User's Manual

Delsa™ Nano Submicron Particle Size and Zeta Potential

Particle Analyzer
Delsa™ Nano UI Software Version 3.73

CE

B08631AA
May 2011

Manufactured for
Beckman Coulter, Inc.
250 S. Kraemer Blvd.
Brea, CA 92821

BECKMAN COULTER

Delsa™ Nano Submicron Particle Size and Zeta Potential
User's Manual

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Find us on the World Wide Web at:
www.beckmancoulter.com

Revision History

Initial Issue, B08631AA, May 2011
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Safety Notice

Read all product manuals and consult with Beckman Coulter-trained personnel before attempting to operate the instrument. Do not attempt to perform any procedure before carefully reading all instructions. Always follow product labeling and manufacturer's recommendations. If in doubt as to how to proceed in any situation, contact your Beckman Coulter Representative.

Alerts for Danger, Warning, Caution, Important, and Note

⚠ DANGER

DANGER indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury. This signal word is to be limited to the most extreme situations.

⚠ WARNING

WARNING indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury. May be used to indicate the possibility of erroneous data that could result in an incorrect diagnosis (does not apply to all products).

⚠ CAUTION

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. May be used to indicate the possibility of erroneous data that could result in an incorrect diagnosis (does not apply to all products).

IMPORTANT IMPORTANT is used for comments that add value to the step or procedure being performed. Following the advice in the Important adds benefit to the performance of a piece of equipment or to a process.

NOTE NOTE is used to call attention to notable information that should be followed during installation, use, or servicing of this equipment.

Precautions for Use of This Equipment

Check the following prior to using this equipment:

- Check the outside of the equipment for damage, and check the inside of the equipment for foreign materials.
- Check the contact points, such as the position of the switches, and confirm that the equipment is functioning properly.
- Ensure that the equipment is properly grounded.
- Confirm that all cords are plugged in properly.

Check the following while using the equipment:

- Constantly monitor the entire equipment for the presence of foreign materials.
- Do not touch any moving parts aside from parts indicated by labeling.
- If foreign materials are discovered in the equipment, take appropriate measures, such as following the markings to stop the equipment.
- Do not use devices that communicate with radio waves near this equipment.
- When using this equipment, pay attention to the instructions on the warning labels attached to the equipment. See *Labels*.

Check the following after using this equipment:

- Maintain properly after use.
- Check for spills and debris in the sample compartment.

Check the following regarding the storage location:

- Avoid tilting, vibrating, or striking the equipment (even during transport), and maintain in a steady state.
- Store in a dry location.
- Store in a location free from extreme fluctuations in air pressure.

If the equipment malfunctions, immediately stop operations and contact Beckman Coulter Field Service.

Labels

Laser Product Identification Label (Rear Panel of DelsaNano)

Class 1 Laser Product Label (Rear Panel of DelsaNano)

Class 3B Service Laser Label (Inside DelsaNano Sample Area Lid)

Hot Surface Warning Label (Inside DelsaNano Sample Area)

Specifications

Bench Dimensions (minimum requirements)
- Width (with DelsaNano Auto Titrator): 152.4 cm (5 ft)
- Width (without DelsaNano Auto Titrator): 91.4 cm (3 ft)
- Depth: 61 cm (2 ft)
- Height: 61 cm (2 ft)
- Supportable weight: 68 kg (150 lbs)

DelsaNano Dimensions
- DelsaNano C: 380 mm W × 550 mm D × 212 mm H (15 in W × 21.7 in D ´ 8.3 in H)
- DelsaNano S: 380 mm W × 550 mm D × 212 mm H (15 in W × 21.7 in D ´ 8.3 in H)
- DelsaNano HC: 380 mm W × 550 mm D × 212 mm H (15 in W × 21.7 in D ´ 8.3 in H)
- DelsaNano Z: 380 mm W × 550 mm D × 212 mm H (15 in W × 21.7 in D ´ 8.3 in H)
- DelsaNano AT: 250 mm W × 310 mm D × 290 mm H (9.8 in W × 12.2 in D ´ 11.4 in H)

Weight
- DelsaNano C: Approx. 21 kg (46.3 lbs)
- DelsaNano S: Approx. 20 kg (44.1 lbs)
- DelsaNano HC: Approx. 21 kg (46.3 lbs)
- DelsaNano Z: Approx 21 kg (46.3 lbs)
- DelsaNano AT: Approx. 7.5 kg (16.5 lbs)

Environment
- Ambient operating temperature range: 15° C to 35° C (59° F to 95° F)
- Recommended ambient temperature operating range: 15° C to 30° C (59° F to 86° F) with minimal temperature fluctuation

Relative Humidity
30-80% (without condensation)

Power requirements
- 100–120 or 220–240 VAC, 50–60 Hz, single phase
- 250 VA (rated input power). Neutral to ground: <0.5 V Electrical Supply, Class 1

Fuses
- 100-120 V Current: T4A; Voltage: 125 V
- 220-240 V Current: T2A; Voltage: 250 V

Altitude restrictions
- Up to 2000 m (6562 ft)

Location restrictions
- Indoor use only

Installation category
- Category II

Pollution degree
- 2

Laser classification

Class 1 for operation (no access to radiation)
Class 3B for service and maintenance (trained Beckman Coulter personnel only)

Ventilation clearances (minimum requirements)
12.7 cm (5 in) on all sides of the instrument and controller

Nitrogen requirements (if measurements will be performed at 15° C or less)
Nitrogen air, maximum 10 psi
Tubing 4-mm OD, 2.5-mm ID, soft nylon
(Tubing length depends on the distance between the DelsaNano instrument and the nitrogen source.)

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Particle Sizing by Dynamic Light Scattering

Particles in Brownian Motion

Particles suspended in liquids are in Brownian motion due to random collisions with solvent molecules. This motion causes the particles to diffuse through the medium. The diffusion coefficient, D, is inversely proportional to the particle size according to the Stokes-Einstein equation:

Equation 1

$$D = \frac{k_B T}{3\pi\eta_0 d}$$

D: diffusion coefficient
k_B: Boltzmann's constant
T: absolute temperature
η_0: viscosity
d: hydrodynamic diameter

This equation shows that, for large particles, D will be relatively small, and, thus, the particles will move slowly while for smaller particles, D will be larger and the particles will move more rapidly. Therefore, by observing the motion and determining the diffusion coefficient of particles in liquid media, it is possible to determine their size.

Light Scattering From Particles in Brownian Motion

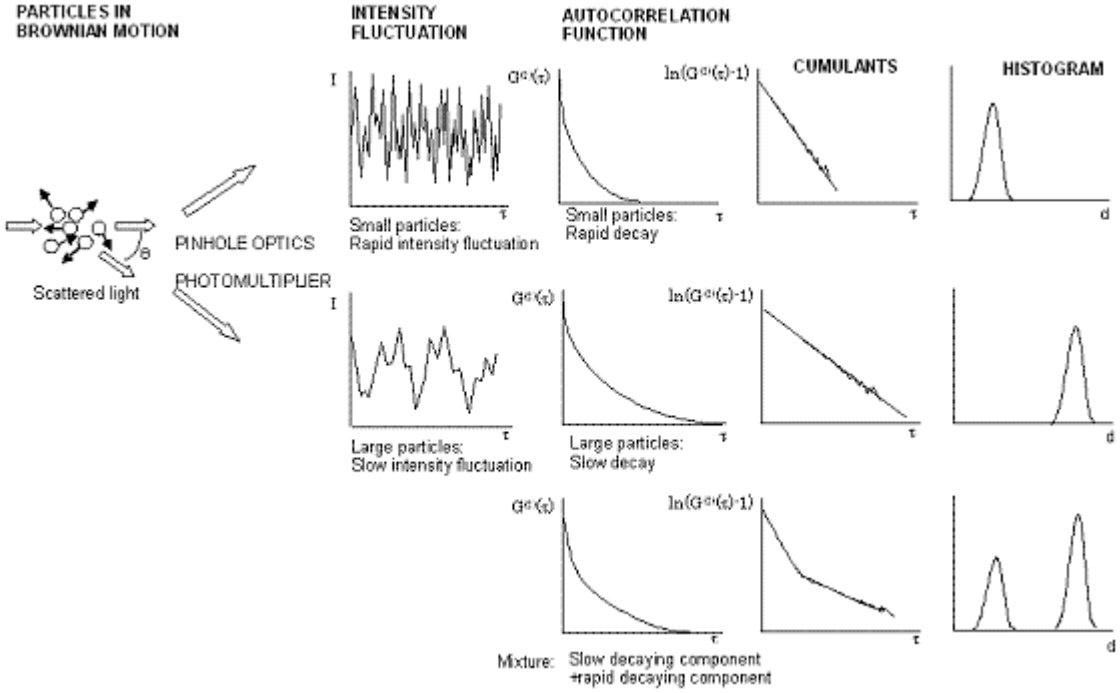
In dynamic light scattering, the fluctuations in time of scattered light from particles in Brownian motion are measured, photon correlation spectroscopy after dynamic light scattering. Figure 1 shows schematically how particle size and distributions are determined by the dynamic light scattering method.

When laser light is directed onto the particles, light is scattered in all directions. The scattered light that is observed comes from a collection of scattering elements within a scattering volume that is defined by the scattering angle and detection apertures. The observed intensity of the scattered light at any instant will be a result of the interference of light scattered by each element; and thus,

will depend on the relative positions of the elements. If the particles are in motion, the relative positions of particles will change in time; and thus, fluctuations in time of the scattered light intensity will be observed.

Because particles in Brownian motion move about randomly, the scattered intensity fluctuations are random. The fluctuations will occur rapidly for smaller, faster moving particles and more slowly for larger, slower moving particles. The fluctuations of the scattered light are analyzed using the autocorrelation function.

Figure 1 Determination of Particle Size by Dynamic Light Scattering



The Autocorrelation Function for Size Measurement

The calculation of the autocorrelation function $G^{(2)}(\tau)$ is one method of analyzing time dependent signals such as the random intensity fluctuation in Figure 2.

Equation 2

$$g^{(2)}(\tau) = \frac{\langle I(t) \cdot I(t+\tau) \rangle}{\langle I(t) \rangle^2}$$

$g^{(2)}(\tau)$: normalized intensity autocorrelation function
I(t): intensity detected at time t
τ: delay time
$\langle I(t) \rangle^2$: nomalization factor
< >: time average

For intensity fluctuations that are random, it can be noted that signals that are close to each other in time (small τ) are also close to each other in value and can be said to have high correlation, while

signals that are far apart (large τ) are different in value and can be said to have low correlation. The autocorrelation function will, thus, be a function that decays as a function of delay time (τ).

