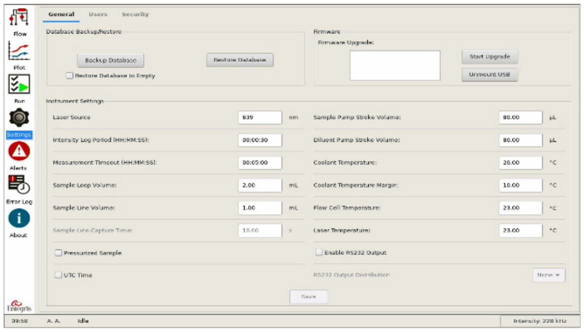
The Run screen contains the options for creating protocols and starting a run.

See Section 5.4.4 (page 23) for more information on setting up a protocol for a run.



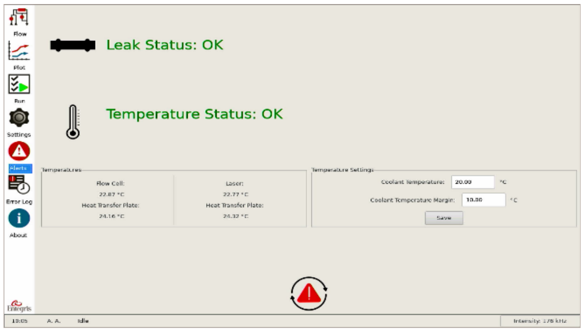
The Settings screen contains the instrument, user, security, and database settings.

See Section 5.3 (page 17) for more information on settings.

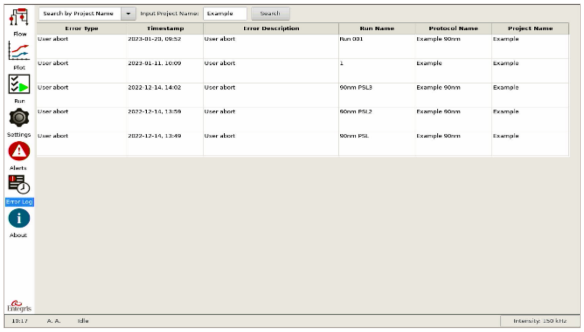


The Alerts screen displays the leak status and temperature status. The software will automatically lock to this screen when an alert is active.

If the alert has been resolved, tap the red triangle button at the bottom of the screen to clear the alert.




The Error Log contains a log of all errors that have occurred in the database.



The About screen displays the license agreement and other product information

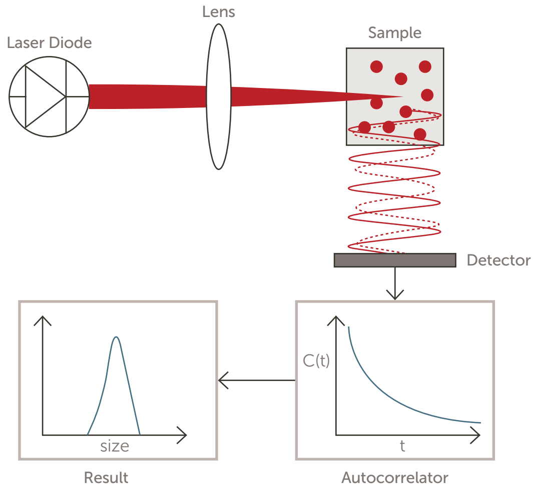



*Figure 8. Dynamic light scattering.*

## SECTION 4: OPERATING PRINCIPLES

### 4.1 DYNAMIC LIGHT SCATTERING (DLS)

#### 4.1.1 Overview of DLS

The Mini DLS system measures particle sizes from 2 nm to 2000 nm and is designed to operate on a process line. Dynamic light scattering (DLS) technology works to produce a mean particle size as shown in Figure 8.

The DLS module focuses a laser into a dilute suspension of particles. Each particle illuminated by this laser beam scatters light in all directions. The detector measures the intensity fluctuations that result from the random motion of the particles, and it sends signals to the autocorrelator during the measurement. The autocorrelation function can then be mathematically deconvoluted into a particle size distribution.

In recent years, the technique of dynamic light scattering (DLS)—also called quasi-elastic light scattering (QELS) or photon correlation spectroscopy (PCS)—has proven to be an invaluable, analytical tool for characterizing the size distribution of particles suspended in a solvent (usually water). The power of the technology is most apparent when applied to the difficult particularly for diameters below 300 nm submicron size range, where most competing measurement techniques lose their effectiveness or fail altogether. Consequently, DLS-based sizing instruments have been used extensively to characterize a wide range of particulate systems, including synthetic polymers (e.g., latexes, PVCs, etc.), oil-in-water and water-in-oil emulsions, vesicles, micelles, biological macromolecules, pigments, dyes, silicas, metallic sols, ceramics and numerous other colloidal suspensions and dispersions.

#### 4.1.2 Intensity Fluctuations: The Effects of Diffusion

In the Mini DLS, the quality of interest is not the absolute measured intensity, per se. Rather, DLS concerns itself with the time behavior of the *fluctuations* in intensity. Scattered intensity fluctuates with time because it is measured as the net intensity resulting from the interference of many different scattered waves, each originating from a different particle.

The suspended particles are not stationary. They move, or diffuse, in random-walk fashion by a process called Brownian motion. The positions of the particles – and, consequently, the phases of the scattered waves – fluctuate randomly with time. Relatively small movements in particle position can effect significant changes in phase. The key physical concept underlying DLS particle sizing measurement is the fact that the time signatures of these intensity fluctuations depend on the size of the particles.

These fluctuations are used to create the correlation function from which a diffusion coefficient *D* can be determined. The particle size *R* is then calculated using the Stokes-Einstein equation:

$$D = \frac{kT}{6\pi\eta R}$$

where $k$ is Boltzmann's constant ($1.38 \times 10^{-23}$ J·K$^{-1}$), T the temperature in Kelvin, and $\eta$ the shear viscosity of the solvent (e.g. $\eta$ = 0.89 cP for water at 298 K).

#### 4.1.2.1 SIMPLIFIED SITUATION: TWO PARTICLES

Consider a simplified situation in which there are only two particles in suspension. Then, the net intensity at the detector is a result of the superposition of only two scattered waves.

In the figure below, light travels from the laser to the particles, where it is scattered by the particles. The scattered light travels to the PMT detector through a pinhole aperture. Each path length (*l*) is labeled for Particle 1 and Particle 2.

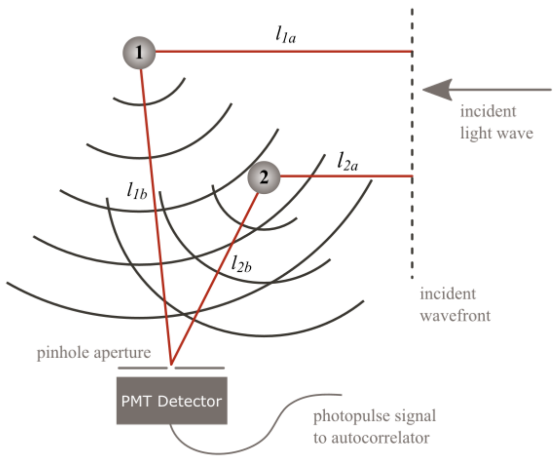



*Figure 9. Simplified scattering model (two diffusing particles).*

The optical path lengths are then defined as follows (assuming a refractive index of 1.0 for simplicity):

$$L_1 = l_{1a} + l_{1b}$$

$$L_2 = l_{2a} + l_{2b}$$

The difference in optical path lengths ($\Delta L$) can be defined as equal to $L_1 - L_2$.

When the positions of the two particles are such that $\Delta L$ becomes equal to an integral multiple of the laser wavelength $\lambda$, then the two scattered waves will arrive in phase at the detector. This constructive interference produces the largest possible intensity at the detector. At the other extreme, the particles will sometimes position themselves such that $\Delta L$ equals an odd number of half wavelengths, $\lambda/2$. In this case, the two waves arrive at the detector completely out of phase with each other, resulting in destructive interference, or 0 net intensity.

Over time, diffusion of the particles will cause the net intensity at the detector to fluctuate between these two extreme values.

#### 4.1.2.2 INTENSITY FLUCTUATIONS VS. PARTICLE SIZE

The time scale of the fluctuations depends on the particle diffusivity, and therefore on the particle size. Representative total intensity signals for "small," "medium," and "large" particles are shown below, assuming all other factors such as temperature and refractive index are equal.

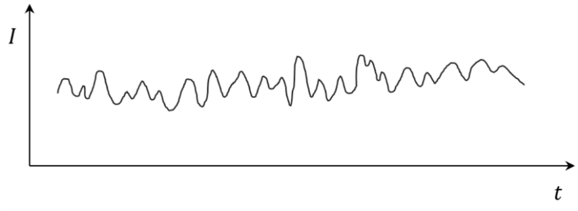



*Figure 10(a). Representative intensity vs. time for "small" particles.*

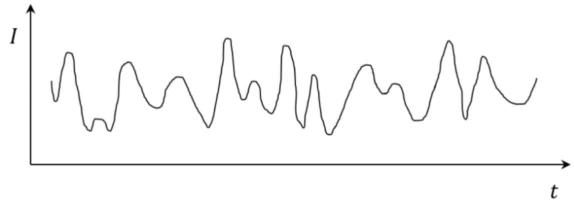



*Figure 10(b). Representative intensity vs. time for "medium" particles.*

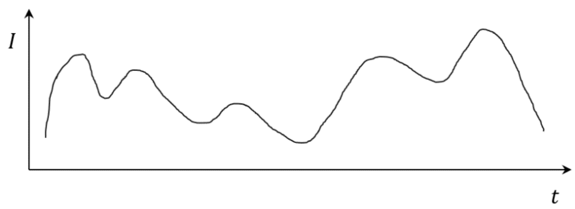



*Figure 10(c). Representative intensity vs. time for "large" particles.*

### 4.1.3 The Autocorrelation Function

The DLS particle sizing technique uses an autocorrelation function, denoted by $C(t')$, to study the intensity fluctuations previously described. The autocorrelation function makes many different comparisons between the value of the net scattering intensity Is at a given time t and the value of $I_s$ at an earlier time, $t - t'$, according to the following relationship:

$C(t') = < I_s (t) \times I_s (t - t') >$

The brackets < > are shorthand symbols that represent the running sum over many values of $t$ for a given time interval $t'$.

For example, consider a typical intensity signal. We arbitrarily choose a time $t$ and record the value of $I_s$ at that time – $I_s (t)$. We next consider a previous time equal to $t - t_1'$, where $t_1'$ is very small time interval. We evaluate $I_s$ at this slightly earlier time, $I_s (t - t_1')$. Because $t_1'$ is presumed to be small, $I_s (t - t_1')$ must be very similar to $I_s (t)$. Thus, the two intensity values are said to be highly correlated.

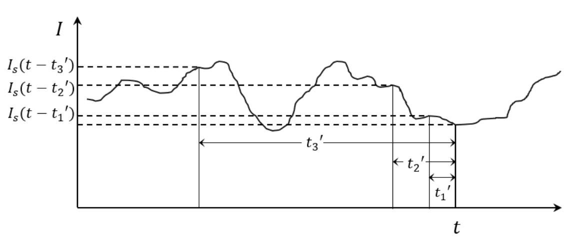



*Figure 11. Computation of the autocorrelation function.*

With a larger time interval, $t_2'$, the two values of $I_s$ are more likely to differ. This means that the two sampled intensities are less correlated, on average, but a relationship remains.

With an even larger time interval, $t_3'$, there is hardly any relationship between the two $I_s$ values. The two values could easily both be high, both be low, be opposite (one high and the other low), or some other intermediate possibility.

A meaningful result from the autocorrelation function $C(t')$ requires many products of $I_s (t) \times I_s (t - t')$ using many different values of $t$, for a given $t'$. Additionally, this calculation must be repeated over many values of $t'$. The result is a well-defined, smooth representation of $C(t')$. In the ideal case of non-interacting particles of uniform particle size, $C(t')$ becomes an exponentially decaying function that can be easily modeled.

In a 5-minute particle size analysis of 200 nm particles, the Mini DLS performs approximately 15 *million* multiplications in order to obtain the autocorrelation for *one* value of $t'$ (e.g. for $t'$ = 20 microseconds in the first channel). The instrument makes 64 such sets of calculations simultaneously in order to obtain $C(t')$ for 64 different values of $t'$.

### 4.1.4 Digital Autocorrelation

With the Mini DLS, the scattered light intensity Is is measured as a discrete number of photopulses per unit of time. The larger the number of pulses counted in that time unit, the larger the intensity. For example, in typical operation the Mini DLS might display a rate of about 300 kHz, updated every second. The sequence of values might resemble the series 302, 297, 299, 304, etc. These values represent the number of photopulses detected in each preceding one-second interval.

During a measurement, the sampling of $I_s$ may need to occur at a much smaller time interval than one second. For example, measuring 100 nm particles would typically require $I_s$ sampling about every 10 microseconds. In the typical example of 300 kHz, this rate of 300,000 pulses per second corresponds to an average instantaneous intensity of 3 pulses per 10 microseconds.

The autocorrelation function is sensitive to rapid changes in $I_s$, caused by rapid diffusion of particles. The smaller the particle, the smaller the time interval should be. This time interval is referred to in the Mini DLS software as *channel width*.

In the 300 kHz example, the number of pulses counted in each 10-microsecond time interval may vary anywhere from 0 to 5 on average. An example of these pulses counted over several time intervals of width *t'* is represented below.

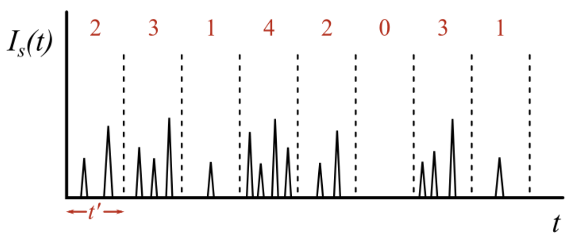



*Figure 12. A typical photopulse sequence representing $I_s(t)$ divided into time intervals of width t'.*

The Mini DLS uses 64 different multiples of *t'* to compute *C*(*t'*). For example, the Figure 7 data would yield the following computations of *C*(*t'*):

*t'*=D*t'*: *C*(*t'*)=2×3+3×1+1×4+4×2+2×0+0×3+3×1+...

*t'*=2D*t'*: *C*(*t'*)=2×1+3×4+1×2+4×0+2×3+0×1+...