In the case of particles in Brownian motion, the normalized intensity autocorrelation function, $g^{(2)}(\tau)$, will be an exponential function or a sum of exponentials. For further analysis, it is useful to convert the intensity autocorrelation function to the autocorrelation function of the electric field of the scattered light $g^{(1)}(\tau)$ by using the Siegert relationship:

Equation 3

$$g^{(2)}(\tau) = \left|g^{(1)}(\tau)\right|^2 + 1$$

If all the particles in the scattering volume are of the same size (monodisperse), $g^{(1)}(\tau)$ will be a single exponential:

Equation 4

$$g^{(1)}(\tau) = B \cdot \exp(-\Gamma\tau)$$

where B is a constant dependent on instrumental parameters, such as the aperture (pinhole) size, and Γ is the decay constant which is proportional to the diffusion coefficient:

Equation 5

$$\Gamma = Dq$$

D: diffusion coefficient
q: magnitude of the scattering vector ($=4\pi n\sin(\theta/2)/\lambda$), where:
n: refractive index of media
λ: wavelength of incident light
θ: scattering angle

For small particles with rapid motion and rapid intensity fluctuations, the autocorrelation function is a rapidly decaying exponential function with a large decay constant, while for large particles the exponential decays more slowly with a smaller decay constant. It should also be noted that Γ is a function of the scattering angle and that the higher the angle, the faster the decay and lower the angle, the slower the decay of the correlation function.

For a mixture of particles (polydisperse), the intensity fluctuations will be due to particles differing in diffusion coefficients; therefore, autocorrelation will be a sum (or integral) of exponentials with differing decay constants:

Equation 6

$$g^{(1)}(\tau) = B\sum_i (A_i \exp(-\Gamma_i\tau))$$

Here, A_i is the relative intensity of light scattered by a particle with decay constant Γ_i and is related to relative amount of such particles.

Figure 2 Log Correlator for Size Measurement

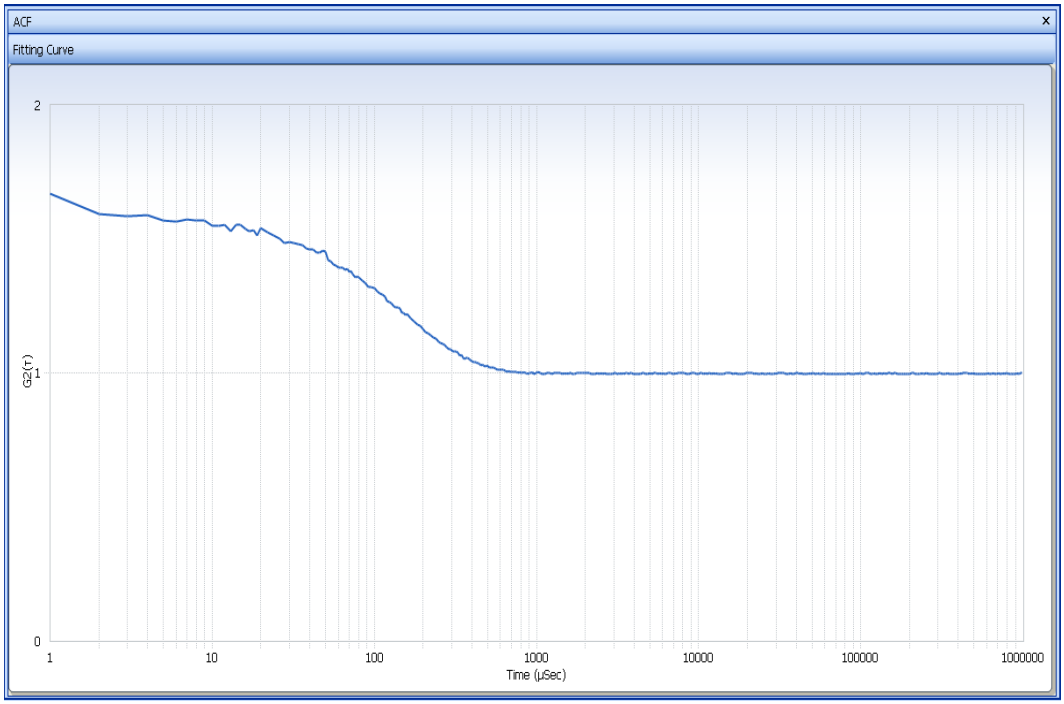
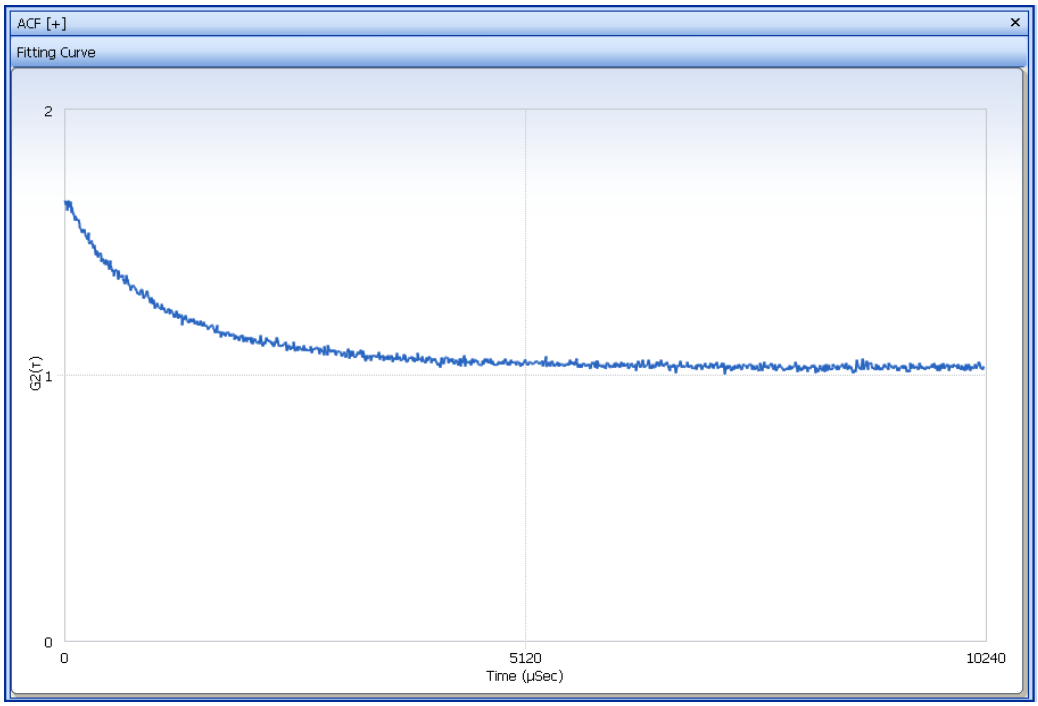


Figure 3 Linear Correlator for Size Measurement



Correlator

Two correlators are included in the instrument. One correlator is based on a log-scale decay time, and the other correlator is based on a linear-scale time. The log-scale correlator covers the range of six orders of decay time; thus, it can be adapted for most particles. On the other hand, the linear-scale correlator has high resolution within the limited range of decay time; it is suitable for very small particles of narrow distribution.

The Correlation Method

During a sample run, the scattered light intensity is collected as a data train of photon pulses per sampling time $\Delta\tau$, or sampling times (number of clock pulses) between two photon pulses. The former method is called the Time Domain (TD) method and the latter is called the Time Interval (TI) method. The length of the data train is specified by the parameter. The correlation function is then calculated for a specified number (channel number) of multiples of sampling times. The process is repeated for a number of times (accumulation times) and the correlation function is summed to reduce contributions due to noise.

The Time Domain Method

In the Time Domain (TD) method, the number of photon pulses between sampling times are collected. The calculation of TD method is a straightforward application of Equation 7.

The TD method is advantageous when the photon counts per sampling time is large; that is, for large particles with strong scattering levels and slowly decaying correlation functions that require larger sampling times.

The Time Interval Method

For very small particles that diffuse quickly, sampling times short enough to capture the rapid fluctuations are needed. But such particles usually scatter weakly and the situation is such that the photon counts per sampling time become very low. In this case, the TD method is an inefficient means of data collection. In the Time Interval (TI) method, the number of sampling times (clock pulses) between two photon pulses are collected as data and allows for more efficient determinations of the autocorrelation function for low-signal levels. In this method, the frequency distribution of time intervals between photon pulses (the total number of photon pulse pairs that are $\Delta\tau$ apart, $2\Delta\tau$ apart, and so on) is determined to obtain the autocorrelation function.

Having both TI and TD methods allows for the observation of a wide range of particle sizes and applicability. The large number of channels is important for analyzing a mixture of small particles or to fully capture the movement of large particles.

Determining Particle Size/Size Distributions

The Cumulants Method

For monodisperse particles, it can be seen from Equation 4 that the logarithm of $\mathbf{g}^{(1)}(\tau)$ will become a straight line. For polydisperse samples, the logarithm of $\mathbf{g}^{(1)}(\tau)$ will exhibit a curvature line.

In the Cumulants method, one fits the logarithm of $g^{(1)}(\tau)$ to a polynominal in τ to determine the coefficients, K_m:

Equation 7

$$\ln(g^{(1)}(\tau)) = \sum K_m(-\tau)^m / m!$$

The first order coefficient (or the slope of $\ln(g^{(1)}(\tau))$ is the average decay constant, $\langle\Gamma\rangle$, from which the average diffusion coefficient and particle diameter can be calculated by use of Equation 5 and Equation 1.

The second order coefficient divided by the square of $\langle\Gamma\rangle$ is the polydispersity index, $(\langle(\Gamma-\langle\Gamma\rangle)^2\rangle / \langle\Gamma\rangle)^2)$. The value of the polydispersity index is low, typically smaller than 0.1, for monodisperse samples and become larger for polydisperse samples.

The Methods for Particle Size Distribution

There are three methods included in the program to resolve particle size distributions from the measured autocorrelation functions.

- The CONTIN Method

  The well-documented computation routine known as CONTIN utilizes regularized non-negative least-squares technique combined with eigenfunction analysis. It has been implemented as the main data retrieval algorithm. CONTIN uses a non-linear statistical technique to smooth the solution and reduce the number of degrees of freedom to an acceptable level. Users must specify the distribution range, the number of data points, and other constants. The regularization parameter can be automatically chosen. CONTIN considers account the weighting of the distribution, due to the use of discrete data points in the continuous distribution, and then calculates different moments of the computed distribution.
- The Marquardt Method

  The Marquardt method is an iterative method in which an initial “guess” is repeatedly processed to give a final answer. In the analysis program, the initial “guess” is a histogram with all the steps being of equal height. The algorithm is repeated to change the histogram to fit the raw data. The number of times the algorithm is repeated is the step number. The lambda parameter adjusts how much the answer changes in one step; if it is small the answer changes rapidly, if it is large, the answer changes slowly. Distributions obtained by this method tend to be broad and have connected peaks.
- The Non-Negative Least-Squares (NNLS) Method

  This is a least-squares algorithm which solves the matrix so that only positive values for A_i are obtained (in the Marquardt algorithm, negative values of A_i may be obtained but the values are

set to “0” when this happens). Distributions obtained by this method tend to have narrow and separated peaks.

Molecular Weight (MW) Analysis

The Molecular Weight (MW) analysis is performed for all types of particle size distributions. In the Cumulants analysis, the average diameter is converted into MW. The MW analysis requires that you enter two sample-dependent parameters, a and b, in order to calculate MW. The transformation equation for mean molecular weight is as follows:

Equation 8

$$MW = \left(\frac{\alpha}{D}\right)^{\frac{1}{\beta}}$$

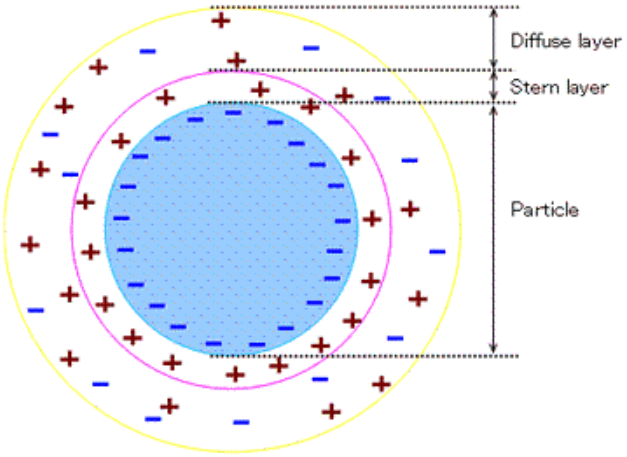
where D is the diffusion coefficient as defined in Equation 1.