*t'*=3D*t'*: *C*(*t'*)=2×4+3×2+1×0+4×3+2×1 +...

*t'*=4D*t'*: *C*(*t'*)=2×2+3×0+1×3+4×1 +...

etc.

Figure 13 below shows the autocorrelation plot obtained from DLS analysis of a 91 nm polystyrene latex particle standard.

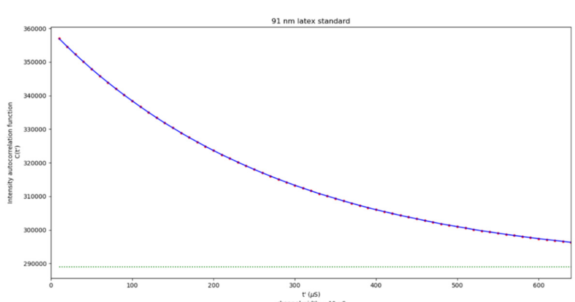



*Figure 13. Autocorrelation function for a 91 nm latex standard.*

### 4.1.5 Gaussian Analysis: Determining Particle Size from Autocorrelation

The relationship shown in Figure 8 can be described by the expression,

$$C(t') = Ae^{-\frac{t'}{\tau}} + B$$

where $A = \langle I_s^2(t)\rangle - \langle I_s(t)\rangle^2$ and $B = \langle I_s(t)\rangle^2$.

This expression can also be written in logarithmic form to produce a straight line of negative slope:

$$\log_e [C(t') - B] = -\frac{1}{\tau} t' + \log_e A$$

The variable $\tau$ is the characteristic decay time constant of the exponential function; $\tau$ characterizes quantitatively the speed with which the autocorrelation function *C*(*t'*) decays toward the long-*t'* limiting value (baseline *B*). In effect, the value of $\tau$ describes the characteristic lifetime, or duration, of a major "bump," or fluctuation, in the scattered intensity. The larger the particles, the slower the diffusivity and resulting fluctuations in $I_s$ and, therefore, the longer the decay time constant $\tau$.

From $\tau$, it is possible to obtain the diffusion coefficient *D* of the particles, using the following equation:

$$D = \frac{1}{\tau} \cdot \frac{1}{2K^2}$$

The constant *K* is dependent on the laser wavelength $\lambda$, the refractive index n of the solvent, and the angle $\theta$ at which scattered light is detected by the PMT detector (usually 90°):

$$K = \frac{4\pi n}{\lambda} \sin\frac{\theta}{2}$$

Once *D* is determined, the computation of *R* is straightforward, using the Stokes-Einstein equation.

However, a mixture of particle sizes will give rise to an autocorrelation function for which the value of $\tau$ is not well-defined. In these situations, the plot of $\log_e [C(t') - B]$ has a slight curvature. The goal of Gaussian analysis is to determine the mean particle size by attempting to fit data to a Gaussian-like distribution of sizes.

To accomplish this goal, the Mini DLS applies a quadratic fit to the plot, finding a function that lies closest to the reduced data points on a least squares basis:

$$\frac{1}{2}\log_e[C(t') - B] \leftrightarrow a_0 + a_1(t') + a_2(t')^2$$

Once the fit is determined, the Mini DLS can calculate a diffusivity distribution and, thus, report an intensity-weighted mean particle size for the distribution.

## 4.2 DLS DATA INTERPRETATION

### 4.2.1 Particle Size Distribution

During measurement, the Mini DLS provides trendline data for the Gaussian distribution onscreen. It does this by reporting the mean particle size and other statistical data over time.

Figure 9 shows an example of how data can be displayed. As seen in the legend, the purple line represents the mean diameter result over time. The shaded purple area represents the area of standard deviation from the mean.

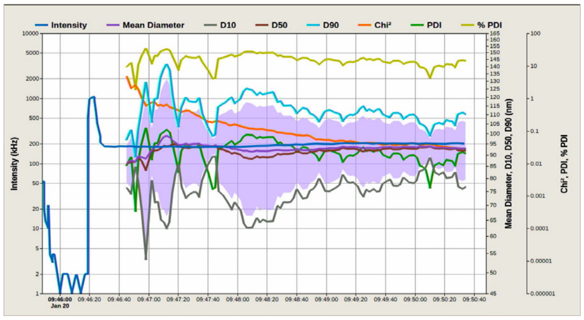



*Figure 14. Example data during a measurement.*

At the end of each measurement, the Mini DLS reports the particle size distribution as part of the RS232 output, according to instrument settings. The output also contains the raw autocorrelation data points from which the Mini DLS calculates the distribution.

### 4.2.2 Statistical Parameters

#### 4.2.2.1 DISTRIBUTION NUMBERS (D10, D50, D90)

In particle size distributions, a cumulative size distribution number (D#) represents the particle size in the cumulative distribution at the given percentile value.

For example, if D50 is reported as 100 nm, this means that 50% of the sample was found to be below 100 nm. D50 is also known as the median in the distribution.

#### 4.2.2.2 CHI² (GOODNESS OF FIT)

The software uses a fit error calculation to quantify the Gaussian fit for the autocorrelation function. Chi² is calculated from a least square fit of order 2. Values closer to 0 represent a good fit for the distribution. The Chi² value may decrease for the first few minutes of measurement as more data is collected. This behavior is seen in Figure 10 (orange trendline).

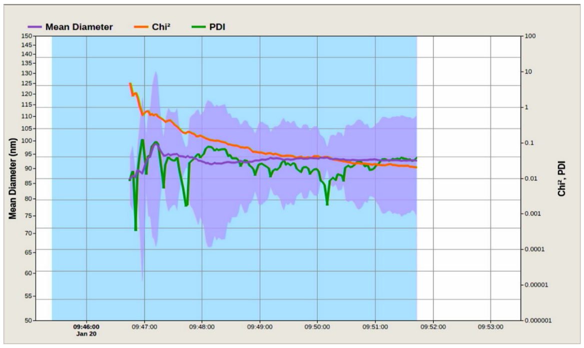



*Figure 15. Example data for mean diameter, Chi², and PDI.*

#### 4.2.2.3 PDI (POLYDISPERSITY INDEX)

The polydispersity index represents the heterogeneity of particle sizes in the sample. A relatively uniform (monodisperse) sample will have a low PDI.

- PDI is calculated from (standard deviation ÷ mean size)².
- % PDI is calculated from the square root of PDI.

## 4.3 AUTODILUTION WITH MINI DLS

### 4.3.1 Sample Concentration for DLS

The DLS sizing technique requires a low sample concentration for several reasons. One reason is that low concentration minimizes particle-particle interactions within the sample; higher concentrations would lead to restricted diffusion of the particles, introducing errors into the measurement. Another reason is that the application of DLS theory assumes that light interacts with only a single particle and then scatters into the detector. As concentration increases, the probability that the scattered light interacts with additional particles before reaching the detector increases. This effect distorts the reported particle size.

If the sample concentration is too low, however, scattering from the particles may be too weak, causing the diluent scattering to have a larger effect on results. The lower concentration limit for the DLS samples is influenced by the scattering properties of the sample.

#### 4.3.2 Mini DLS Autodilution with Intensity Set Point

Most samples are ideally analyzed at a count rate of 300 kHz. To accomplish a certain count rate, the Mini DLS automatically dilutes the concentrated sample.

Two parameters used by the system during a run are (1) the intensity set point and (2) the intensity overshoot factor. Typical values entered for these parameters are 300 kHz and 1.2, respectively. After the concentrated sample enters the system, the Mini DLS adds diluent to the sample loop, monitoring the count rate. The measured count rate first needs to exceed the overshoot intensity (300 kHz × 1.2, or 360 kHz). The purpose of this criteria is to ensure that the sample was detected and was concentrated enough before diluting. When the intensity reaches a value less than the overshoot intensity, the system will stop diluting the sample.

## SECTION 5: OPERATION

### 5.1 OVERVIEW OF BEST PRACTICES

In general,

- **DO** flush the instrument with clean water when first starting up or when shutting down the instrument. Make sure that the intensity reading returns to a clean baseline. Leaving chemicals in the cell can ruin the optical path.
- **DO** make sure that your chemicals are compatible with the wetted components of the system before operation. (See Appendix for component information.)
- **DO** keep the Mini DLS doors closed during operation.
- **DO NOT** allow the flow cell to run dry over extensive periods. Residue from the sample can build up a film on the flow cell. This buildup will affect the baseline noise level and particle sizing capabilities of the detector.
- **DO NOT** run the Mini DLS pumps dry over extensive periods. Running a pump dry for too long will ruin the seals inside.

### 5.2 PREPARING THE MINI DLS STATION

This section provides an overview of preparing the monitoring station. Check the instrument specifications (A-1) for more information on preparing the installation site.

#### 5.2.1 Coolant Line

The Mini DLS requires a steady flow of a coolant such as chilled water to regulate the temperature of the thermoelectric cooler. This water needs to be pumped independently from the Mini DLS according to the specifications in the appendix (A-1).

#### 5.2.2 Diluent, Sample, and Drain Lines

Make sure that the **diluent line** is placed into a source of diluent—i.e., clean water. Each measurement will use this diluent during flush sequences and during autodilution.

The **drain line** must be placed into a suitable drain system or container. The Mini DLS uses pumps to drain liquid out of the system.

The **sample line** must be connected to the sample source. The sample will enter the Mini DLS system by this tubing; the Mini DLS will either pump it in using the sample pump (unpressurized sample) or open valves to allow sample to flow through (pressurized sample).

Be sure that all tubing is free of kinks or blockage.

### 5.3 PREPARING THE SOFTWARE FOR OPERATION (INSTRUMENT SETTINGS)

Before first-time use and after any changes to instrument components, review the instrument settings by tapping the **Settings** shortcut on the left-side menu. The Settings screen has three tabs:

- **General:** for instrument settings, database backup/restore, and upgrades
- **Users:** creating and editing users, assigning user permissions
- **Security:** updating security settings such as password requirements

### 5.3.1 General Instrument Settings

General instrument settings apply to all measurements and are not frequently changed. Be sure to save any changes to this screen by tapping the **Save** button.

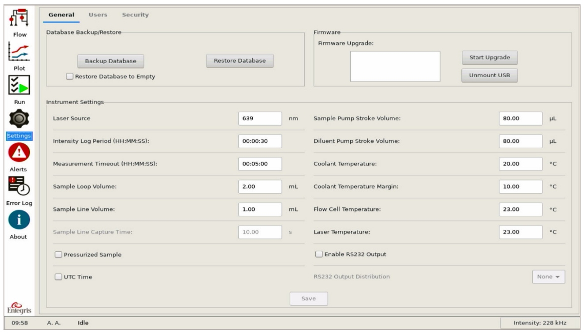



Figure 16. Instrument settings (General) screen.

| INSTRUMENT SETTING | DESCRIPTION |
|---|---|
| **Laser Source** | This is the wavelength of the installed laser. Wavelength is a parameter used in DLS calculations.<br><br>The wavelength value appears on the laser information tag inside of the lower chamber of the unit. |
| **Intensity Log Period** | This value specifies how often an intensity data point is stored and plotted onscreen while the instrument is idle—i.e., not performing a run. |
| **Measurement Timeout** | This is the amount of time the system will attempt to perform a measurement or determine the automatic channel width during a run. If these attempts are not successful by the end of the time limit, the measurement will abort.<br><br>Recommended: at least 2 minutes. |
| **Sample Loop Volume** | This is the volume of the tubing installed between Valve 1 and Valve 2.<br><br>The system uses this setting to determine the sequence for pulling the Sample Volume (Protocol) during a run. If the sample volume is smaller than the size of the loop, a partial fill sequence is employed. |

| INSTRUMENT SETTING | DESCRIPTION |
|---|---|
| **Sample Line Volume** | This is the volume of the tubing installed between the sample source and Valve 1.<br><br>The system pulls this volume at the beginning of a run to ensure that fresh sample reaches the sample loop before the sample is analyzed. |
| **Sample Line Capture Time** | In pressurized sample mode, this parameter is used instead of sample line volume.<br><br>The system opens Valve 1 and Valve 3 for this amount of time at the beginning of a run to ensure that fresh sample reaches the sample loop before the sample is analyzed. |
| **Pressurized Sample** | Enabling this setting configures the instrument for pressurized sample. In this mode, the sample pump will not be used, and flow of the sample will rely on capture time parameters entered in the software. |
| **UTC Time** | When enabled, the bottom-left of the screen will display the UTC time instead of local time. The software will determine UTC time based on the time zone selection (see Time and Date, page 22). |
| **Sample Pump Stroke Volume**<br><br>**Diluent Pump Stroke Volume** | Stroke volume is the volume associated with each turn of the pump and can be adjusted to calibrate pump flow rates if needed. The system uses the stroke volume to decide what pump rotation speed will achieve an entered flow rate. I.e., the higher the entered stroke volume, the lower the actual pump rotation speed, which means a lower actual flow rate.<br><br>Typical stroke volumes for the installed FMI pumps are 80 µL per stroke. |
| **Coolant Temperature** | This is the temperature of the coolant entering the system. The range for this setting is 20 – 30°C. |

| INSTRUMENT SETTING | DESCRIPTION |
|---|---|
| **Coolant Temperature Margin** | If the heat transfer plate temperature reaches a value beyond the Coolant Temperature ± Margin, an alert will trigger the system to abort any operation in progress. While the alert is active, the laser will also shut off. |
| **Flow Cell Temperature** | The system will bring the flow cell and laser, respectively, to these temperatures after saving. |
| **Laser Temperature** | |
| **Enable RS232 Output** | When enabled, measurement data will be directed to RS232 output. (See RS232 Output, page 27). |
| **RS232 Output Distribution** | This is the number of size channels to include in the RS232 output, when enabled. The options are None, 128, 256, 512, or 1024. |

## 5.3.2 Users

To view user settings, tap the **Users** tab at the top of the **Settings** screen.

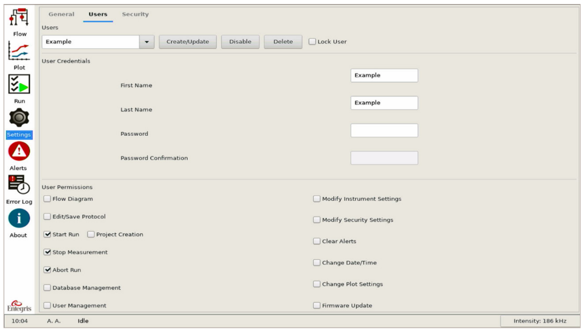



*Figure 17. Users settings.*

### 5.3.2.1 CREATE A USER

To create a user,

1. Enter a new username to the **Users** field. (The username must be unique in the database, including deleted users.)
2. Enter the new user's credentials:
   a. **First Name** and **Last Name**
   b. Enter a **Password** and matching password confirmation. The password must comply with security settings (see Security, page 20).
3. Select the user permissions that will apply to the new user.
4. Tap **Create/Update**.

The user will enter their username and password to log into the software. Site administrators should encourage users to change their password to something more secure once they have logged in for the first time.

### 5.3.2.2 UPDATE A USER

To update a user,

1. Type or select the user from the **Users** field.
2. Edit any field or permission as needed. Entering the current password is not required, but any changes made to the password will be saved after updating.
3. Tap **Create/Update**.

**NOTE: The software does not allow users to edit their own accounts except for changing their password.**

### 5.3.2.3 PERMISSIONS

Each user permission grants the following access when enabled:

| PERMISSION | ACCESS |
|---|---|
| **Flow Diagram** | Operate pumps and valves using the Flow screen controls. |
| **Edit/Save Protocol** | Create and edit protocols on the Run screen. |
| **Start Run** | Start runs using premade protocols and premade projects on the Run screen. |
| **Project Creation** | Create projects on the Run screen when starting a run. |
| **Stop Measurement** | Use the Stop button to stop a measurement manually. (The measurement is recorded as successful.) |
| **Abort Run** | Use the Abort button to stop a run. (The measurement is recorded as aborted.) |
| **Database Management** | Create and restore backup files to the database using the Settings screen—System tab. |

| PERMISSION | ACCESS |
|---|---|
| **User Management** | Edit and save users (except for their own account) on the Settings screen – Users tab.<br><br>This user can only edit user permissions that they themselves have. |
| **Modify Instrument Settings** | Edit and save instrument settings on the Settings screen – Settings tab.<br><br>If an alert occurs, this user can also edit and save the Coolant Temperature from the Alerts screen. |
| **Modify Security Settings** | Edit and save security settings on the Settings screen—Security tab. |
| **Clear Alerts** | Clear alerts on the Alerts screen after they have been resolved. |
| **Change Date/Time** | Open the Time Settings to modify the time zone or date/time. |
| **Change Plot Settings** | Change settings for the Plot screen. |
| **Firmware Update** | Update firmware on the Settings screen – System tab. |

#### 5.3.2.4 DELETING, DISABLING, AND LOCKING ACCOUNTS

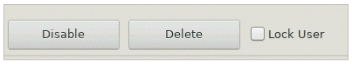



*Figure 18. User management options.*

**Deleting** an account permanently deletes the user. There is no way to undo this action once completed. Once deleted, the software will not permit the creation of a user with the same username again.

**Disabling** or **Locking** an account temporarily disables this user from logging in.

- An account can be **disabled** or **reenabled** from the Users page.
- An account can either be **locked** from the Users page or **locked automatically** when the user attempts to log in too many times with an incorrect password (see Security).
- An account can only be **unlocked** from the Users page.

Users may not delete, disable, enable, lock, or unlock their own accounts.

**NOTICE: The software ensures that administrative accounts can only be disabled if another administrative account exists. Administrative accounts are defined as accounts with all permissions granted.**

**Always use caution when editing any account.**

### 5.3.3 Security

To view security settings, tap the **Security** tab at the top of the **Settings** screen.

Security settings apply to all users. Be sure to save any changes to this screen by tapping the **Save** button.

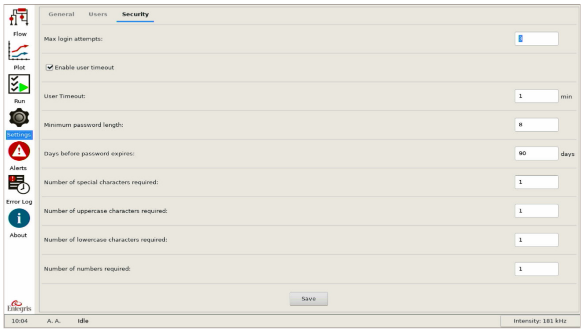



*Figure 19. Security settings.*

| SETTING | DESCRIPTION |
|---|---|
| **Max login attempts** | This is the number of times a user can attempt to log in with an incorrect password before the software automatically locks the account. Once locked, only another user with access to user settings can unlock it. |
| **User timeout** | If no input is detected on the user interface, the software will automatically log out the current user. Any operation/run currently in progress will continue.<br><br>Select (✓) the **Enable User Timeout** checkbox to enable this feature. |
| **Minimum password length** | User passwords will need to be created with at least this number of characters.<br><br>The rule will only apply to new passwords – i.e., creating new users and updating passwords for existing users. |
| **Days before password expires** | Passwords will expire at the end of this period, starting from when the password was created or updated. Users will be prompted to change their expired password upon logging in. |
| **Number of ___ required**<br>• **special characters**<br>• **uppercase characters**<br>• **lowercase characters**<br>• **numbers** | The software will require passwords to have these properties.<br><br>The rule will only apply to new passwords – i.e., creating new users and updating passwords for existing users. |

### 5.3.4 Database and Firmware Upgrade

Database and firmware upgrade tools appear at the top of the **Settings** screen.

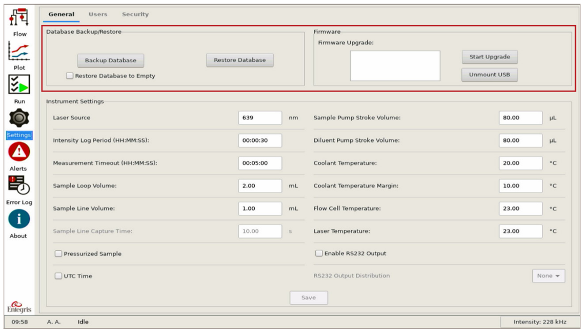



*Figure 20. Database backup/restore and firmware upgrade.*

#### 5.3.4.1 BACKUP DATABASE

The **Backup Database** tool saves an encrypted copy of the database (*.dls) to a connected USB device. This file can later be restored to the Mini DLS if needed.

To create a backup file,

1. Connect a USB storage device to the USB port at the top of the Mini DLS.
2. Choose whether to apply the **Restore Database to empty** option:
    a. Selected (✓): The database will be reset to an empty state after the backup, including run data, settings, and protocol. Users will be retained.
    b. Deselected: The database will remain in its current state after the backup.
3. Tap **Backup Database**.
4. Enter a name and choose a location to save the backup file.
5. Confirm selection. The *.dls file will be saved to the USB device.

#### 5.3.4.2 RESTORE DATABASE

The **Restore Database** tool overwrites the current database with a *.dls file loaded from a connected USB device.

To restore a database file,

1. Connect a USB storage device to the USB port at the top of the Mini DLS. This USB device must contain a *.dls file that was generated using Backup Database.
2. Tap **Restore Database**.
3. Select the backup *.dls file to restore. Tap **OK**.
4. The backup database will replace the existing database: all plot data, users, protocols, and projects.

#### 5.3.4.3 FIRMWARE UPGRADE

The **Firmware Upgrade** tool processes all required upgrades for the firmware and software.

To perform an upgrade,

1. Connect a USB storage device to the USB port at the top of the Mini DLS. This USB device must have the required firmware files.
2. Wait for the software to recognize the firmware files on the USB device. They will appear in **Firmware Upgrade List**.
3. Tap **Start Upgrade**. Wait for the upgrade to complete. The software will automatically restart. DO NOT disconnect any device while the upgrade is in progress.

### 5.3.5 Time and Date

#### 5.3.5.1 MODIFY TIME AND DATE

To open time and date settings, tap the time at the bottom-left of the screen (Figure 16).


*Figure 21. Bottom-left of screen.*

Select the correct **Time Zone** and **Time/Date** as needed. Then, tap **OK** to save (Figure 17).

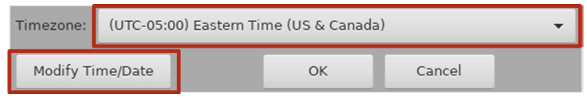



*Figure 22. Time and date settings.*

#### 5.3.5.2 DAYLIGHT SAVINGS

The local time will automatically adjust for daylight savings according to the time zone choice.

## 5.4 OPERATING THE MINI DLS

### 5.4.1 Power On

Check that all cables are correctly connected.

Use the power switch to power on the instrument. The touchscreen will display the Entegris splash screen while the software finds all required devices.

#### 5.4.1.1 CONNECTION TROUBLESHOOTING

If the splash screen shows a lost connection,

1. Use the power switch to power off the instrument.
2. Open the upper chamber door and check that all cables are properly connected.
3. Close the chamber door and switch the power back on.
4. If connection issues persist, contact Entegris.

### 5.4.2 Sign In (Log In)

Only valid users may sign in to access operating features of the Mini DLS software.

To sign in, tap **Login** on the bottom-left corner of the screen. This opens the **Username** and **Password** fields. Enter your username and password and tap **Login** to confirm. (To cancel login, tap **Dismiss**.) Enter admin and admin to login for the first time.

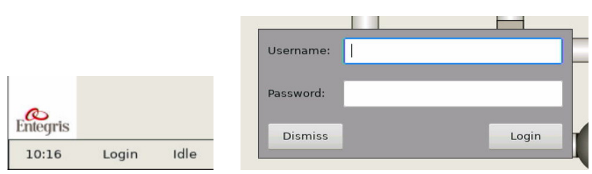



*Figure 23.*

### 5.4.3 System Flush

Starting a run will automatically flush the flow cell with diluent until the intensity reaches the specified target. However, operators may prefer to perform a manual flush after powering up the instrument for diagnostic purposes.

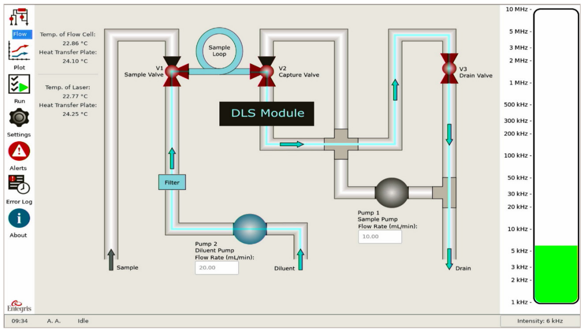



*Figure 24. Flow path of a flush.*

You can start a system flush using the Flow screen:

1. **Set valves:** Toggle each valve to the correct state by tapping the valve icons on the screen (Figure 25).


*Figure 25. V2 directed toward the flow cell.*

2. **Set pump speed:** Set the Diluent Pump speed for flushing—typically, 20 – 30 mL/min.
3. **Start pump:** Tap the Diluent Pump icon to start the pump (Figure 26).

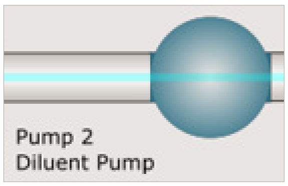



*Figure 26. Pump 2 switched on.*

Monitor the intensity. A typical target background intensity after flushing is below 40 kHz. (This target value may depend on the customer's hardware configuration or cleanliness of water.)

4. **Stop pump:** When the intensity falls below the desired value, stop the pump by tapping the Pump 2 icon.

#### 5.4.3.1 FLOW PATHS

The appendix contains images of each flow path.

### 5.4.4 Start a Run From a Protocol

The Run screen contains selection fields for the parameters of a **run**. The run will use these parameters to flush the system, pull sample, dilute the sample, and perform measurements. The saved set of parameters is a **protocol**. You can create a new protocol before a run, or you can select from already saved protocols.

Starting a run requires the following:

1. Save a new **protocol** or select one from the list. A protocol is the set of measurement parameters shown on this screen.
2. Enter a unique **run** name.
3. Select or enter a **project** name.

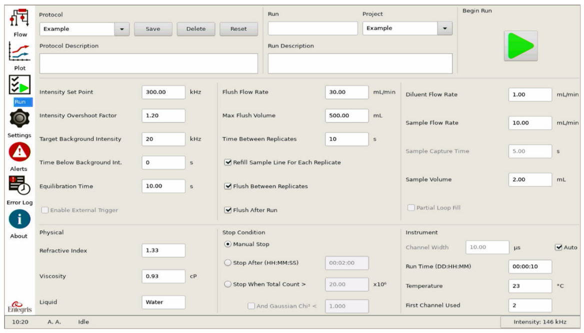


*Figure 27. Run screen.*

A description of each protocol parameter is provided in Table 1 below. For a better understanding of these parameters, see Operating Principles.