Zeta Potential Determination by Electrophoretic Light Scattering

Electrical Double Layer

Most particles dispersed in a liquid have positive or negative charge. In a liquid, the ions that have opposite charge to the particle surface gather close to the particle to keep an electric neutrality. Because the particle surface is surrounded by such ion clusters, ionized layers with opposite charge surround the layer on the surface of a particle (Figure 4). This phenomenon is expressed as an “electrical double layer.”

Figure 4 Schematic Diagram of Electrical Double Layer



Because the ion in a liquid moves by thermal diffusion, concentration of the counter ion is high in the area near the particle’s surface and gradually decreases with distance from the surface. Equal

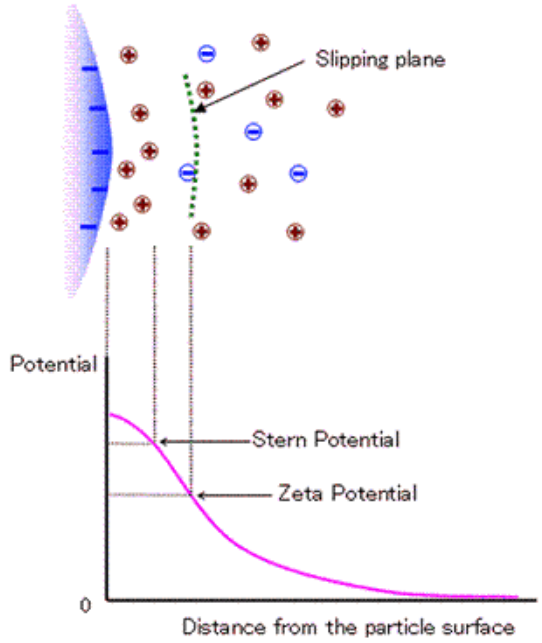
numbers of positive and negative ions exist in the area far from the particle's surface so that electric neutrality is maintained. This ion distribution is called a "diffuse electrical double layer." The diffuse electrical double layer can be divided into two layers:

- The layer of ions near the particle surface is called the "Stern layer." The ions in the Stern layer are strongly attracted to the surface of the particle.
- The layer outside the Stern layer is called the diffuse layer. In this layer, the ions are diffused.

Zeta Potential

The stability of the dispersing particles is influenced by their surface charge. Zeta potential is used as the index of the surface charge of the particles. It is assumed that the particles undergoing Brownian motion in a liquid move not only with the ions in the Stern layer where the ions are attracted strongly near the particle surface but also with part of the diffuse layer. The field from which this movement takes place is called the "slipping plane." Zeta potential is considered to be the potential at the slipping plane and the potential at the position far from the particle surface is defined as zero (Figure 5). If zeta potential is high, the particles are stable due to high electrostatic repulsion between particles. On the contrary, a low zeta potential value (approaching zero) increases the probability of particles colliding; therefore, forming particle aggregates. Thus, zeta potential is used as an index of the dispersion stability of particles.

Figure 5 Concept of Zeta Potential



Electrophoretic Light Scattering

When an electric field is applied to charged particles in the suspension, particles move toward an electrode opposite to its surface charge. Because the velocity is proportional to the amount of charge of the particles, zeta potential can be estimated by measuring the velocity of the particles. Electrophoretic light scattering is the method most generally used to determine the velocity of the particles. To determine the speed of the particles movement, the particles are irradiated with a laser light, and the scattered light emitted from the particles is detected. Because the frequency of the scattered light is shifted from the incident light in proportion to the speed of the particles movement, the electrophoretic mobility of the particles can be measured from the frequency shift of the scattered light. This method is based on the Doppler effect; therefore, it is also called the "Laser Doppler Method."

Figure 6 displays the optical path of the DelsaNano. When measuring zeta potential, the DelsaNano detects the scattered light from the particles by combining incident light (reference light) with the scattered light. Because the intensity fluctuation of the combined light is equivalent to the frequency difference between the scattered and incident light observed, it is possible to precisely measure small frequency shifts. The incidence light is also used for particle size measurement.

The amount of frequency shift ν_D of scattered light is related to the mobility of particles, U:

Equation 9

$$\nu_D = \frac{Uq}{2\pi}\cos\frac{\theta}{2} = \frac{Un}{\lambda}\sin\theta$$

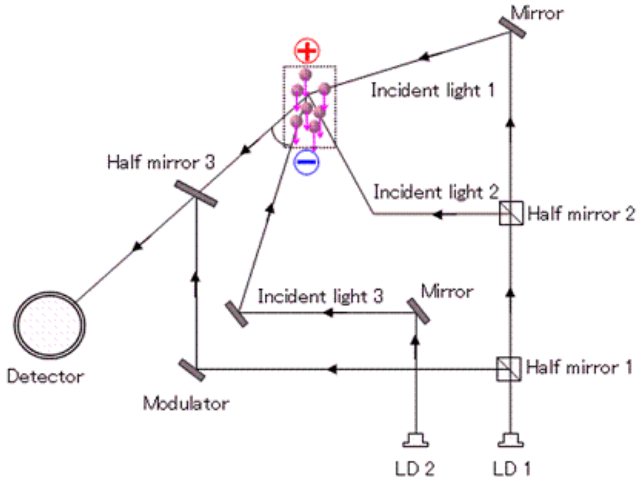
where q is the scattering vector and $q = 4\pi n\sin(\theta/2)/\lambda$. λ is the wavelength of the incident light, n is the refractive index of a medium, and θ is the scattering angle. In many aqueous solutions containing an electrolyte, zeta potential can be calculated from the Smoluchowski equation.

Equation 10

$$Z = \frac{\eta}{\varepsilon_0\varepsilon_r}U$$

Where ε_0 and ε_r are dielectric constants in vacuo and of the solvent, respectively.

Figure 6 Optical Diagram of DelsaNano C and HC



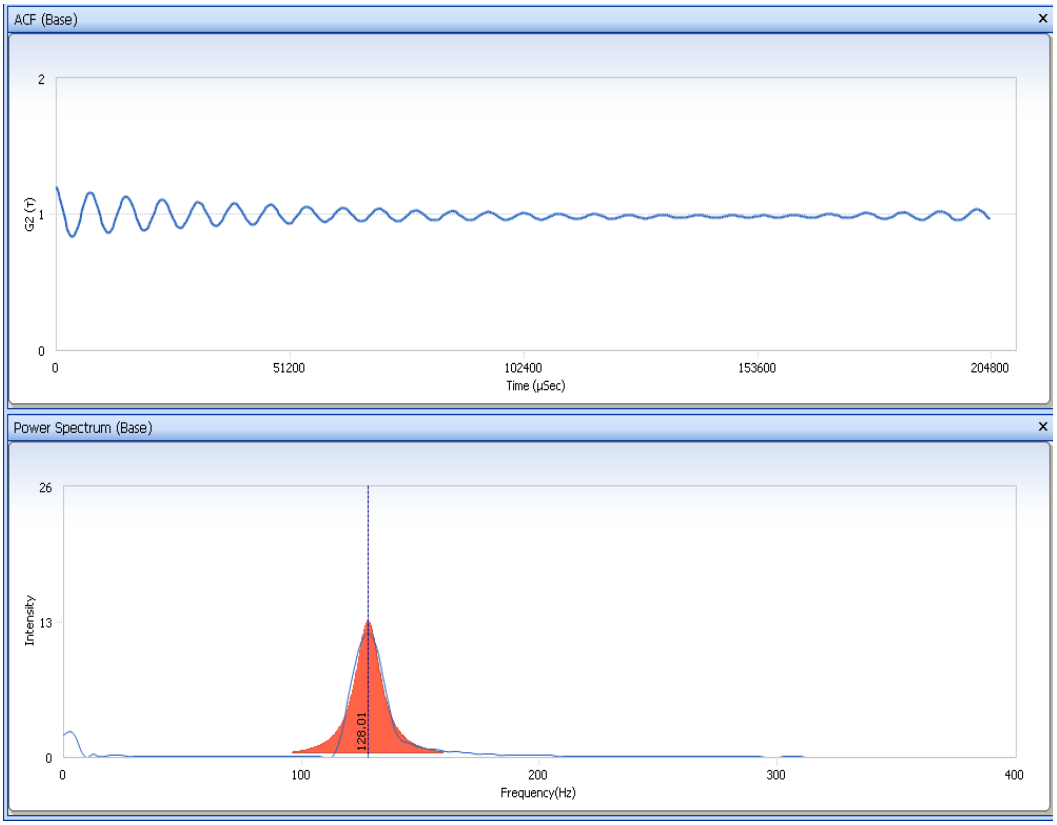
The Power Spectrum for Zeta Potential Measurement

Power Spectrum analysis provides an easy and direct way to obtain electrophoretic mobility information.* The instrument acquires the ACF first and then converts into power spectrum by the Fourier transformation (Figure 7).

In the distribution graph (Figure 8), the Brownian motion of the particles is characterized by a Lorentzian peak, centered at a frequency shift that characterizes electrophoretic mobility of the particles.

If the sample is a mixture of particles of different mobility, for example, 2, then 2 peaks can be selected for Lorentzian fit.

Figure 7 ACF and Power Spectrum of Base Measurement



* Renliang Xu, Particle Characterization: Light Scattering Methods (Particle Technology Series)

Figure 8 ACF and Power Spectrum of Test Measurement

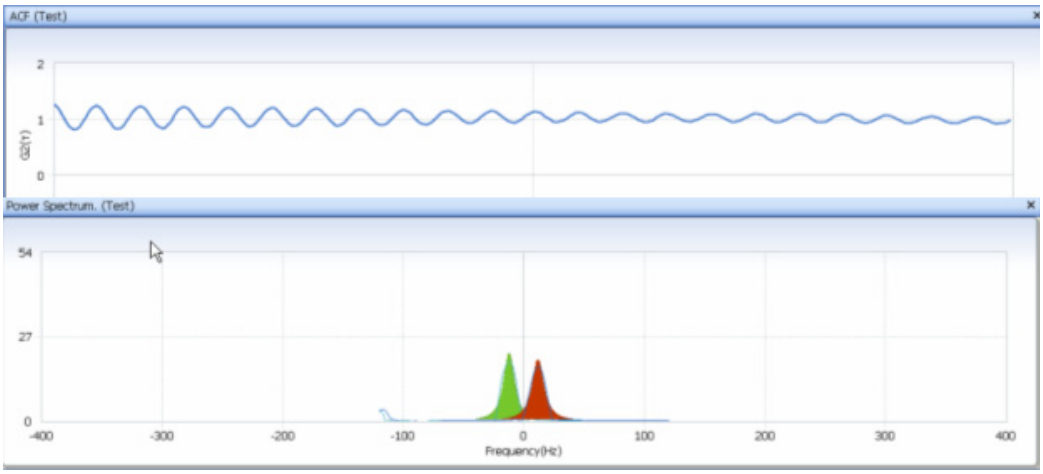
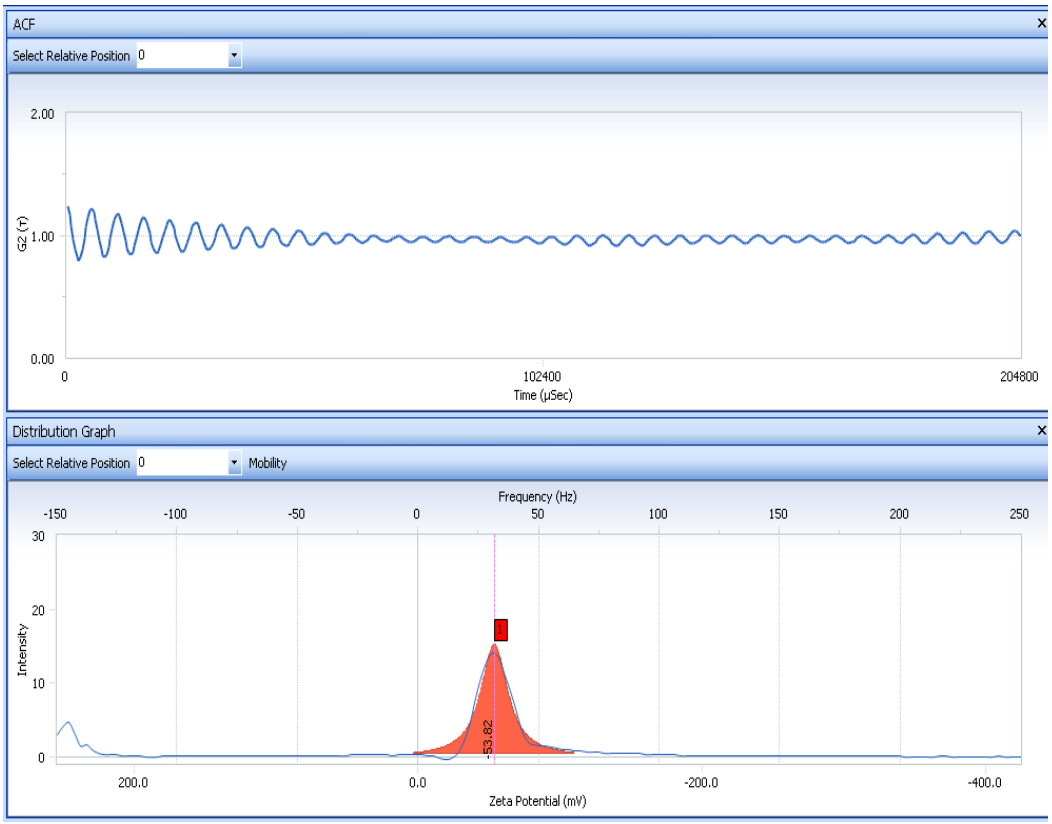


Figure 9 ACF and Power Spectrum of Sample Zeta Potential Measurement

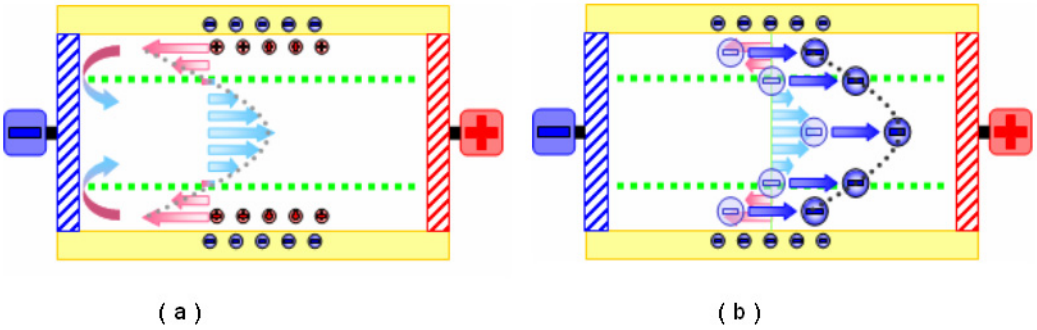


NOTE In Figure 9, the blue line indicates raw data; the red curve indicates fitted data; and 1 indicates the Lorentzian fit.

Zeta Potential Measurement Using Electrophoretic Light Scattering

Most colloidal particles have an electrical charge on their surface when dispersed in liquids. The particles move towards the electrode that has an opposite charge if an electric field is applied to the cell that contains the particle suspension. Besides the particle movement, electroosmotic flow is also induced in the cell due to the surface charge of the cell wall. Because the cell is typically a closed system, electroosmotic flow occurring at the position close to the cell wall moves towards the opposite electrode, then hits the side wall of the cell, and flows back to the center of the cell. When measuring the zeta potential of the particles, an apparent mobility of the particles, which is equal to the sum of the electroosmotic flow and particle true mobility, is observed. See Figure 10.

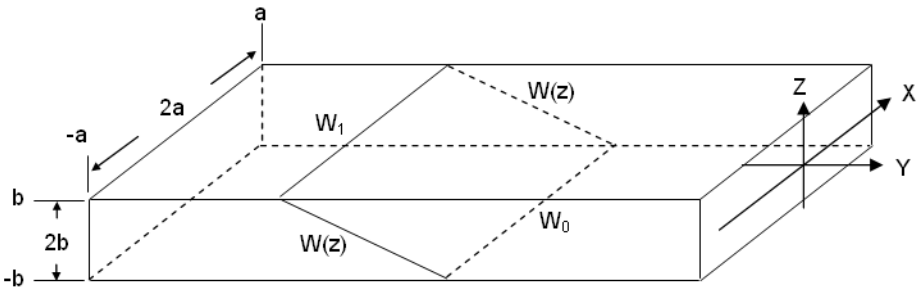
Figure 10 Schematic Diagram of the Electroosmotic Flow Occurring in a Close Cell



(a) is the electroosmotic flow
(b) is the electroosmotic flow and particle true mobility

Electroosmotic flow has a symmetric parabolic profile when the particle concentration in the cell is homogeneous and the electrical charges on the upper and lower surfaces of the cell are equal. However, electroosmotic flow is asymmetric in many cases due to the sedimentation of particles, differences in the charges of the upper and lower cell surfaces, or for other reasons. Mori and Okamoto expanded Komaga's model to generate an equation that can be applied to both symmetric and asymmetric osmotic flows.

Figure 11 Boundary Condition of the Electroosmotic Flow in the Cell



Assume the velocity of the electroosmotic flow at upper and lower surfaces of the cell is W_1 and W_0, respectively. The cross-sectional dimensions of the rectangular cell as 2a (X axis) and 2b (Z axis), with a>b, as shown in Figure 11. The electrophoretic mobility of the particles is the ratio of velocity of the particles to the electric field strength. The apparent mobility $v(x, z)$ of the particles can be represented as the sum of true mobility and electroosmotic flow, where the velocity of electroosmotic flow changes linearly from W_1 to W_0 in the Z direction on the sides of the cell.

Equation 11

$$v_{obs} = v_p + v(x,z)$$

ν_{obs} is apparent mobility
ν_p is particle true mobility
$\nu(x, z)$ is the electroosmotic flow variation in z direction

Electroosmotic flow in Z direction $\nu(0, z,)$ is:

Equation 12

$$v(0,z) = v_o + \Delta v_0 z/b - A v_0 (1 - z^2/b^2)$$

where:

$$A = (\frac{2}{3} - \frac{0.420166}{k})^{-1}, \quad k = a/b$$

where:

ν_0 is the average of electroosmotic flow at upper and lower surfaces of the cell ($\nu_0=(W_1+W_0)/2$), $\Delta\nu_0$ is the difference of electroosmotic flow between upper and lower surfaces of the cell ($\Delta\nu_0$=W1+W0).

Hence, apparent particle mobility $\nu_{obs}(0, z)$ transforms into:

Equation 13

$$v_{obs}(0,z) = A v_o (z/b)^2 + \Delta v_0 (z/b) + (1-A) v_0 + v_p$$

Equation 13 indicates that nobs(0, z) is quadratic expression in (zi=z/b).

Equation 14

$$v_{obs}(0,z) = k_2 z_i^{\ 2} + k_1 z_i + k_0$$

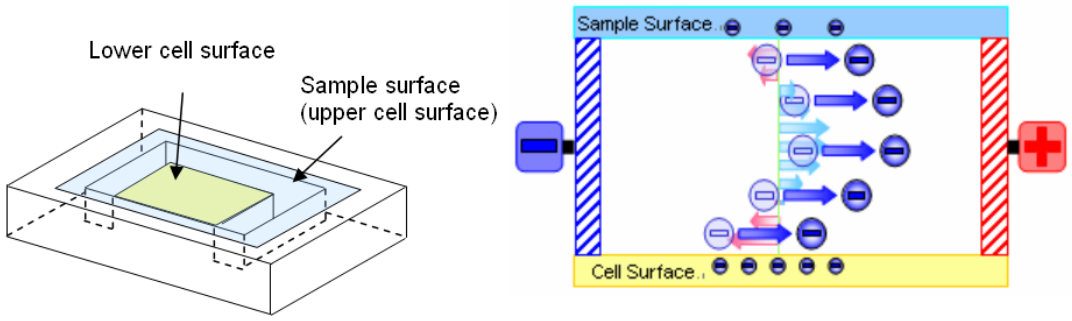
where:

$k_2=Av_0$、 $k_1=\Delta v_0$, $k_0=(1-A)v_o+v_p$

Thus, ν_0, $\Delta\nu_0$, and ν_p can be calculated from the coefficients K_0~K_2 in Equation 14 if least square fitting is applied to $\nu_{obs}(0, z)$ observed from a different position in z direction.

Zeta potential of a flat surface sample can be determined from Equation 12 to Equation 14 using a flat surface cell (Figure 12) in which the surface of the sample is the upper wall component of the cell.

Figure 12 Schematic Diagram of the Flat Surface Cell and Apparent Particle Mobility in the Cell



Electroosmotic flow becomes asymmetric in this cell due to the difference in the surface charges of upper and lower surfaces of the cell. However, the Mori and Okamoto equation described above can be applied to this cell to determine the velocity of electroosmotic flow at upper (sample) surface W_1 as:

Equation 15

$$W_1 = v_0 + \frac{\Delta v_o}{2}$$

W_1 can also be calculated from Equation 15 by subtracting true mobility from apparent mobility for the position $z=b$; i.e., $v_{obs}(0, b)$. From W_1, the surface zeta potential of the sample is calculated by using Smolochowski equation.

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CHAPTER 1

Operation

Introduction

The DelsaNano models are as follows:

- DelsaNano S (A53876) measures the particle size of samples in suspension in a range from 0.6 nm to 7 µm. Samples may be concentrated or dilute.
- DelsaNano C (A53878) measures the particle size of samples in suspension in a range from 0.6 nm to 7 µm. Samples may be concentrated or dilute. In addition, zeta potential measurements on dilute, concentrated samples, as well as solid flat surface materials.
- DelsaNano HC (A53879) measures the particle size of samples in suspension in a range from 0.6 nm to 7 µm at a higher sensitivity. Samples may be concentrated or dilute. In addition, high sensitivity zeta potential measurements on dilute, concentrated samples, as well as solid flat surface materials.
- DelsaNano Z (A53877) measures the zeta potential of samples in suspension in a range from 0.6 nm to 30 µm. Samples may be concentrated or dilute.
- DelsaNano Auto Titrator (AT) (A53880) is an optional accessory for use with all DelsaNano models. It provides automatic three titration modes, pH titration in the range from 1 to 13, and additive and circulation titrations.