Table 1. Protocol Parameter Descriptions

| PARAMETER | DESCRIPTION |
|---|---|
| **Intensity Set Point** | The Intensity Set Point is the desired minimum intensity signal for DLS analysis to occur. The system will dilute the sample until the intensity signal has exceeded the overshoot threshold and then dipped below it. Typically DLS is performed at 300 kHz. |
| **Intensity Overshoot Factor** | The overshoot threshold is equal to Intensity Set Point × Intensity Overshoot Factor. |

| PARAMETER | DESCRIPTION |
|---|---|
| **Target Background Intensity** | The system will flush the flow cell with diluent until the intensity falls below the Target Background Intensity for the specified amount of time. |
| **Time Below Background Int.** | |
| **Equilibration Time** | This is the amount of time the instrument will take to thermally equilibrate after collecting and diluting the sample. |
| **Enable External Trigger** | When an external trigger is installed to the system, this setting enables input from the external device to control the start of each measurement. |
| **Flush Flow Rate** | This is the flow rate of the diluent pump during flush to background, limited to 0.10 to 35 mL/min. |
| **Max Flush Volume** | This is the maximum volume of diluent that the system will use to flush the system before considering the flush failed. At this volume, the replicate will abort. |
| **Time Between Replicates** | This is the time delay from the end of a replicate to the start of the next replicate. |
| **Refill Sample Line For Each Replicate** | This setting enables a fresh pull of sample into the sample line before pulling the sample volume at the beginning of each replicate. |
| **Flush Between Replicates** | This setting enables a flush to background at the beginning of each new replicate. |
| **Flush After Run** | This setting enables a flush to background at the end of the run once the Run Time condition has completed. |
| **Diluent Flow Rate** | This is the flow rate of the diluent pump during autodilution, limited to 0.10 to 35 mL/min. |
| **Sample Flow Rate** | This is the flow rate of the sample pump during sample pull, limited to 0.10 to 35 mL/min. |
| **Sample Capture Time** | In pressurized sample mode, this is the time valves will be open to allow pressurized sample to enter the sample loop. |

| PARAMETER | DESCRIPTION |
|---|---|
| **Sample Volume** | This is the amount of sample to be pumped into the sample loop during sample pull. This value defaults to the sample loop volume (Settings) until changed.<br><br>The Mini DLS can perform measurements whether the sample loop is full or partially full of sample. |
| **Partial Loop Fill** | In pressurized sample mode, this setting allows the user to specify whether the sample loop will only be partially filled during the sample capture. If enabled, the Sample Volume value will be used to calculate the remaining diluent needed to fill the loop.<br><br>(For non-pressurized sample, the software determines this condition automatically by comparing Sample Volume to the Sample Loop Volume setting.) |
| **Refractive Index** | Enter the refractive index of the diluent. This value depends on temperature and laser wavelength. A correct value for refractive index is extremely important for correct calculation. |
| **Viscosity** | Enter the viscosity of the diluent. This value depends highly on temperature. A correct value for viscosity is extremely important for correct calculation. |
| **Liquid** | Enter the name of the liquid. This field is for reference only. |
| **Manual Stop** | Decide upon visual inspection when to stop a measurement. This option allows the replicate to run indefinitely until the user taps the Manual Stop button. (This button is still available while other stop conditions are enabled, however.)<br><br>Once the Manual Stop button has been used, the run will continue to the next replicate, unless the Run Time has also been reached. |
| **Stop After** | Set a time limit for analysis of each replicate.<br><br>Once the sample has been analyzed for this time, the run will continue to the next replicate, unless the Run Time has also been reached. |

| PARAMETER | DESCRIPTION |
|---|---|
| **Stop When Total Count >** | Set a total count to achieve before analysis of a replicate completes. Note that this is a mathematically manipulated pulse count measured by the PMT detector—not a particle count.<br><br>Once the sample has achieved this total count, the run will continue to the next replicate, unless the Run Time has also been reached. |
| **And Gaussian Chi$^2$ <** | This setting can be combined with the "Stop When Total Count >" criterion. If enabled, analysis will stop when Chi$^2$ is below this value – only after also meeting the total count criteria.<br><br>Early in the run, a low Chi$^2$ value may falsely indicate that analysis has found a good Gaussian fit. A count requirement should be set to prevent the Chi$^2$ criteria from ending a measurement before enough data has been collected. |
| **Channel Width** | This is the desired spacing for the autocorrelator channels, in microseconds. |
| **Auto (Channel Width)** | When enabled, the system will automatically determine the optimal channel width when a sample is pulled. |
| **Run Time** | This will be the total time for all measurements during the run.<br><br>At the end of the Run Time, the final replicate will continue to completion based on the chosen Stop Condition. Then, the run will end. |
| **Temperature** | This is the temperature to which the Mini DLS will bring the sample during the run. (What you enter for refractive index and viscosity should depend on this temperature to ensure accuracy of the size result.) |
| **First Channel Used** | This is the first channel used by the autocorrelator, which is set to 2 by default. |

## 5.5 DATA COLLECTION

**NOTICE: The Mini DLS allows any user to view previous data regardless of log in. Only authorized personnel should be permitted to use the Mini DLS in order to protect customer information.**

### 5.5.1 Mini DLS Touchscreen

The Mini DLS touchscreen displays data in real time as it is collected. To view data on the Mini DLS, tap the **Plot** shortcut on the left side of the screen.

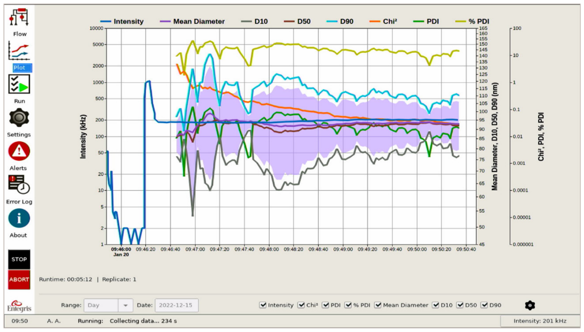


*Figure 28. Plot screen.*

#### 5.5.1.1 LEGEND

The color of the trendlines can be customized in Plot Settings (page 27). By default, the trendlines have the following colors:

- Intensity (dark blue)
- Mean Diameter (purple)
- D10, or custom value (gray)
- D50, or custom value (brown)
- D90, or custom value (light blue)
- Chi² (orange)
- PDI Polydispersity Index (green)
- % PDI (light green)

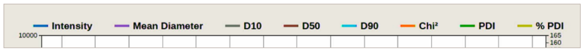



*Figure 29. Plot legend.*

To show or hide each parameter, use the checkboxes at the bottom of the screen.


*Figure 30. Show/hide checkboxes.*

#### 5.5.1.2 PLOT NAVIGATION

The following tools provide control over the display of data:


*Figure 31. Range and date selection.*

- **Range:** Select a range from the following options. (Options greater than one day will display trending data points as averages.)
  - Day
  - Week
  - Month
  - 6 Months
  - Year
- **Date:** The plot will display data for the date selected here. For Day range, this data will be limited to the selected date. For larger ranges, the selected date will act as the end date for the range.
- **Zoom in or out** (Day range only): To zoom in on data at smaller time frames, place two fingers on the plot area and drag outward. To zoom back out, use two fingers to pinch inward.
- **Pan** (Day range only): To pan left or right, place one finger on the plot area and drag left or right. This gesture only works while zoomed in.

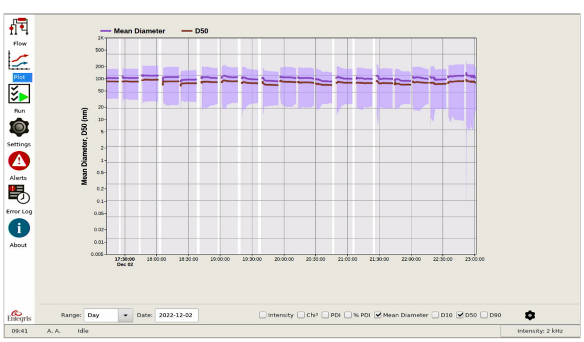



*Figure 32. Day range example, zoomed in on about 6 hours of data.*

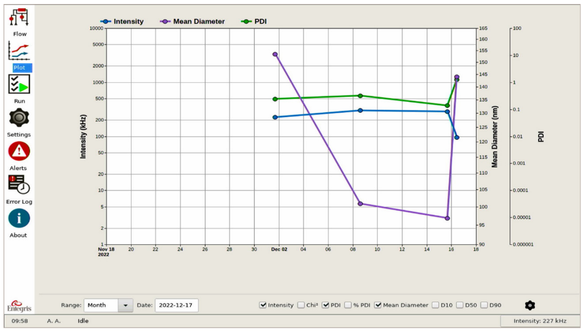



*Figure 33. Month range example, showing four average data points.*

#### 5.5.1.3 PLOT ANNOTATION

Tap on a measurement to open the plot annotation for the measurement (Figure 29). You can either tap within the purple region to view annotation for that point in time, or you can tap just outside the purple region to "snap" to the last data point.

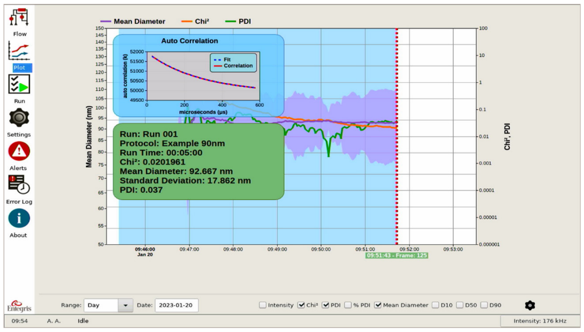



*Figure 34. Plot annotation example, short range.*

For plot ranges greater than one day, tapping near a data point will open information on the averages instead of each individual measurement. (To show or hide what is listed, use the checkboxes at the bottom of the screen.)

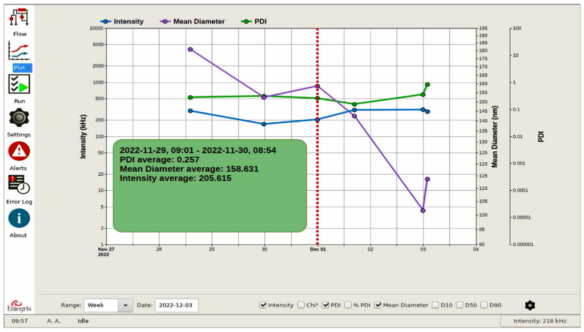



*Figure 35. Plot annotation example, long range.*

#### 5.5.1.4 PLOT SETTINGS

Click the Plot Settings button (arrow, Figure 36) to open the following settings. For more background on interpreting this information, see DLS Data Interpretation, page 16.

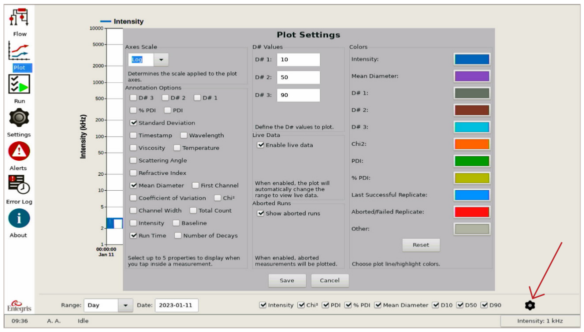



*Figure 36. Plot settings.*

- **Axes Scale:** Select a scale to use for all vertical axes, either Log or Linear.
- **Annotation Options:** Select the properties that will be listed for the measurement in the plot annotation (maximum 5 selections).
  - D# 3, D# 2, D# 1, % PDI, PDI, Standard Deviation, Timestamp, Wavelength, Viscosity, Temperature, Scattering Angle, Refractive Index, Mean Diameter, First Channel, Coefficient of Variation, Chi², Channel Width, Total Count, Intensity, Baseline, Run Time, Number of Decays
- **D# Values:** These numbers represent the particle size in the cumulative distribution at the given percentile value.
  - Using the default settings shown in Figure 31, the plot will report the values for D10, D50, and D90. You can change these values to any number between 1 and 99.
  - These values will be reported as trendlines. They also appear in plot annotation if selected under Annotation Options.
- **Live Data:** If enabled, starting a run will automatically switch the plot to the "live" plot for the run data, which constantly updates as data is collected.
- **Aborted Runs:** Show or hide aborted measurement data from the plot.
- **Colors:** The trendlines will be plotted with the colors shown in each color box. The last three items (Last Successful Replicate, Aborted/Failed Replicate, and Other) are for measurement highlighting. Tapping a color box opens the color selection menu for the item, which allows you to customize colors. To reset all colors back to default, tap Reset.

### 5.5.2 RS232 Output

The Mini DLS sends data as a serial output (RS232) based on the defined RS232 settings. This data includes the following information in a formatted data block. (For the full RS232 schema, see Appendix A-1.)

- status
- time stamp of data output
- project name
- run name
- start time stamp
- username
- protocol
- configuration
- replicate
- intensity
- runtime
- total count
- chi squared
- standard deviation
- mean size
- manual stop condition
- autocorrelation values
- size distribution data (optional, controlled in settings)

If a measurement error causes the measurement to abort, the RS232 output will contain only the status (error, abort), time stamp, and an error description.

If a user aborts a measurement by tapping the Abort button, no data will be transmitted to RS232 output.

#### 5.5.2.1 RS232 OUTPUT SUCCESS EXAMPLE


```
{
 “$schema”: “./rs232_schema.json”,
 “status”: “OK”,
 “timestamp”: “20220622T155843Z”,
 “data”: {
  “baseline”: 930501.0,
  “channel width us”: 8.0,
  “count rate kHz”: 871,
  “autocorrelation function”: [
   985260.4,
   983699.7,
   ... fixed at 64 entries
  ],
  “chi squared”: 0.00015,
  “mean diameter nm”: 90.888,
  “replicate”: 1,
  “runtime s”: 300,
  “standard deviation nm”: 13.838,
  “total count”: 266823500,
  “configuration”: {
   “serial number”: “0000300”,
   “laser wavelength nm”: 635.0
  },
  “project”: {
   “name”: “A Project”
  },
  “protocol”: {
   “name”: “A Protocol”,
   “scattering angle deg”: 90,
   “refractive index”: 1.33,
   “viscosity cP”: 0.933,
   “first channel”: 2,
   “temperature”: 23
  },
  “manual stop”: {
   “value”: false
  },
  “run”: {
   “name”: “A Run”,
   “start timestamp”: “20220223T140134Z”,
   “username”: “admin”
  },
  “distribution”: [
   [
    1.0,
    0.0
   ],
   [
    1.008,
    0.0
   ],
   ... up to 1024 size channels defined in settings
  ]
 }
}
```


#### 5.5.2.2 RS232 OUTPUT ERROR EXAMPLE


```
{
  “status”: “ERROR”,
  “timestamp”: “20210607T143510Z”,
  “error description”: “Error description”
}
```


# SECTION 6: MAINTENANCE AND SERVICE

This section provides instructions on how to perform required maintenance tasks. Administrative end users must establish a policy that permits safe maintenance, inspection, and testing of the Mini DLS to ensure continued safety and functionality.

## 6.1 ROUTINE MAINTENANCE

### 6.1.1 Components Without Maintenance

The following components of the DLS module are either permanently set in manufacturing or rely on principles of physics and do not drift over time. These components and their properties do not require any periodic adjustment or inspection:

- Laser wavelength
- Scattering angle
- Thermoelectric system

### 6.1.2 Frequent Maintenance

#### 6.1.2.1 SYSTEM FLUSH

Flush the system adequately to achieve the same baseline in counts per second as was present before the measurement. A system flush is performed automatically at the start of each run and is essential to the operation of the instrument. For instructions on performing a flush manually, refer to System Flush (page 23).

#### 6.1.2.2 TUBING INSPECTION

The tubing system should be visually inspected on a weekly basis for deterioration and signs of wear. If any part of the tubing is cracked or shows signs of wear, they should be replaced. Inspection can be performed with the system powered off.

#### 6.1.2.3 FILTER INSPECTION

The filter should be checked on a weekly basis for signs of discoloration, leaks, or cracks on the filter body. If any of these signs appear, the filter should be replaced. (See Replacing the Filter, page 30.) Inspection can be performed with the system powered on or off.