NOTE This manual applies to all DelsaNano models. If you are using a DelsaNano S system, the section related to zeta potential is not applicable to your configuration.

Powering on the Equipment

To power on the components:

1 Turn the power switch to the On position. The switch is located on the left rear of the DelsaNano.

NOTE Turn the power on at least 30 minutes prior to starting the measurements. It takes approximately 30 minutes for the laser to stabilize before taking measurements.

2 Observe the LEDs located on the top panel of the DelsaNano. During startup, the power LED on the DelsaNano will turn orange. Startup is complete when the LED turns green, enabling communications with the PC.

Figure 1.1 Power LED on DelsaNano

3 Turn on the PC.

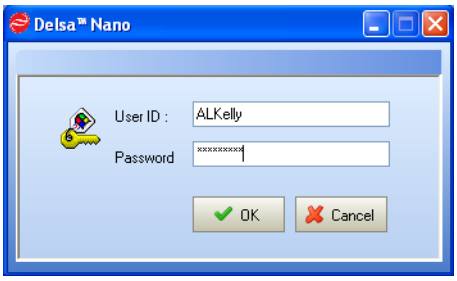
Starting the Software

To start the software:

1 From Windows, double-click the DelsaNano icon on the desktop, or select the program from your program list.

- If Security is set to "No Security" (default), after a brief initialization process, the software is ready for use.
- If Security is set to "Security" or "21 CFR Part 11," a login screen appears. (For information on Security, see *Setting Security*.)

Figure 1.2 Security Login Dialog



2 Enter a user ID and password, and click **OK**. After a brief initialization process, the software is ready for use.

When the software is ready for use, the DelsaNano main screen appears (Figure 1.3). It contains several panels with functions that allow you to prepare for measurements.

Figure 1.3 DelsaNano Main Screen

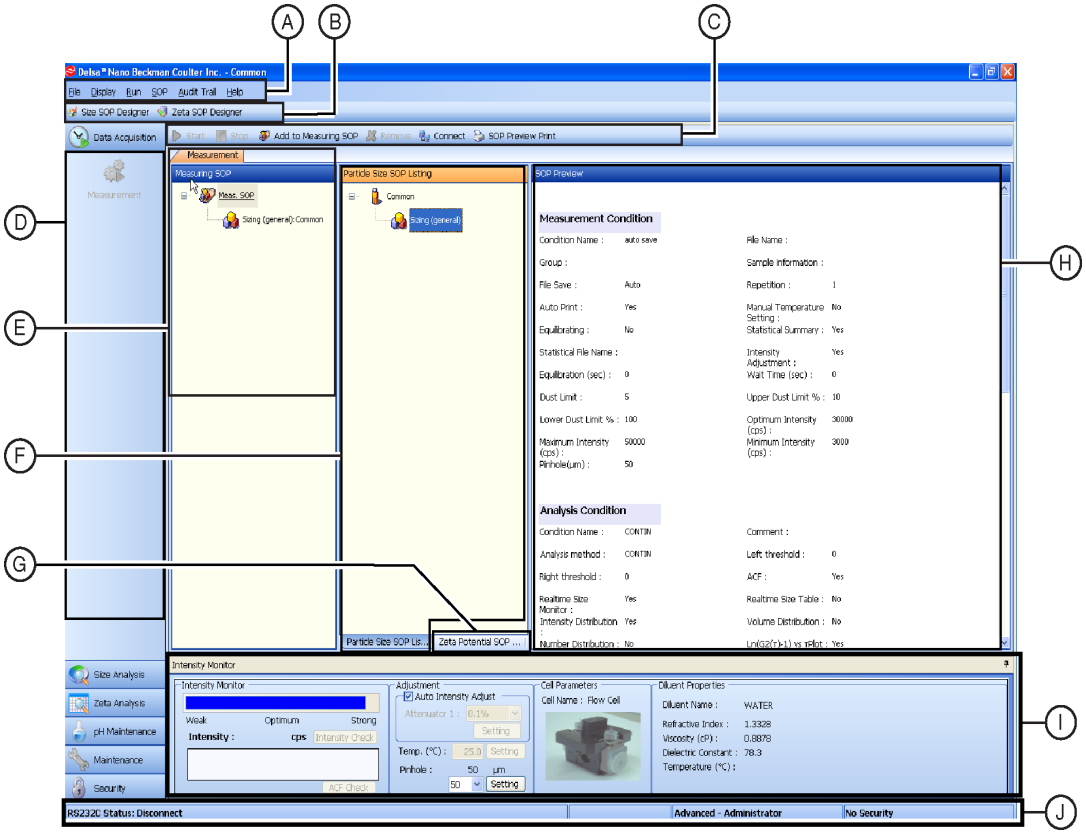


Table 1.1 DelsaNano Main Screen Elements

	Screen Element	Description
A	Main Menu Bar	For information on the menus on the menu bar, see Using the Main Menu Bar.
B	SOP Designer Toolbar	Contains two buttons: • **Size SOP Designer** is used to create and view SOPs for size measurement. • **Zeta SOP Designer** is used to create and view SOPs for zeta potential measurement.
C	Button Bar	This button bar is not present in all screens. When it is present, the buttons that appear on the bar apply to the selected function or screen. For example, when you select the Measurement function icon in the Data Acquisition function panel, the following buttons appear: • **Start** turns green when the instrument is connected. • **Stop** turns red when the instrument is measuring. • **Add to Measuring SOP** is used to add either size or zeta SOP to measuring SOP. • **Remove** is used to remove the size or zeta SOP used for measurement from the measuring SOP. • **Connect** is used to initialize the instrument, which must be turned on. • **SOP Preview Print** is used to print an SOP. When you select the Security function panel, the following buttons appear: • **Add New User** is used to add a new user. • **Edit User Profile** is used to edit an existing user's profile. • **Delete User** is used to delete a user. This button is replaced by the following when 21 CFR Part 11 security is enabled: — **Disable User** is used to disable a user's login. — **Enable User** is used to enable a user to log into the DelsaNano software. Other buttons appear in the function panel as appropriate. When you are working in an SOP Designer, this button bar is not available. Instead, a different button bar appears inside each panel in the SOP Designer.

Table 1.1 DelsaNano Main Screen Elements *(Continued)*

	Screen Element	Description
D	Function Icons Panel	This panel contains the main software functions. Select a function name to open the corresponding set of task icons: • Data Acquisition function icon: Measurement • Size Analysis function icons: Analysis, QC, pH Analysis • Zeta Analysis function icons: Analysis, QC, pH Analysis, Low Conductivity Cell Analysis • pH Maintenance function icons: Configuration, pH Calibration, Priming, Consumable Check, Sample Circulation, pH Monitor • Maintenance function icon: System Configuration • Security function icons: User Management, Security Settings, and Data Mirroring
E	Measuring SOP Listing	This panel displays the SOP that will be used in the measurement.
F	Particle Size SOP Listing	This panel/tab displays the registered particle size measurement SOPs.
G	Zeta Potential SOP Listing	This panel/tab displays the registered zeta potential measurement SOPs.
H	SOP Preview	This panel displays the parameters within a selected SOP. To display the parameters of an SOP, select the SOP in the SOP Listing.

Table 1.1 DelsaNano Main Screen Elements *(Continued)*

	Screen Element	Description
I	Intensity Monitor	This form at the bottom of the screen displays the parameters that must be checked prior to starting a measurement, such as sample's scattering intensity strength, Auto Intensity adjustment, pinhole size, temperature, the appropriate cell used for measurement, and diluent properties within the SOP conditions that have been registered for the selected SOP. The Pinhole option allows you to select one of the pinholes that will be used for the size or zeta potential measurements. To change the Pinhole from the default setting, select the desirable Pinhole size from the drop menu and click Setting. The intensity of the sample appears in cps (photon counts per second), and the message box provides instant feedback on whether or not to proceed with measurement. Appropriate action must be taken based on the feedback. The software adjusts the scattering intensity from the sample automatically. If the scattered intensity is very high, it is attenuated appropriately by the neutral density filters. The Intensity Check displays the number of scattering elements based on their scattering intensity levels. You can select the minimum and divisions. The mean SD of the scattering appears at the end of measurement. The ACF Check allows you to see the preview of the ACF prior to the size measurements.
J	Status Panel	The Status panel at the bottom of the window indicates connection status, the mode (Normal or Advanced), the permission level of the currently logged-in user, and the Security setting (for example, 21 CFR Part 11).

Using the Main Menu Bar

The Main menu bar and its drop-down menu items change based on the Security settings. All available menu commands per menu are listed in Table 1.2.

Table 1.2 Main Menu Commands

Menu	Available Commands
File	**Save Measured Data**
	Export Text
	Print Preview
	Print
	Log Out
	Exit
Display	**Normal Mode**
	Advanced Mode
	Restore Default Display
Run	**Start**
	Stop
	Connect
SOP	**Size SOP Designer**
	Zeta SOP Designer
	Add to Measuring SOP
	Remove
	Favorite SOP > **Add to Favorite SOP**
	Favorite SOP > Add to Favorite SOP Series
	Favorite SOP > **Organize Favorite SOP**
	Favorite SOP > Organize Favorite SOP Series
Option > **21 CFR Part 11**	**Signature**
	Playback (Read File)
	Playback (Real Mode)
Audit Trail	**Error Audit Trail**
	pH Calibration Audit Trail
	Operation Audit Trail
	Parameter Audit Trail
Help	**Index**
	Search
	Printable
	About

Setting Security

Three security levels are available in the DelsaNano software:

- No Security

  No user ID or password is required upon software startup. No restrictions are placed on access; therefore, all users can use all functions.

- Security

  A user ID and password are required upon software startup. The SOPs and measurement data accessibility are controlled based on user level. Having a Security setting allows you to block the viewing of SOPs and measurement data by other users.

- 21 CFR Part 11

  A user ID and password are required upon software startup. Authorized users can use electronic records and electronic signatures in compliance with the FDA's Electronic Records and Electronic Signatures Rule (21 CFR Part 11).

  NOTE 21 CFR Part 11 security must be enabled. The Administrator can set Password Policy parameters for each user: password expiration, auto-logout after a period of inactivity, and auto-close of the Signature dialog after a period of inactivity.

Changing Security Settings

To change the security settings of the DelsaNano, you need the serial number of the instrument. The serial number is located on the rear panel next to the USB communication port. The serial number and the MAC address are used to generate the authentication code for 21 CFR Part 11.

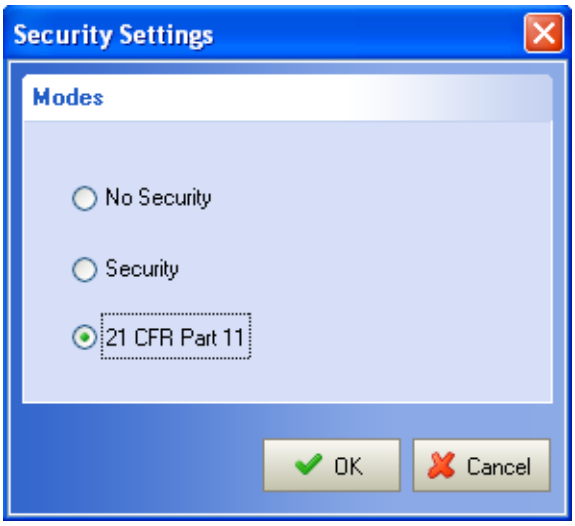
⚠ WARNING

Do not change these numbers after you enable 21 CFR Part 11.

To change security settings:

1 Select the Security Settings icon in the Security function panel. The Security Settings dialog opens.

Figure 1.4 Security Settings Dialog



2 Select the desired security setting, and click **OK**.

NOTE To change from No Security to Security or 21 CFR Part 11, there must be at least one user name registered as Administrator. If there is no Administrator, a warning message appears and the security setting does not change. For information on setting up user levels, see *User Administration*.

User Administration

The DelsaNano software supports four user levels; Table 1.3 lists their associated permissions and system access.

Table 1.3 User Levels and Associated Permissions

User Level	Perform Measurements and Analyses?	Edit SOPs?	Edit System Parameters?	Change User Information?
Operator	Yes	No	No	No
Advanced Operator	Yes	Yes	No	No
Supervisor	Yes	Yes	Yes	No
Administrator	Yes	Yes	Yes	Yes

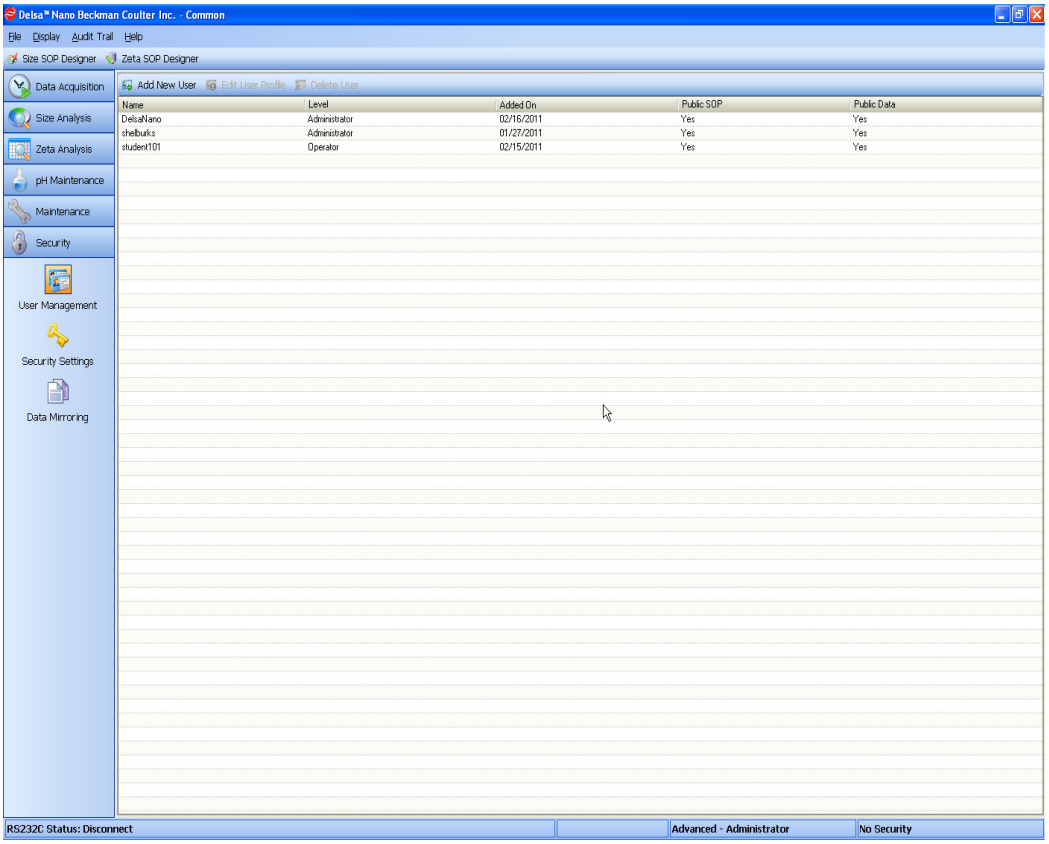
Viewing User Information

NOTE To access information on all system users, you must be logged in as Administrator.

To view user information:

Select the User Management icon from the Security function panel. A list of registered users appears.

Figure 1.5 User Management Screen



The User Management screen includes the information described in Table 1.4.

Table 1.4 User Management Screen

Screen Element	Description
User Status (21 CFR Part 11 security must be enabled)	"Enable" appears if the user is currently enabled. "Disable" appears if the user is currently disabled.
Name	The user name.
Level	The permission level assigned to this user name. Can be one of: Operator, Advanced Operator, Supervisor, Administrator.
Added On	The date on which the user information was created.
Public SOP	When selected, the user's SOPs can be viewed by other users.
Public Data	When selected, the user's data to be viewed by other users.

Table 1.4 User Management Screen *(Continued)*

Screen Element	Description
Password Policy (21 CFR Part 11 security must be enabled)	The Administrator can set the following parameters for each user: • Password Expiration (45–300 days) • Automatic logout from software after a period of inactivity (4–120 minutes) • Automatic Signature dialog close after a period of inactivity (4-120 seconds)
User Management Toolbar Buttons	Three buttons are available for performing user management functions: • **Add New User** is used to add a new user. • **Edit User Profile** is used to edit an existing user's profile. • **Delete User** is used to delete a user. This button is replaced by the following when 21 CFR Part 11 is enabled: — **Disable User** is used to disable a user's login. — **Enable User** is used to enable a user to log into the DelsaNano software.

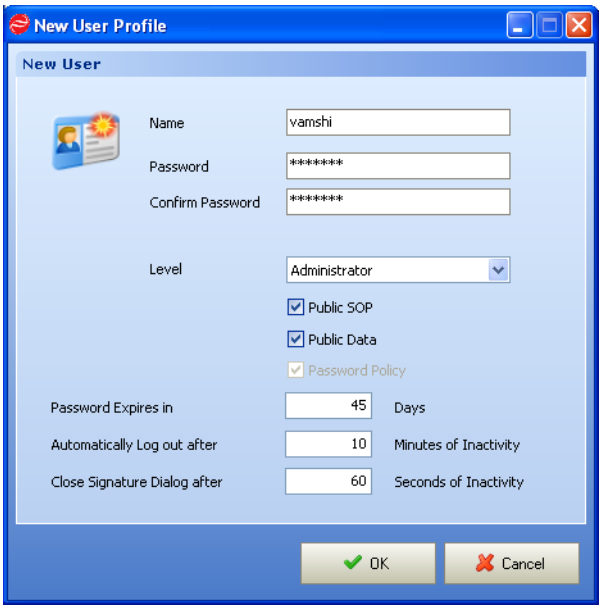
Registering New Users

NOTE To register new users, you must be logged in as Administrator.

To register a new user:

1 In the User Management window, click **Add New User**. The New User Profile dialog opens.

Figure 1.6 New User Profile Dialog



2 Enter the user name, password, and other parameters for the new user. The parameters are described in Table 1.5.

Table 1.5 New User Dialog

Parameter	Description
Name	Requires at least six alphanumeric characters.
Password	Requires at least six alphanumeric characters.
Confirm Password	Enter the same password again to confirm.
Level	Select one of the four available user levels: Operator, Advanced Operator, Supervisor, Administrator.
Public SOP	Select to allow other users to view and copy SOPs. Other users cannot edit the SOPs.
Public Data	Select to allow other users to view measurement data.
Password Policy (21 CFR Part 11 only)	Set the following constraints: • Password Expiration (45-300 days) • Automatic logout from software after a period of inactivity (4–120 minutes) • Automatic Signature dialog close after a period of inactivity (4-120 seconds)

3 When finished, click **OK**.

Changing Your Password

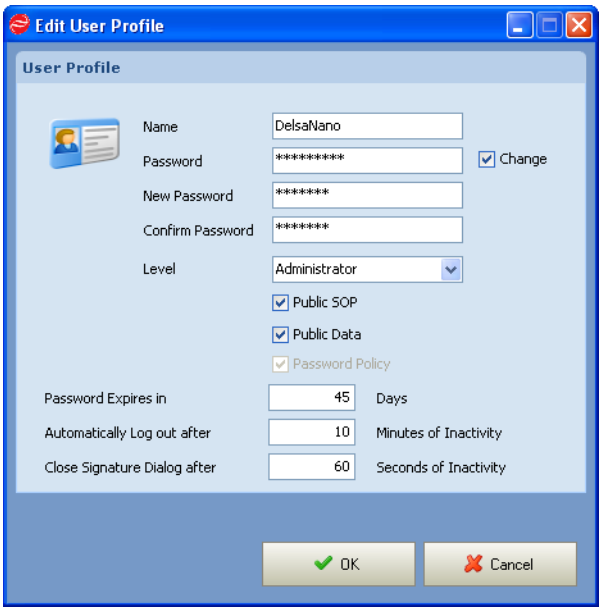
NOTE You cannot change another user's password. Only the Administrator can reset another user's password.

To change your password:

1 Select the User Management icon in the Security function panel.

2 Click **Edit User Profile**. The Edit User Profile dialog opens.

Figure 1.7 Edit User Dialog



3 Select Change (next to the Password field).

4 Enter the current password in the Password field.

5 Enter a new password in the New Password and Confirm Password fields.

6 If you have Administrator privileges, you can change the settings for Public SOP, Public Data, and Password Policy.

7 When finished, click **OK**.

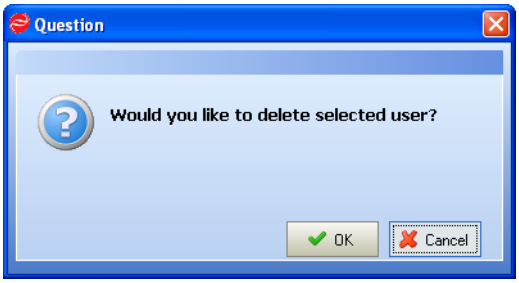
Deleting Users

NOTE This feature is not available under 21 CFR Part 11 security.

To delete a user:

1 In the User Management window, select the user name to be deleted, and click **Delete User**. A confirmation dialog appears.

Figure 1.8 Delete User Dialog



2 Click **OK**.

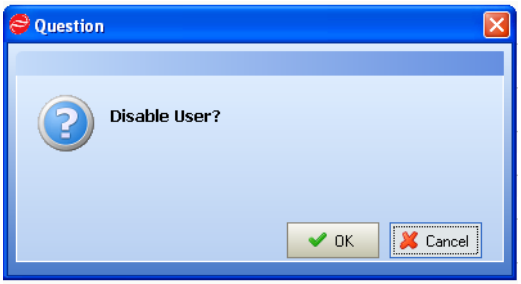
Disabling or Enabling Users (21 CFR Part 11)

To disable or enable a user:

1 In the User Management window, check the user's current status in the User Status column.

2 Select the user name to be disabled or enabled, and click **Disable User** or **Enable User** as appropriate. A confirmation dialog appears.