**CAUTION: Do not touch moving pump components. Doing so can lead to injury or damage to the equipment.**

### 6.1.3 Periodic Maintenance

#### 6.1.3.1 PERFORMANCE QUALIFICATION (PQ)

A PQ is recommended as a periodic verification that the instrument is performing accurately. DLS systems are typically very stable, so the frequency of PQ should be a risk-based decision. Entegris recommends that a certified PQ take place every 6 – 12 months. Contact Entegris regarding certified validation services, or refer to the technical note at the link below for more information:

System Verification (entegris.com)

#### 6.1.3.2 TOUCHSCREEN PANEL

To clean the touchscreen panel of dirt or residue, apply an ammonia-free glass cleaner to a soft cloth and gently wipe the surface of the panel.

#### 6.1.3.3 CLEANING THE LEAK DETECTOR

If a leak alert has occurred, then liquid (diluent or sample) has contacted the bottom tip of the leak detector and should be cleaned off once the leak has been resolved. To remove residue and ensure accurate leak detection in the future, wipe the level sensor with lint-free cloth.

*Figure 37. Leak detector.*

#### 6.1.3.4 FLOW RATE

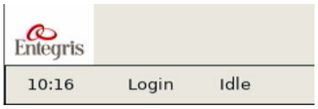
Each pump included with installation has been calibrated and verified in manufacturing. If the flow rate at the customer site is found to be inaccurate, the most likely cause is air in the system. The Mini DLS software provides controls for increasing or decreasing the pump flow rates during a run. If necessary, pump stroke volumes may be adjusted in Settings to calibrate each pump's flow rate.

## 6.2 SERVICE

### 6.2.1 Login with Service Account

Service agents must retrieve a service key from Entegris to log in with the username service. This account has all the privileges of an administrative account. Service personnel may log in with the following steps.

1. Tap **Login** in the bottom-left corner.




2. Type "**service**" into the username field.

3. Tap the password field to open the service password options. You can either manually type the password using the on-screen keyboard or load the password file from USB. Then tap **Accept**.


**NOTE: To prepare a USB flash drive for the Password from USB option, copy a service key to a *.txt file and save this file to the flash drive. Then connect the flash drive to the USB port.**

## 6.2.2 Replacing the Filter

### 6.2.2.1 MATERIALS

0.2 micron filter or equivalent

Disconnect the Old Filter: Draining filter drains all, actuate valves and pumps with no liquid till no water.

1. Drain the system around the filter to minimize the amount of spillage when the filter is disconnected.
2. Power off the Mini DLS.
3. Open the fluidics front panel and locate the installed filter assembly.
4. Place absorbent towels directly under the filter assembly to catch any water that may leak during disassembly or testing.
5. Loosen the two outlet fittings from the top of the filter assembly.
6. Loosen the screws holding the filter bracket to the instrument. Do not remove the screws completely.
7. Push the filter down to slide it out of the bracket.
8. Remove the water inlet from the bottom of the filter.
9. Use a wrench to remove the metal fittings from the top and bottom of the filter.

### 6.2.2.2 INSTALL THE NEW FILTER

1. Remove the new filter from its sealed packaging and wind Teflon tape around the inlet and outlet barbs.
2. Remove and discard the extra white fitting from the white top (outlet side) of the new filter. Wind Teflon tape around the barb.
3. Reconnect all fittings to the new filter, paying attention to the direction of water flow through the filter as indicated by markings. The clear side marked “inlet” is the bottom. Also reattach the filter and filter bracket to the fluidics unit.

**CAUTION: Do not over-tighten fittings or bracket. Over-tightening will case the filter housing to break and render the filter useless.**

### 6.2.2.3 PURGE THE FILTER

1. Power on the instrument and access the system controls.
2. Toggle on the Diluent Pump switch until diluent flows freely out of the outlet tubing. Then, toggle off the Diluent Pump switch.

   **NOTE: If air seems to be trapped in the filter, open the filter bleed cap to let the air out during this process until diluent starts to flow out of the top.**

3. Wipe any visible water from surfaces and remove the towels. Close the front panel.
4. Perform a manual flush to verify that background intensity returns to a steady baseline (see page 23).

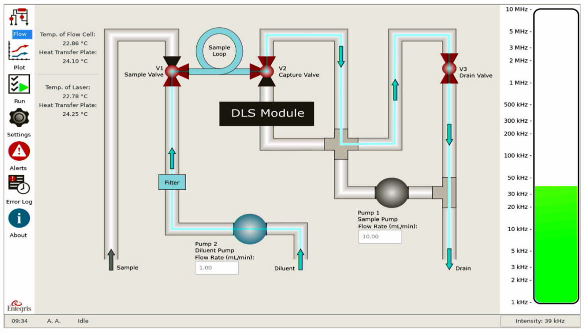



*Figure 38. Flow screen operation.*

## 6.2.3 Other Replacement Parts

To request a replacement part for the Mini DLS system, contact Entegris and provide the serial number and model number of the equipment. Some specifications for the system may be found in Appendix A-1.

⚠ **WARNING: DO NOT replace the detachable power cable with an inadequately rated cable. Doing so could lead to injury or equipment damage.**

### 6.2.4 Aligning the Laser

The need for laser alignment is rare and should be performed by trained Entegris personnel only. Contact Entegris if measurement does not pass a 90 nm PQ check as defined in the Appendix, page 47.

## SECTION 7: TROUBLESHOOTING

### 7.1 GENERAL TROUBLESHOOTING

1. Check that the unit and touchscreen are powered on.
2. Check the software for error messages.
3. Check that you are logged in. (The bottom-left corner of the screen will display user initials if logged in.)
4. Ensure that all cables are properly connected.
5. Check incoming sample and diluent lines. Be sure that tubing is properly connected and inspect connections for leakage.

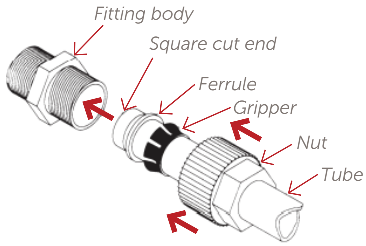
### 7.2 HIGH BACKGROUND INTENSITY

If the instrument is reporting a high intensity with only diluent running through the DLS module, check the following table (Table 2) for possible troubleshooting solutions.

⚠ **CAUTION: Only trained operators should access the lower chamber.**

Table 2. High Background Intensity Troubleshooting

| POSSIBLE CAUSE | SOLUTIONS |
|---|---|
| **Diluent contamination** | • Check for causes of contamination at the source of the diluent.<br>• Check if the water filter needs replacing. Typical filter life is 6 months, but this depends on the cleanliness of the diluent. |
| **Particulates trapped in the tubing leading to the detector** | • Remove and clean or replace the tubing. |
| **Water filter has reached maximum load and is shedding** | • Replace the water filter. Typical filter life is 6 months, but this depends on the cleanliness of the diluent. |
| **Water filter is too new** | • Continue to flush water through the system until the filter has all interior surfaces completely wetted and all air is removed.<br>• If you suspect that air is trapped in the filter, purge the filter using steps on page 30. |
| **Aerated diluent** | • Let diluent sit overnight before using it to allow small air bubbles to percolate out. |
| **Air in the fluidics** | • Examine all tubing for bubbles. You can remove bubbles by tapping on the tubing while the system is flushing to release the bubbles from the sides of the tubing.<br>• Air enters tubing at loose tubing connections. If bubbles are found after a tubing connection, or if liquid seems to flow while pumps are off, be sure that all tube fittings are tight. (Do not overtighten.)<br>• If disconnecting tubing, be cautious of dropping small parts. The connectors that connect tubing at the stainless-steel enclosure rely on small internal ferrules to create a seal. If these small parts are lost, contact Entegris for replacement. |



## 7.3 RS232 OUTPUT

If the RS232 output does not appear, check the following list for possible troubleshooting solutions. If it still does not appear, contact Entegris.

- Check that the serial cable is properly connected to the correct DB9 port, circled below.

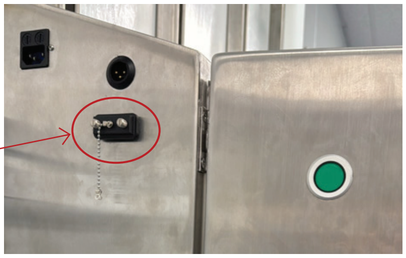



*Figure 39. Serial cable connection.*

- Check that the Instrument Settings show RS232 output as enabled.
- Be sure to use a crossover serial cable – null modem.
- On the separate system receiving the output, check that the baud rate has been set to 115200.

## 7.4 ERROR MESSAGES

To access a history of all error messages, tap the Error Log shortcut button. Errors that occur during operation will appear in the Error Log (Figure 40).

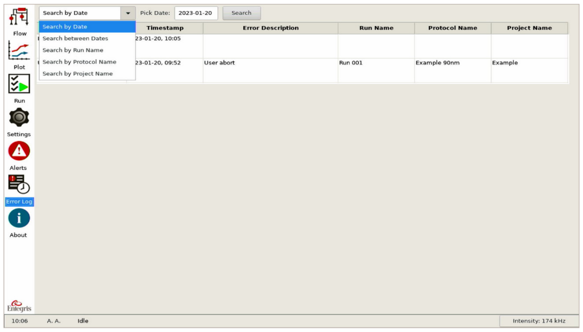



*Figure 40. Error log.*

### 7.4.1 Recoverable Errors

The software may display one of the following error messages in the Error Log when a problem prevents the instrument from performing an automated task. The run may continue until the error is logged three consecutive times, in which case the run will abort.

If the error persists, contact Entegris.

- Start Run
  - Failed to initialize fluidics
  - Failed to save sample to database (create sample record)
  - Failed to save measurement to database (create measurement record)
  - Could not stop measurement (Halt)!
- Set Protocol
  - Could not set protocol to device
  - Could not set temperature
  - Could not clear autocorrelator data
  - Failed to select detector fiber
- Start Flush to Background
  - Could not start flush
  - Over max flush volume limit
  - Could not reach Time Below Threshold
  - Could not stop flush
  - Could not open sample valve
- Pulling sample line volume
  - Could not open sample valve
  - Could not start sample pump to draw sample
  - Could not stop drawing sample
- Pulling sample
  - Could not open sample valve
  - Could not start sample pump to draw sample
  - Could not stop drawing sample
- Waiting to reach temperature
  - Could not stop pump

- Configuring Measurement
  - Could not start correlator
  - System did not update status within configured measurement timeout
- Performing auto dilution
  - Could not start pumps for auto-dilution
  - Auto-dilution timed out
  - Sample too weak for auto-dilution. No injection seen or sample too weak. Raise sensitivity and try again
  - Sample is too concentrated. Inject less material in the future
  - Could not stop pumps during auto-dilution
- Automatically adjusting channel width
  - Failed to set channel width
  - Failed to halt
  - Failed to clear correlator
  - Could not start correlator
  - Failed to start auto channel width measurement
  - Could not reset instrument
  - Could not stop correlator
  - Failed to set channel width
  - System did not complete automatic channel adjustment within configured measurement timeout
- Starting measurement
  - Could no start correlator
  - Could not start measurement
- Collecting data
  - Failed to save data point
  - System did not update within configured measurement timeout
  - Failed to save measurement
- End of run
  - Failed to save sample to database
  - Failed to save run to database

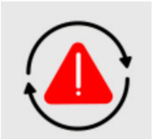
### 7.4.2 Alerts

The following errors are nonrecoverable, meaning that the indicated problem must be resolved before the measurement or operation can continue. Any run in progress will be aborted when the alert triggers.

- Start Run
  - Failed to turn on laser
  - Failed to communicate with laser
  - Failed to turn on TE controller 0
  - Failed to communicate with TE controller 0
  - Failed to turn on TE controller 1
  - Failed to communicate with TE controller 1
  - Failed to read project from database
  - Failed to save run to database (create run record)
  - Failed 3 consecutive measurements
- Configuring Measurement
  - DLS Error encountered
- Critical Alerts
  - Leak detected
  - Heat transfer plate temperature (flow cell)
  - Heat transfer plate temperature (laser)

#### 7.4.2.1 LOCKED ONTO THE ALERTS SCREEN

When an alert is active, the issue must be resolved before resuming operation. To clear the alert onscreen, tap the red alert button that appears onscreen.



# APPENDIX

## A-1 INSTRUMENT SPECIFICATIONS: MINI DLS STAINLESS STEEL

**WARNING: If equipment is used in a manner not specified by Entegris, the protection provided by the equipment may be impaired.**

### CABINET DIMENSIONS

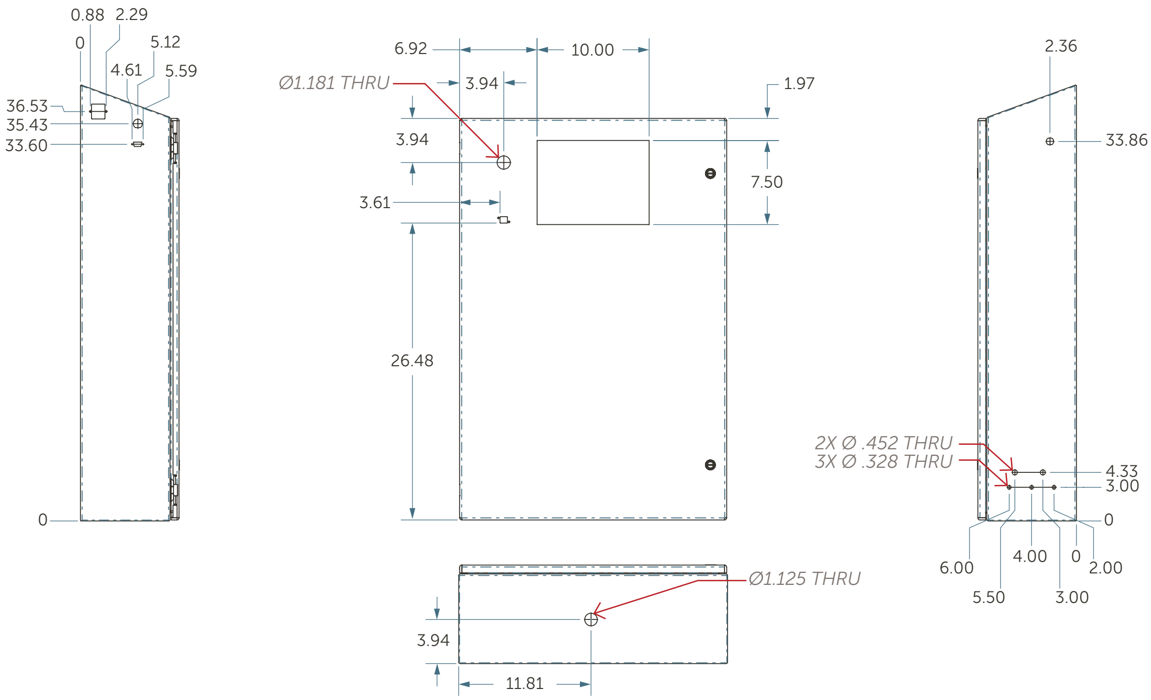



*Figure 41. Physical dimensions.*

### LIST OF SPECIFICATIONS

Dimensions and Installation Considerations

- 38.71″ × 7.70″ × 24.00″
  (98.32 cm × 19.56 cm × 60.96 cm)
  - Clearance Recommendations:
    - 6″ to the left to allow access for power entry port
    - 12″ to the right of unit to allow space for tubing connections
- Weight: 88 lb (~40 kg) not including fluid
- Mounting: requires four ⅜″ bolts
  - Mounting placement up to 3.5 ft above diluent source
  - For hollow wall, use thru bolt with backing on other side of wall.
  - For solid wall, use bolts that are rated for twice the weight of the instrument.
  - Wall construction should also be rated accordingly so that entire mounting assembly can bear four times the weight of the instrument.
- Ventilation:
  - The Mini DLS must be installed under proper ventilation according to site requirements for the sample chemistry.
- Environmental Conditions:
  - Indoor use only
  - Temperature: 40 – 80°F
  - Humidity: 0 – 80%
  - Altitude: 0 – 2000 m
  - Pollution: Class 2 pollution degree rating

### SYSTEM PARAMETERS

- Temperature control range for sample measurement: 4 to 40°C
- Power requirements: 100 – 240 V~, 0.8 A, 50 – 60 Hz. 1 phase. No more than 10% fluctuation.
- Fuses: 5 × 20 mm slo-blo 5 A
- Ingress of protection: IP 65 (finger-safe)

### COOLANT SUPPLY FOR HEAT TRANSFER SYSTEM

- External coolant pump required
- Minimum flow rate of water: 250 mL/min
- Temperature of water: 20 to 30°C
- Fittings: Swagelok SS-400-61

### NITROGEN

- Optional to reduce humid air
- Bulkhead connector ¼″

### MEASUREMENT

- Detection range: 20 nm – 2000 nm hydrodynamic diameter (sample dependent)
- Accuracy for Gaussian mean particle size (90 nm polystyrene latex standard):
  - ±10% of certified mean, accounting for standard deviation:
    Upper size limit = (certified mean + certified standard deviation) × 1.10
    Lower size limit = (certified mean – certified standard deviation) × 0.90
  - Repeatability ±5% from average of four consecutive measurements
- Detector type: Photomultiplier tube (PMT)
- Laser wavelength accuracy: ±10%
- Measurement angle: 90°
- Calibration not required.

### DILUENT AND SAMPLE LINES

- Fittings: Swagelok SS-200-61
- Provided tubing, inner diameter: 0.062 inch
- Range: Up to 25 ft (7.6 m) tubing, or 15 mL
- Pressure: Up to 20 psig
- Pump flow rates: Up to 35 mL/min

### TOUCHSCREEN

- Panel: LED/LCD
- Active Area Diagonal: 10.1 inches
- Display Active Area: 217.0 mm × 135.6 mm
- No contact pressure required.
- No need for touch calibration.

### EXTERNAL DATA OUTPUT

- Requires serial connection to external computer
- Raw data transmitted using JSON format via RS232 output (one direction only)
- Serial cable must be a crossover cable.
- RS232 baud rate: 115200

### EXTERNAL TRIGGER

- 3.3 V input

## WETTED MATERIAL COMPATIBILITY

**WARNING: The Mini DLS has not been assessed and certified for use with hazardous chemistries. The machine has been certified to use with non-hazardous chemistries and chemistries that are compatible when mixed. Customers MUST evaluate chemical compatibility of their liquids with all wetted components of the system as well as components that may come in contact with liquids in the event of a leak.**

Entegris uses a polystyrene latex standard (90 nm) to qualify the instrument (see installation instructions). This standard, diluted in 10 mM NaCl, is chemically compatible with the wetted materials of the Mini DLS and is nonreactive with water during operation:

- Thermofisher Polymer Microsphere Suspension
  - Polystyrene (CAS no. 9003-53-6) or Polystyrene divinylbenzene (CAS no. 9003-70-7) at 0.1 – 10%
  - Sodium azide (<0.09)-CAS no.: 26628-22-8

The wetted materials included with a standard Mini DLS Stainless Steel system are listed below.

| COMPONENT | MATERIAL | LIQUIDS IN CONTACT |
|---|---|---|
| **Tubing assemblies** | 316 Stainless Steel<br>PEEK<br>PFA<br>Delrin | Coolant, sample, and diluent |
| **FMI Pumps** | Ceramic<br>PVDF | Sample and diluent |
| **Valves** | PTFE<br>PPS | Sample and diluent |
| **Flow cell assembly** | Viton Fluoroelastomer<br>ES Quartz | Sample and diluent |
| **Filter** | Hydrophilic polyether sulfone<br>Polypropylene | Diluent |
| **Water block** | 6061T6 Aluminum (black anodized finish per mil-a-8625 Class II)<br>Viton Fluoroelastomer | Coolant |
| **Leak sensor** | Polysulfone | (Spillage event)<br>Coolant, sample, or diluent |
| **Steel enclosure** | 316 Stainless Steel | |

*Note: This document is not controlled when printed. For any questions concerning customizations or updates to these specifications, contact Entegris.*

## FLUIDICS DIAGRAM AND FLOW PATHS

### Fluidics Diagram

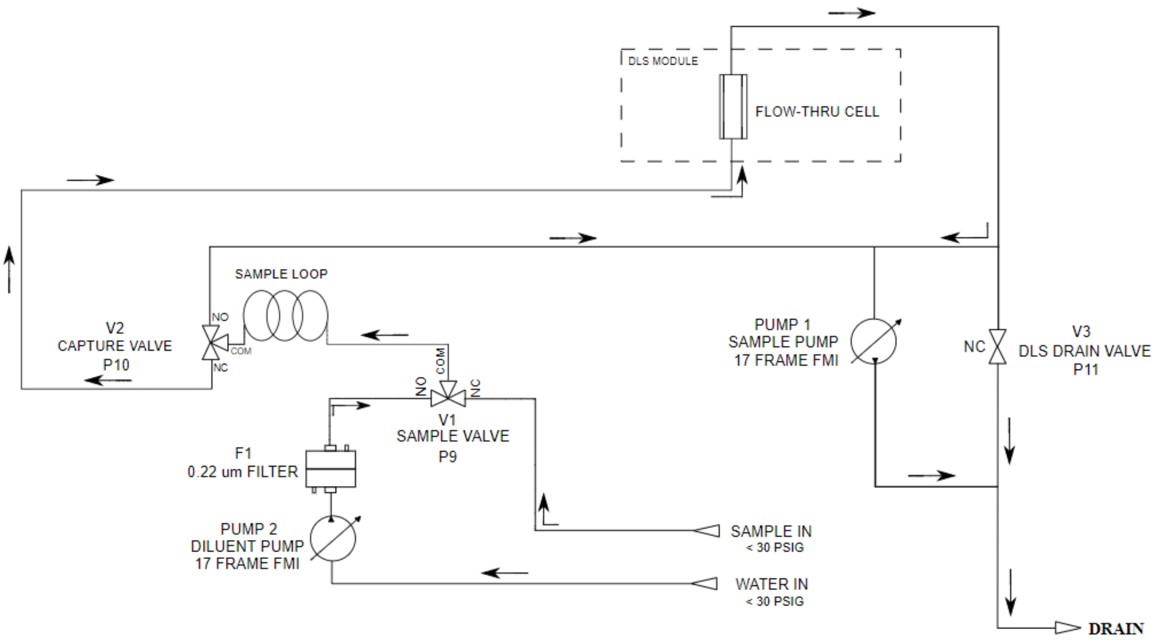



*Figure 42. Fluidics Diagram.*

### Flow Path: Pump 1 Bypass, "Sample Pull"

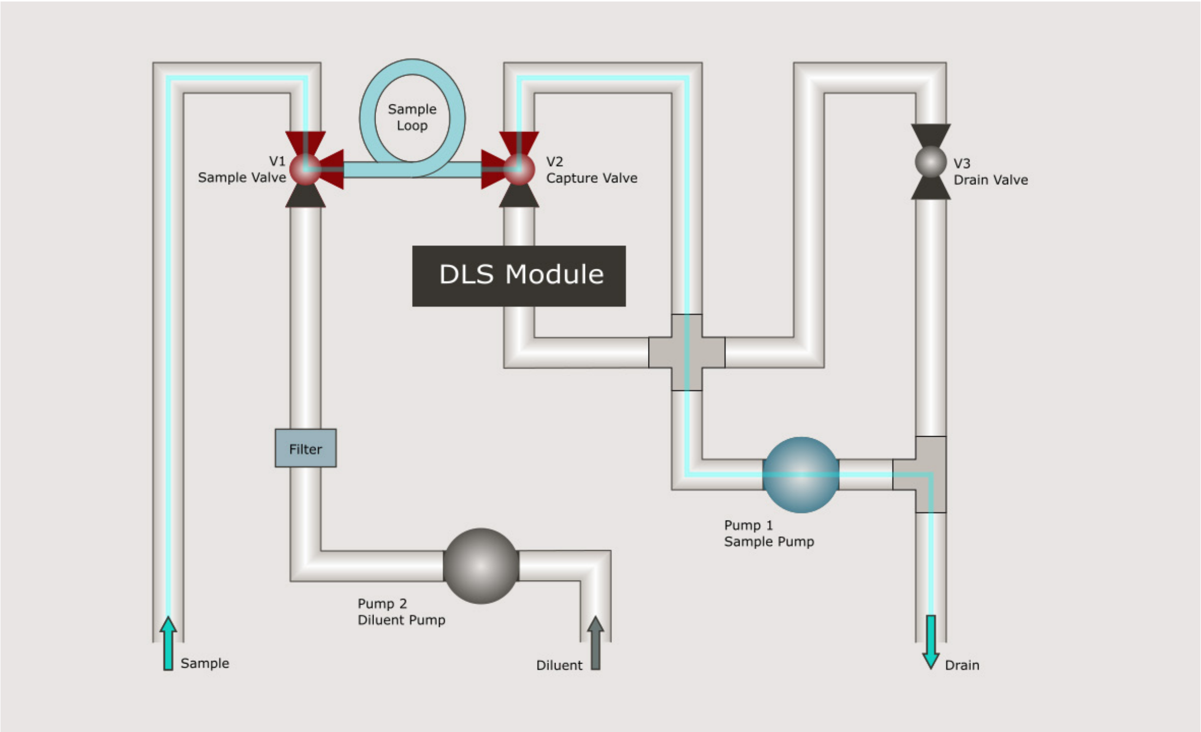



*Figure 43. Sample pull.*

Flow Path: Pump 1 Flow Cell (not used)

**CAUTION: Using overly concentrated samples to manually check intensity levels with this flow path can cause damage to the PMT detector. This flow path is not used in automated measurement and is only achievable through manual use of the Flow Screen. Only trained personnel should operate the Flow Screen.**

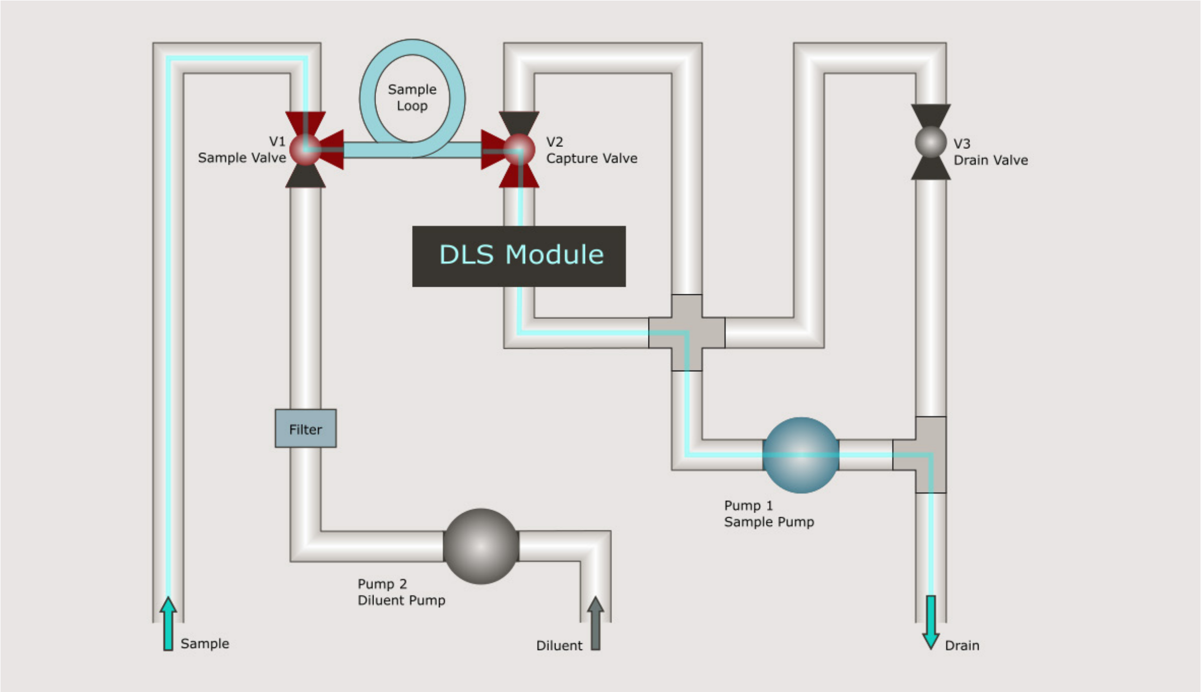



*Figure 44. Sample pull.*

Flow Path: Pump 2 Bypass, "Partial Fill"