Figure 1.9 Disable User Dialog



3 Click **OK**.

Enabling Data Mirroring

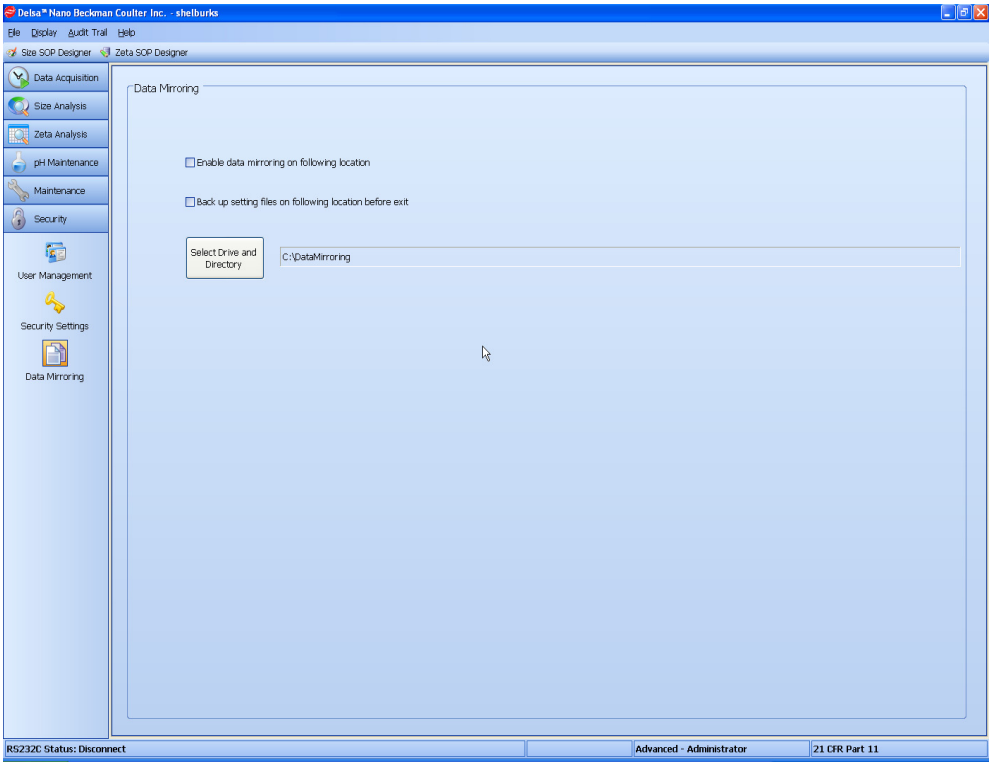
NOTE This feature allows you to securely store files in a separate location. You must be an Administrator or Supervisor to enable data mirroring.

To enable data mirroring:

1 Select the Maintenance function icon panel. The System Configuration screen opens.

2 Select Data Mirroring in the System Configuration panel. The Data Mirroring screen opens.

Figure 1.10 Data Mirroring Parameters Screen



3 Select the Enable data mirroring on following location check box.

4 If you want your files to be backed up before you exit, select the Back up setting files on following location before exit check box.

5 To specify the location of the backup, click **Select Drive and Directory**, browse to the appropriate location in the Browse dialog, and click **OK**.

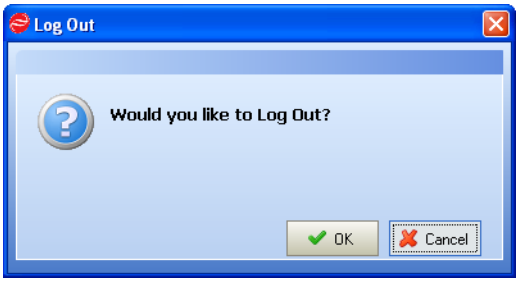
Logging Out

NOTE The logout feature is not available when Security is set to "No Security."

To log out:

1 Select **File > LogOut** on the Main menu bar. A confirmation dialog appears.

Figure 1.11 Logout Dialog



2 Click **OK**. If Security is on, the Login dialog appears.

3 To log in again, enter the user name and password, and click **OK**.

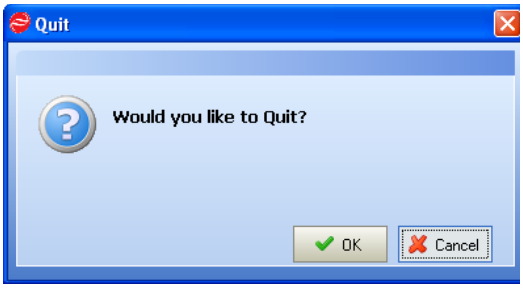
Shutting Down the Software

NOTE Always shut down the software before powering off the instrument.

To shut down the DelsaNano software:

1 Select **File > Exit** on the Main menu bar. A confirmation dialog appears.

Figure 1.12 Quit Dialog



2 Click **OK**. The DelsaNano window closes.

SOP Designer

Use the SOP Designer to create new SOPs. Use the Size SOP Designer to measure particle size, and use the Zeta SOP Designer to measure zeta potential. In each case, you must set the following parameters (each set of parameters appears in its own panel in the SOP Designer screen):

- Measurement
- Analysis
- Cell
- Diluent properties

Opening the SOP Designer Window

To open the SOP Designer window, select one of the following on the SOP Designer Toolbar:

- Click **Size SOP Designer** or **Zeta SOP Designer**.
  The appropriate SOP Designer window opens.

Figure 1.13 SOP Designer Window

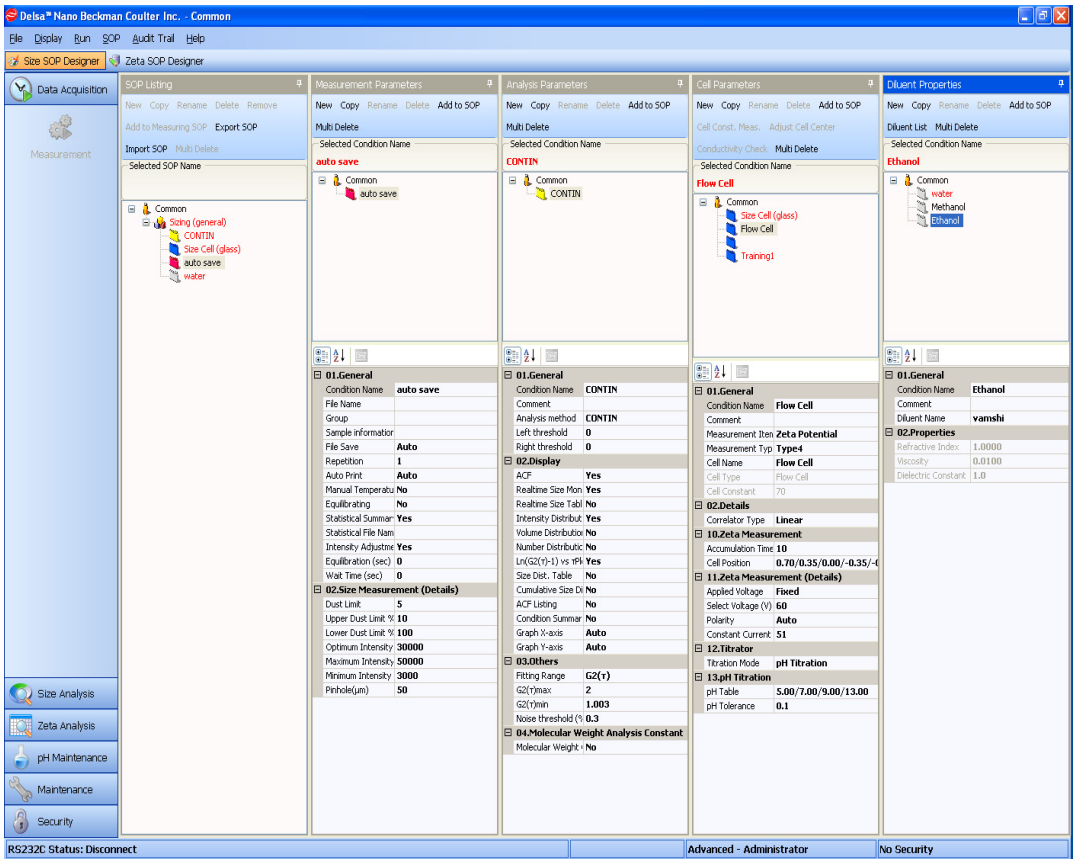


Table 1.6 SOP Designer Window Elements

Window Element	Description
SOP Listing	Displays a list of registered SOPs that have been created for each user name.
Measurement Parameters	Displays measurement parameters that have been created for each user name. Examples of parameters: file name, temperature setting, and the number of repetitions.
Analysis Parameters	Displays analysis parameters that have been created for each user name. Examples of parameters: analysis algorithm and analysis results displays.
Cell Parameters	Displays cell parameters for each user name. This is where you configure the cell used in the measurement.
Diluent Properties	Displays diluent properties, such as diluent name and its refractive index, viscosity, and dielectric constant.

Setting the SOP Designer Display Mode

The details that are displayed in the SOP Designer depend on the selected mode, Normal or Advanced. In Normal mode, only those parameters that must be configured to perform a measurement are displayed. Other parameters are hidden. In Advanced mode, all parameters are displayed and available for adjustment.

To select the display mode, choose one of the following on the Main menu bar:

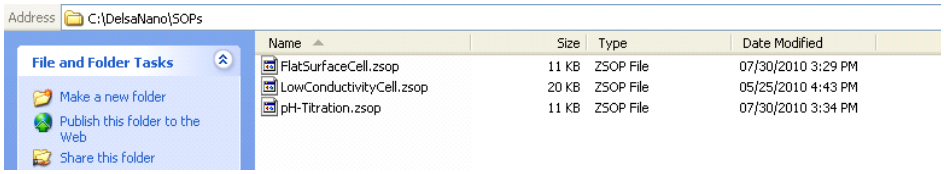
- To use Normal mode, select **Display > Normal mode**.
- To use Advanced mode, select **Display > Advanced mode**.

Working With SOPs

This section describes where default SOPS are located, how to export, import, create, copy, delete SOPs and how to edit SOP names.

Default SOPs

All default SOPs are located on the C:// (hard) drive in a subfolder labeled SOPs in the DelsaNano folder.



Exporting SOPs

To export an SOP:

1. Select **Size SOP Designer** or **Zeta SOP Designer**.
2. Under SOP Listing click **Export SOP**. Export File window opens.
3. Click **Browse** next to the Export File field to open the Save As dialog.
4. Select the destination, enter a file name in the Save As dialog box, and click **Save**.
5. In the Export File window, click **Export File.** A status message at the bottom of the window indicates the completion of the export.
6. Click **Close** to close the Export File window.

Importing SOPs

To import an SOP:

1. Select **Size SOP Designer** or **Zeta SOP Designer**.
2. Under SOP Listing, click **Import SOP**. Import File window box opens.
3. Click **Browse** next to the Import File field to open and search for desired SOP.
4. Select SOP file(s) and click **Open.**
5. In the Import File window, click **Import Selected Files...** A status message at the bottom of the window indicates the completion of the import.
6. Click **Close** to close the Export File window.