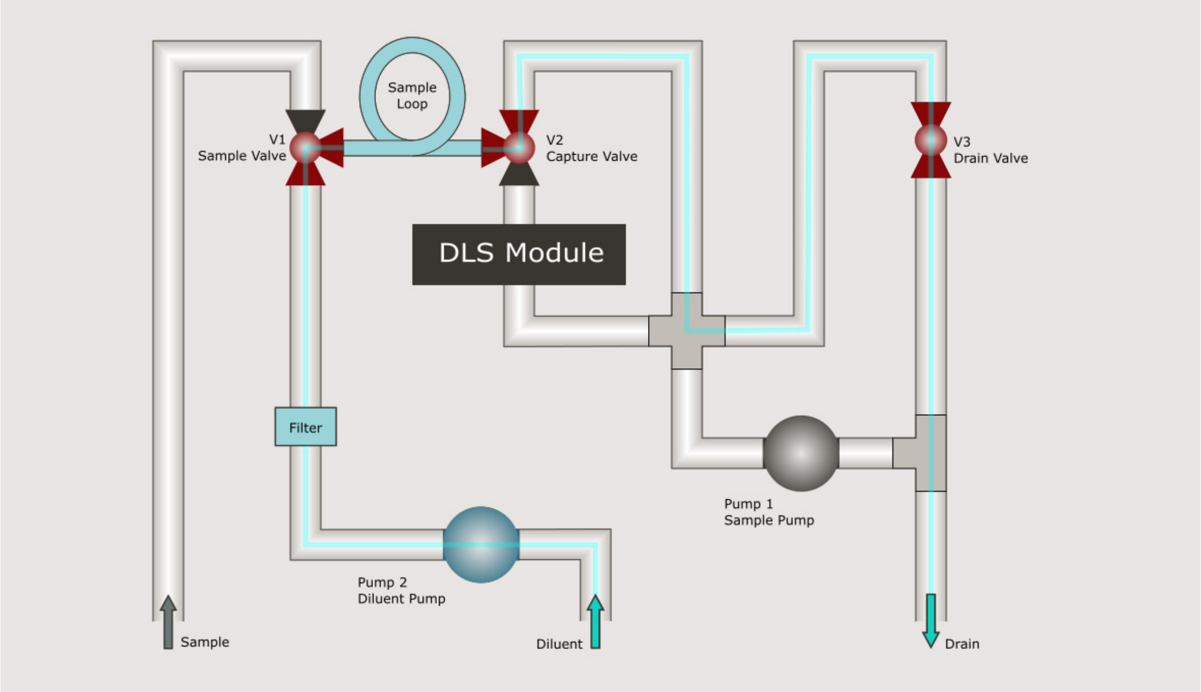



*Figure 45. Partial fill.*

Flow Path: Pump 2 Flow Cell, "Flush"

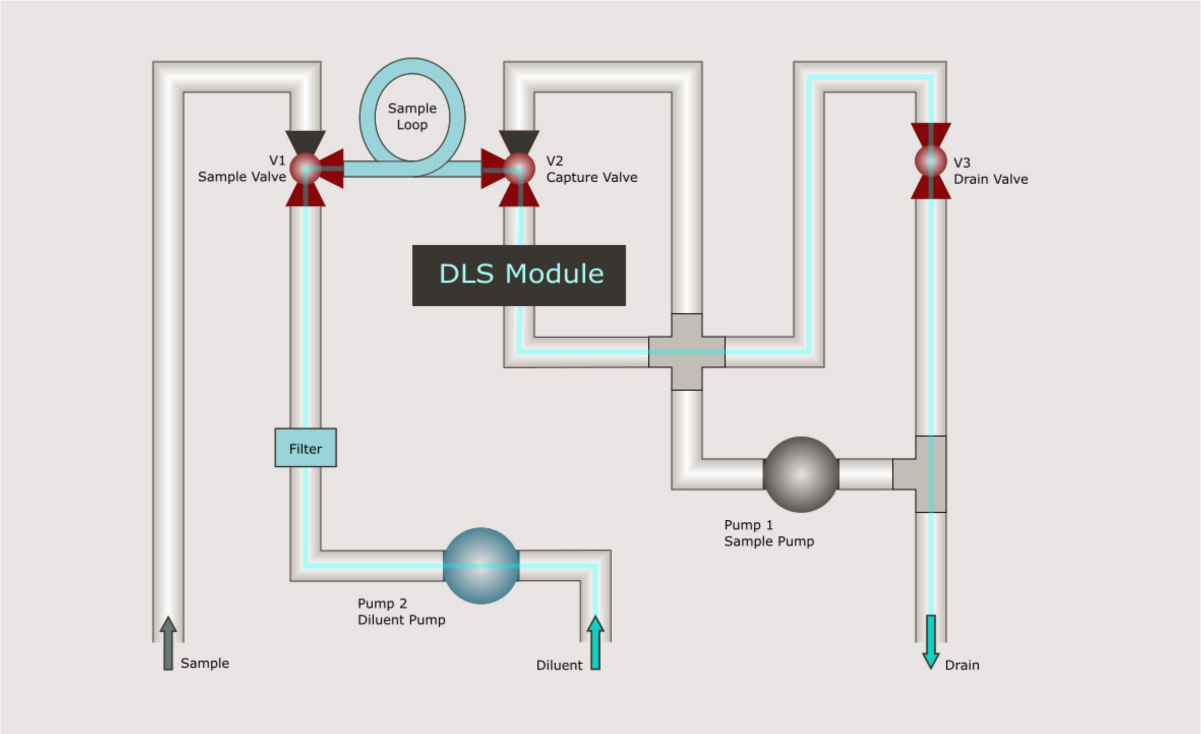



*Figure 46. Flush.*

Flow Path: Pressurized Sample Bypass

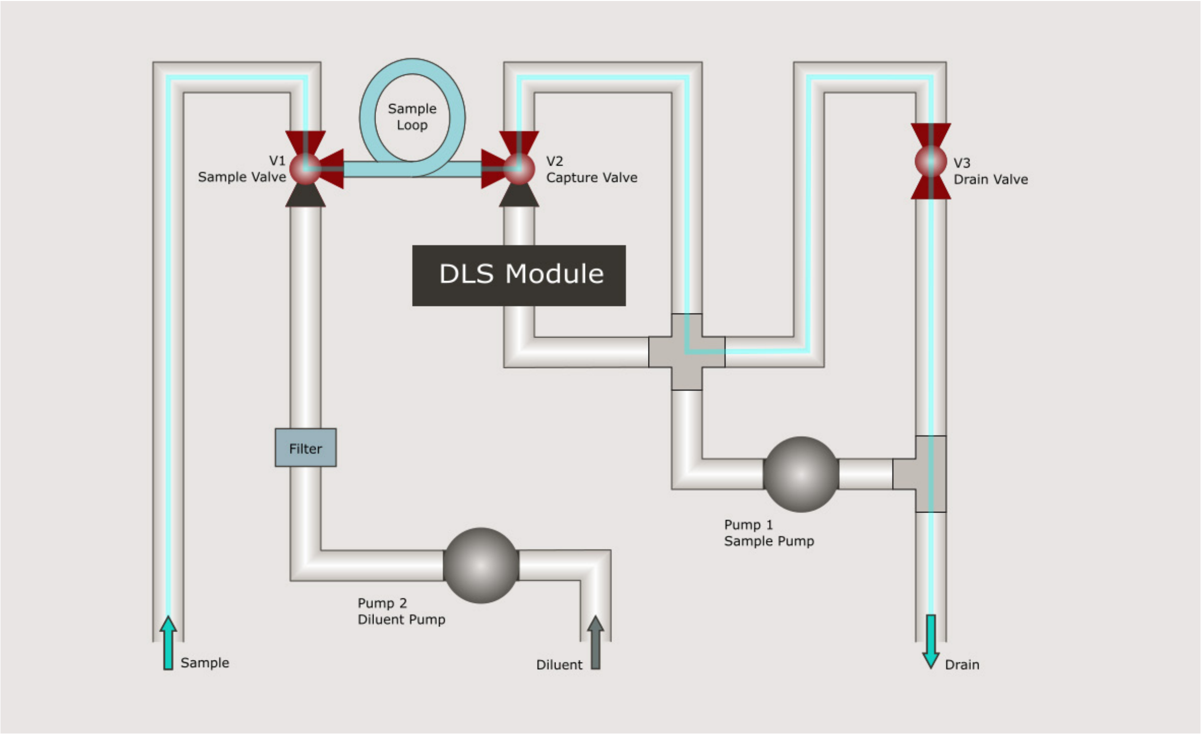



*Figure 47. Sample bypass.*

Flow Path: Pressurized Sample Flow Cell (not used)

**CAUTION: Using overly concentrated samples to manually check intensity levels with this flow path can cause damage to the PMT detector. This flow path is not used in automated measurement and is only achievable through manual use of the Flow Screen. Only trained personnel should operate the Flow Screen.**

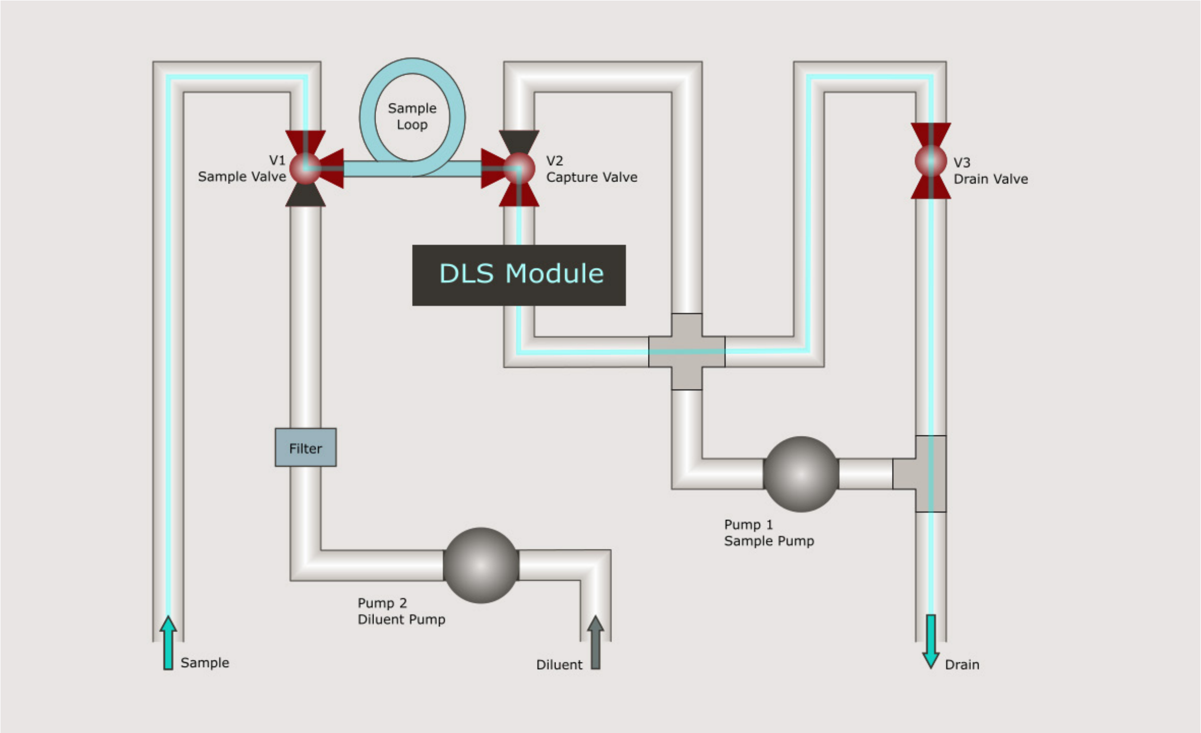



*Figure 48. Sample flow cell.*

## RS232 OUTPUT SCHEMA

When configured, each measurement will output the results via RS232 with the following schema:


```
{
  "title": "Mini DLS RS232",
  "$schema": "http://json-schema.org/draft-07/
schema",
  "type": "object",
  "timestamp": {
    "type": "string",
    "pattern": "^[0-9]{4}[0-9]{2}[0-9]{2}T[0-9]{2}[0-9]{2}
[0-9]{2}Z$",
    "$comment": "UTC date and time in ISO 8601 short
datetime format. YYYYMMDD'T'hhmmss'Z'",
    "examples": [
      "20210607T152139Z"
    ]
  },
  "properties": {
    "$schema": true,
    "status": {
      "enum": [
        "OK",
        "ERROR"
      ]
    },
    "timestamp": {
      "$ref": "#/timestamp",
      "$comment": "Timestamp of the output event"
    },
    "error description": {
      "type": "string"
    },
    "data": {
      "type": "object",
      "properties": {
        "project": {
          "type": "object",
          "$comment": "A collection of runs",
          "properties": {
            "name": {
              "type": "string",
              "$comment": "The name of the project"
            }
          },
          "required": [
            "name"
          ],
          "additionalProperties": false
        },
        "run": {
          "type": "object",
          "$comment": "A collection of measurements",
          "properties": {
            "name": {
              "type": "string",
              "$comment": "Name of the run"
            },
            "start timestamp": {
              "$ref": "#/timestamp",
              "$comment": "Timestamp of the run start"
            },
            "username": {
              "type": "string",
              "$comment": "User who started the run"
            }
          },
          "required": [
            "name",
            "start timestamp",
            "username"
          ],
          "additionalProperties": false
        },
        "protocol": {
          "type": "object",
          "$comment": "Measurement protototocol",
          "properties": {
            "name": {
              "type": "string"
            },
            "scattering angle deg": {
              "type": "number",
              "minimum": 0,
              "maximum": 90,
              "$comment": "Scattering angle in degrees.
Usually 90",
              "$example": 90
            },
            "refractive index": {
              "type": "number",
              "minimum": 1,
              "maximum": 3,
              "$comment": "Refractive index of the fluid",
              "$example": 1.33
            },
            "temperature": {
              "type": "number",
              "minimum": 4,
```

“maximum”: 40,
“$comment”: “Temperature of the fluid”,
“$example”: 23
},
“viscosity cP”: {
“type”: “number”,
“exclusiveMinimum”: 0,
“$comment”: “Viscosity of the fluid in
centiPoise”,
“$example”: 0.933
},
“first channel”: {
“type”: “integer”,
“minimum”: 0,
“maximum”: 4,
“$comment”: “First channel considered for
analysis”,
“$example”: 2
}
},
“required”: [
“name”,
“scattering angle deg”,
“refractive index”,
“viscosity cP”,
“temperature”,
“first channel”
],
“additionalProperties”: false
},
“configuration”: {
“type”: “object”,
“$comment”: “Instrument configuration”,
“properties”: {
“serial number”: {
“type”: “string”,
“$comment”: “Serial number of the
instrument”
},
“laser wavelength nm”: {
“type”: “number”,
“minimum”: 0,
“$comment”: “Wavelength of the laser in
nanometers”,
“$example”: 639
}
},
“required”: [
“serial number”,
“laser wavelength nm”
],
“additionalProperties”: false
},
“replicate”: {
“type”: “integer”,
“minimum”: 1,
“$comment”: “One indexed count of how many
measurements have occured in this run, including this
one”,
“$example”: 1
},
“count rate kHz”: {
“type”: “integer”,
“minimum”: 0,
“$example”: 300,
“$comment”: “Scattering count rate, kilohertz”
},
“runtime s”: {
“type”: “integer”,
“$comment”: “Measurement run time, seconds”,
“minimum”: 0
},
“total count”: {
“type”: “integer”,
“$comment”: “Total count. Use a long format
integer”,
“minimum”: 0
},
“baseline”: {
“type”: “number”,
“$comment”: “Correlation function baseline”,
“minimum”: 0
},
“channel width us”: {
“type”: “number”,
“$comment”: “Channel width, microseconds”,
“minimum”: 0
},
“chi squared”: {
“type”: “number”,
“$comment”: “Chi Squared”,
“minimum”: 0
},
“standard deviation nm”: {
“type”: “number”,
“$comment”: “Intensity weighted standard
deviation, nanometers”,
“minimum”: 0
},
“mean diameter nm”: {

```
      “type”: “number”,
      “$comment”: “Intensity weighted mean diameter, nanometers”,
      “minimum”: 0
    },
    “manual stop”: {
      “type”: “object”,
      “$comment”: “Indicates whether the measurement was stopped manually or automatically”,
      “properties”: {
        “value”: {
          “type”: “boolean”,
          “$comment”: “True if stopped manually, false if stopped automatically”
        },
        “username”: {
          “type”: “string”,
          “$comment”: “If stopped manually, the name of the user that stopped the measurement. Not present if stopped automatically”
        }
      },
      “required”: [
        “value”
      ],
      “if”: {
        “properties”: {
          “value”: {
            “const”: false
          }
        }
      },
      “then”: {
        “not”: {
          “required”: [
            “username”
          ]
        }
      },
      “additionalProperties”: false
    },
    “autocorrelation function”: {
      “type”: “array”,
      “$comment”: “Contains the raw autocorrelation data. Use long format floating point numbers”,
      “items”: {
        “type”: “number”,
        “minimum”: 0
      },
      “minItems”: 64,
      “maxItems”: 64
    },
    “distribution”: {
      “type”: “array”,
      “$comment”: “Array of histogram sizes and values. Up to 1024 channels. Optional”,
      “items”: {
        “type”: “array”,
        “items”: [
          {
            “type”: “number”,
            “$comment”: “Bin diameter, nanometers”,
            “minimum”: 1,
            “maximum”: 5000
          },
          {
            “type”: “number”,
            “$comment”: “Normalized bin value, (0,1]”,
            “minimum”: 0,
            “maximum”: 1
          }
        ],
        “minItems”: 2,
        “maxItems”: 2
      },
      “minItems”: 8,
      “maxItems”: 1024
    }
  },
  “required”: [
    “project”,
    “run”,
    “protocol”,
    “configuration”,
    “replicate”,
    “count rate kHz”,
    “channel width us”,
    “baseline”,
    “runtime s”,
    “total count”,
    “chi squared”,
    “standard deviation nm”,
    “mean diameter nm”,
    “manual stop”,
    “autocorrelation function”
  ],
  “additionalProperties”: false
 }
},
“required”: [
```

```
    "timestamp",
    "status"
  ],
  "if": {
    "$comment": "If 'ERROR', the data section will not
be included",
    "properties": {
      "status": {
        "const": "OK"
      }
    }
  },
  "then": {
    "allOf": [
      {
        "required": [
          "data"
        ]
      },
      {
        "not": {
          "required": [
            "error description"
          ]
        }
      }
    ]
  },
  "else": {
    "allOf": [
      {
        "required": [
          "error description"
        ]
      },
      {
        "not": {
          "required": [
            "data"
          ]
        }
      }
    ]
  },
  "additionalProperties": false
}
```

## A-2 HARDWARE INSTALLATION

Upon delivery of the Mini DLS system, confirm that all items listed on the packing slip were included in the package. Do not proceed with hardware installation until all components are received. Call Entegris at (727) 846 0866 if the packing slip or any component is missing.

Packaged contents may vary by customer needs. In general, you will find the following components with your delivery:

### MAJOR INSTRUMENT COMPONENTS

- DLS unit with integrated touchscreen software installed
- User manual

### PERFORMANCE QUALIFICATION COMPONENTS

- Bottle of 90 nm latex particle size standard (~1% solids)
- Certificate of Analysis for standard

### INSTALLATION STEPS

Installation of the Mini DLS involves the following main steps. Before installing, review all instrument specifications in Appendix A-1 to ensure that requirements are met.

**CAUTION: To prevent injury or damage to the equipment, DO NOT open any of the cabinet doors or remove other secured materials from the unit until the unit is properly mounted.**

1. Unpack and Mount
   a. Remove the Mini DLS unit from the package.
   b. Mount the Mini DLS unit upright using two ⅜″-16 UNC 2A bolts (not provided).

      Team lift required

   c. Do not to position the equipment so that it is difficult to operate the disconnecting device.
   d. Use a flathead key to open the cabinet doors.
   e. Inspect the interior and exterior of the equipment and remove any extra packaging found.

**NOTICE:**
**W = Equipment weight = 88 lbf**
**NB = Number of mounting bolts = 4**
**LB = Bolt proof load = 4,250 lbf**
**PBOLT = Stress load on 1 bolt = 22 lbf**

$$Safety\ Factor = \frac{Lb}{Pbolt} = \frac{4250\ lbf}{22\ lbf} = 193.18$$

**NOTICE: When connecting exterior tubing to the enclosure, use caution not to lose the small transparent ferrules that fit within the connectors. These ferrules must be positioned properly to prevent leaks.**

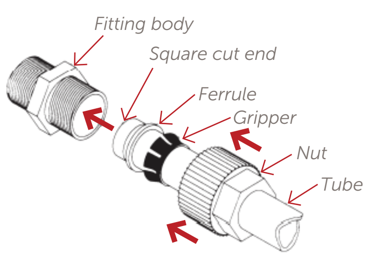



*Figure 49. Tubing connections.*

2. Configure and prepare instrument for operation
   a. Connect BOTH the diluent line and sample line to a supply of clean water. (Sample line must be checked for leaks after installation before connecting to the customer sample.)
   b. Connect the drain line to the site's drain system. Fluid sent to drain will contain both the sample and water.
   c. Connect cables to the top panel. At minimum for operation, the power cable must be connected to the power entry module, circled below.

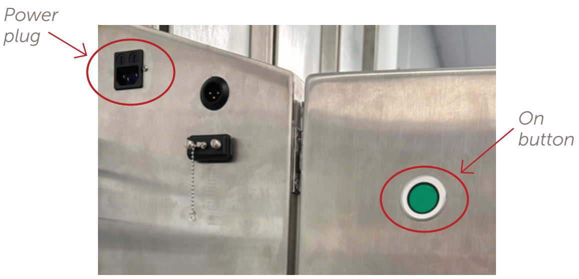



*Figure 50. Power plug and on button.*

   d. Use the push button switch to power on the instrument. The touchscreen panel will automatically turn on and open to the software.

e. Log into the software with a username and password. For first-time use: username and password will both be "admin" to log in.

f. Configure instrument settings and tap Save. (See Preparing the Software for Operation, page 17.)

**CAUTION: To avoid injury, never touch rotating components of pumps.**

g. Using Flow Screen controls, purge air from the water filter. Do this by running the diluent pump with the filter bleed cap open, according to filter service instructions (Purge the Filter, page 30). Monitor tubing connections for leaks (see next step).

h. Using the Flow Screen controls to activate pumps and valves, check each flow path for leaks.

- If liquid drips out of any fitting connection, turn off the pump immediately and clean any spills. Hand-tighten the fitting and use a cloth to wipe any spills. (Stainless steel fittings may need to be tightened with a wrench.)
- Loose fittings may also cause bubbles to appear in the tubing. Be sure to inspect all tubing for bubbles and tighten fittings as needed.

i. Connect the sample line to the customer sample to be analyzed. Confirm that the diluent line is connected to a supply of water and the drain line remains in the drain.

j. Close both the upper and lower cabinet doors and lock with the provided hex key.

## PERFORMANCE TESTING (PERFORMANCE QUALIFICATION, PQ)

**For certified qualification services, contact Entegris.** The purpose of performance testing is to demonstrate the accuracy and repeatability criteria described in appendix A-1. The Mini DLS was designed to be validated with a NIST-traceable 90 nm polystyrene latex particle sizing standard.

To prepare a PQ sample for the Mini DLS:

1. Prepare a 10 mM NaCl solution by adding 0.5844 g of NaCl to 1 L of filtered, distilled water.
2. Mix the bottle of 90 nm standard by gently inverting the bottle 10 times.
3. To 1.5 mL of the standard, add 23.5 mL of 10 mM NaCl for a total volume of 25 mL. (Adjust volume as needed to fill the sample line during the run.)

Settings and protocol may vary according to tubing lengths and DLS module configuration for the customer's product. Be sure to configure instrument settings accordingly.

To perform the test, follow the instructions below:

1. Check that the sample tubing has been secured within the 90 nm sample (prepared above).
2. Confirm instrument settings are correct.
3. Connect RS232 serial output to a terminal or laboratory management system, if required by the customer. Data cannot currently be exported from past measurements—only viewed on the touch-screen. The RS232 output can be utilized to save a log of measurements performed in this session.
4. Create and save a protocol so that at least four measurements will be included in the run. Then start the run.
5. Verify that measurements have demonstrated product criteria.

The flowing table is supplied with the system to verify particle sizing accuracy of the 90 nm PSL standard.

Enter the mean diameter and uncertainty values from the certificate of analysis in cells A11 and B11 in the spreadsheet below. Enter the intensity mean diameters for the four runs in cells B14 – 17. Enter the sample ID, date, and initial of the operator in the appropriate cells. All results must PASS for the system to be verified at installation.

## PASS/FAIL CRITERIA

**Particle Size Accuracy Table**

Double-click the spreadsheet to begin entering data. Instructions:

1. Enter the certified mean of the standard in cell A11. (All sizes must be entered in nanometers.)
2. Enter the certified uncertainty in cell B11.
3. The minimum (C11) and maximum (D11) are automatically calculated.
4. Enter the Gaussian intensity-weighted mean size results from each sample into cells B14-B17.
5. Enter the sample ID and date for each sample, and enter initials of the user validating the system for each result.
6. All measured sizes must be within +/- 10% of the expanded mean in order to pass. Cells C14-C17 will report Pass/Fail results automatically.

| Standard Mean = (x) | Standard Uncertainty = (u) | Min = (x-u)*0.9 | Max = (x+u)*1.1 |
|---|---|---|---|
| | | 0.0 | 0.0 |

| | Measured Size (y) | Criteria: Min ≤ y ≤ Max | Sample ID | Date | Initials |
|---|---|---|---|---|---|
| Sample Run 1 | | Data required. | | | |
| Sample Run 2 | | Data required. | | | |
| Sample Run 3 | | Data required. | | | |
| Sample Run 4 | | Data required. | | | |

## A-3 DEFINITIONS

| | |
|---|---|
| **Autocorrelation** | The function that measures the degree to which a signal is related to time-delayed copies of itself |
| **Background intensity** | (See Intensity)<br><br>The intensity achieved when only clean diluent flows through the flow cell; a baseline to target for flushing the system |
| **Deconvolution** | The transformation from the autocorrelation function to a particle size distribution |
| **Diluent** | Liquid for diluting the sample; usually water<br><br>This text may refer to diluent, solvent, and water interchangeably in reference to the liquid being pulled through the diluent line. When choosing a diluent other than water, be sure to check compatibility with the instrument. |
| **DLS** | Dynamic light scattering (DLS) is the method of particle sizing used by the mini DLS. |
| **Intensity** | The rate at which scattered light from the particles is detected by the PMT, usually reported in kHz |
| **Measurement** | (See Run, Replicate)<br><br>This manual uses "measurement" to refer to the time taken for particle size determination with a single unit of sample. The instrument may perform multiple measurements by redrawing sample for each measurement. Multiple measurements within a run are also called "replicates." |
| **Polydispersity index (PDI)** | Describes the relative variance of the gaussian distribution<br><br>Values range from 0 (perfectly uniform particle size distribution) to 1 (polydisperse).<br><br>Percent polydispersity is reported as "% PDI" in the software and is calculated by the square root of the PDI. |
| **PMT** | Photomultiplier tubes (PMT); an extremely sensitive light detector |
| **Refractive index** | Property of a material that describes how fast light travels through the material, depending on the wavelength of the light and the temperature of the material<br><br>For the mini DLS, the relevant refractive index (Ri) is that of the diluent. |
| **Run** | (See Measurement, Replicate)<br><br>This manual uses "run" to refer to the time taken to complete all measurements/replicates that are performed with the Run name entered at the top of the Run screen before tapping the button to start. The instrument performs the run continuously until it is programmed to end the run. |
| **Replicate** | (See Measurement, Run)<br><br>This manual uses "replicate" to refer to multiple measurements within a run. In the software, a replicate also includes the steps that are programmed to be performed in preparation for the measurement (e.G. Flushing the system, autodilution). |
| **Scattering** | Refers to the Rayleigh scattering that occurs in all directions when the incident laser beam acts on the particles in a sample |
| **Viscosity** | Property of a material that describes resistance to flow, depending on the temperature of the material; dynamic viscosity<br><br>For the mini DLS, samples will be dilute enough to assume that the viscosity of the sample will be that of the diluent. |

## A-4 SOFTWARE LICENSE AGREEMENT

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## A-5 DOCUMENT CHANGE HISTORY

| DATE | NOTABLE CHANGES | REVISION NUMBER |
|---|---|---|
| **2/9/2023** | New Document | 1.0 |
| **2/24/2023** | Specifications<br>Installation<br>Troubleshooting<br>Settings | 1.1 |
| **3/17/2023** | Updates throughout for regulatory requirements | 1.2 |
| **3/24/2023** | Updates throughout for regulatory requirements | 1.3 |
| **4/6/2023** | RS232 troubleshooting | 1.4 |
| **6/20/2023** | RS232 schema and example | 1.5 |
| **1/27/2025** | Specifications | 1.6 |
| **3/30/2025** | Updates throughout to reflect design changes | 2.0 |

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