Creating SOPs

To create an SOP:

1. Select the desired user name in the SOP Listing, and click **New** above the SOP Listing. Alternatively, select the user name, right-click, and select **New**. A new, blank SOP is created under the user name.
2. Enter a name for the SOP.
3. You can add the required condition in one of these ways:
   - Drag and drop the desired condition from the respective parameters panels into the SOP in the SOP Listing.
   - Select the desired condition, and click **Add to SOP** at the top of each parameter panel. Or, right-click on the condition name and select **Add to SOP**.

⚠ CAUTION

You must include the four conditions in a new SOP: Measurement, Analysis, Cell, Diluent.

Copying SOPs

To copy an SOP:

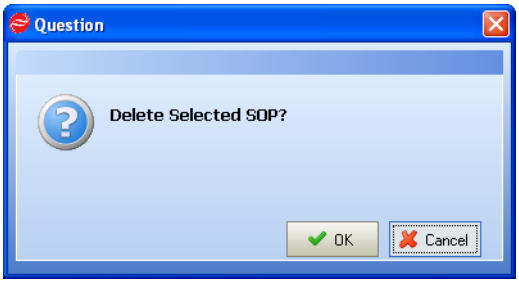
Select the SOP in the SOP Listing, and click **Copy** above the SOP Listing. Or, select the SOP name, right-click, and select **Copy**. An exact copy of the SOP is created under the same user name. The word "copied" and a number are appended to the SOP name to distinguish it from the original.

Deleting SOPs

To delete a single SOP:

1 Select the SOP in the SOP Listing, and click **Delete** above the SOP Listing. Or, select the user name, right-click, and select **Delete**. A confirmation message appears.

Figure 1.14 Delete Selected SOP Confirmation Dialog



2 Click **OK** to delete the SOP.

To delete multiple SOPs:

1 Select the SOP in the SOP Listing and click **Multi Delete**. Multi Delete - SOP window appears.

2 Select the SOPs/Conditions to delete and click **Multi Delete**. A Question window appears.

3 Click **OK** to confirm and **Close** to return to the SOP Designer window.

⚠ CAUTION

Use caution when deleting an SOP. You cannot recover a deleted SOP.

Filenames are not deleted from the database. You cannot use the filename of a deleted SOP.

Editing SOP Names

To edit an SOP name:

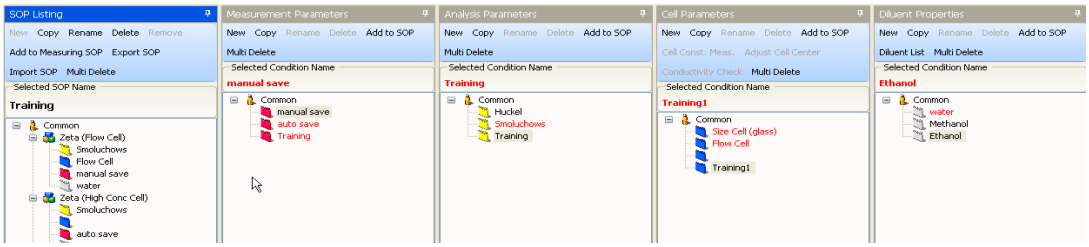
1 Select the SOP in the SOP Listing, and click **Rename** above the SOP Listing. Or, select the SOP name, right-click, and select **Rename**.

2 Modify the name as desired.

Editing a Condition in an SOP

When a condition type (measurement, analysis, cell, or diluent) is registered in an SOP, that condition cannot be modified (it is locked).

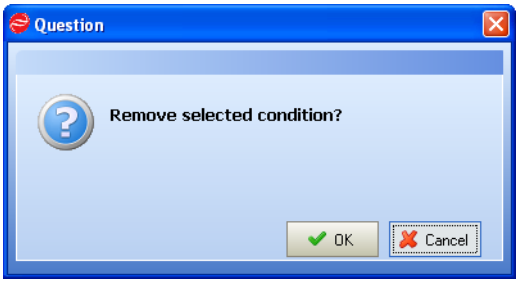
Figure 1.15 Locked Conditions



To edit a condition in an SOP:

1 Remove the condition from the SOP(s) it is registered with. Select the condition, right-click, and select **Remove** or click **Remove** in the SOP Listing. A confirmation message appears. Alternatively, you can make a copy of the condition and edit it in the corresponding panel (Measurement, Analysis, Cell, and Diluent).

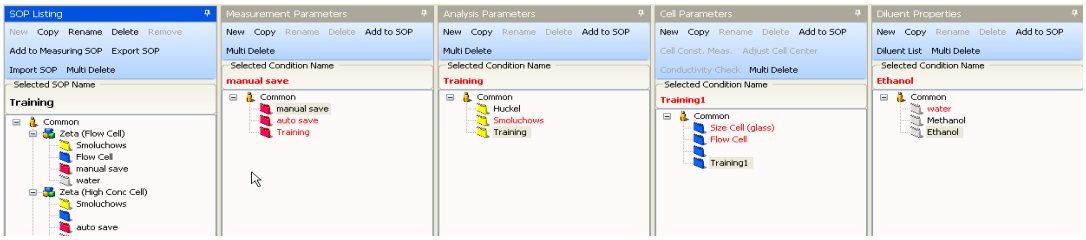
Figure 1.16 Remove Condition Confirmation Dialog



2 Click **OK**.

3 Edit the condition as necessary.

Figure 1.17 Editing a Condition



SOPs for Particle Size Measurements: Measurement Parameters

Figure 1.18 shows the measurement parameters that are set in SOPs for Particle Size Measurements. Parameters in italics are available in Advanced mode only.

Figure 1.18 SOPs for Particle Size Measurements: Measurement Parameters

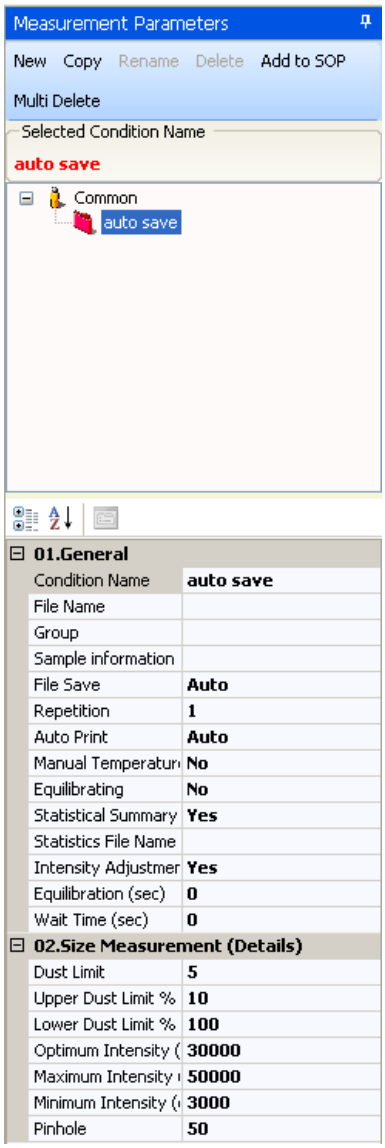


Table 1.7 SOPs for Particle Size Measurements: Measurement Parameters

Parameter Group	Parameter	Description
General	Condition Name	This is a name for identifying the measurement condition. Provide a unique, easily understood name that is different from other condition names. You can enter up to 20 characters.
	File Name	Enter a file name for storing the measured data. You can enter up to 40 characters.
	Group	This is used to create a group in which files can be stored for easy access and searching. In the analysis, the data lists can be sequenced by group. Enter a group name up to 40 characters.

Table 1.7 SOPs for Particle Size Measurements: Measurement Parameters *(Continued)*

Parameter Group	Parameter	Description
	Sample Information	This is used for describing sample information and the measurement parameters. You can enter up to 20 characters.
	File Save	Select Auto to save the data automatically or Manual to save the data manually after the measurements are completed.
	Repetition	Enter the number of times that the analysis is to be repeated. The maximum value allowed is 10,000.
	Auto Print	To print the results automatically after the size measurement is completed, click the button next to "Auto." This opens a Print dialog, where you select the items you want to print. For more information, see *Printing Particle Size Analysis Data*. If you do not want the results printed automatically, leave the options in the Print dialog blank.
	Manual Temperature Setting	This is used to select the measurement temperature. Select Yes, and enter the desired temperature in the Temperature (°C) field, which appears automatically. The temperature range allowed is 5–90°C. If you select No, the measurement is performed at the temperature currently set in the Intensity Monitor. **⚠ CAUTION** **Select No to measure at the same temperature as the current temperature setting. If you select Yes, the temperature setting is performed again at the beginning of the measurement, even if the temperature setting is the same as the current temperature, and some time elapses before temperature stabilization.** **NOTE** For a temperature setting under 10°C, using Nitrogen air is recommended to prevent condensation on the cell surface.
	Equilibrating	If you select Yes, the measurement is not started until the set temperature is reached. Select Yes when measuring at the temperature selected in the "Manual Temperature Setting" field.
	Statistical Summary	Select Yes to save the measurement data in a statistical summary table, then enter the file name in the Statistical File Name field, which appears automatically. You can enter up to 20 characters. You can access the statistical summary table by the selecting the QC icon in the Size Analysis panel.
	Intensity Adjustment	Select Yes to enable the auto intensity adjustment prior to the size measurement.
	Equilibration (sec)	Enter the delay time (in seconds, up to a maximum of 100,000) that will elapse prior to starting a measurement. The equilibration time will start when the run begins, and measurement will start when the equilibration time has elapsed.
	Wait Time (sec)	Enter the wait time (in seconds) between repeated measurements. Enter a value between 0–100,000.

Table 1.7 SOPs for Particle Size Measurements: Measurement Parameters *(Continued)*

Parameter Group	Parameter	Description
Size Measurement (Details)	Dust Limit	This is the amount of time prior to the actual measurement to calculate the average intensity produced by the presence of dust. Enter a value between 0–10. No dust limit will be set if this value is "0."
	Upper Dust Limit %	This is the threshold of the upper limit to be used during the analysis. Enter a value between 0–100. Any data that has an intensity above the upper threshold limit will be ignored. The default value is 10%.
	Lower Dust Limit %	This is the lower dust limit. As with the upper limit, this sets the difference from the average value. Enter a value between 0–100. The default value is 100%, which means this accepts the data lower than average strength as valid data.
	Minimum Intensity (cps)	Enter the minimum intensity allowed in the automatic intensity adjustment. Enter a value between 0–300,000.
	Optimum Intensity (cps)	Enter the optimum intensity allowed in the automatic intensity adjustment. Enter a value between 0–300,000.
	Maximum Intensity (cps)	Enter the maximum intensity allowed in the automatic intensity adjustment. Enter a value between 0–300,000.
	Pinhole (μm)	This sets the pinhole size. The default value is 50 μm, which normally is used without modification. The sizes that can be set for the pinhole are 20, 50, and 100 μm. If it is not possible to obtain an adequate intensity because the sample is small and the concentration is low, set the pinhole to 100 μm. If the intensity is too high at the lowest aperture (ND filter), set to 20 μm. **⚠ CAUTION** **If the pinhole setting is large, the intensity (scattering strength) that can be obtained is increased; however, the state of the coherence will be worse, reducing the signal-to-noise ratio.**

SOPs for Particle Size Measurements: Analysis Parameters

Figure 1.19 shows the analysis parameters that are set in SOPs for Particle Size Measurements. Parameters in italics are available in Advanced mode only.

Figure 1.19 SOPs for Particle Size Measurements: Analysis Parameters

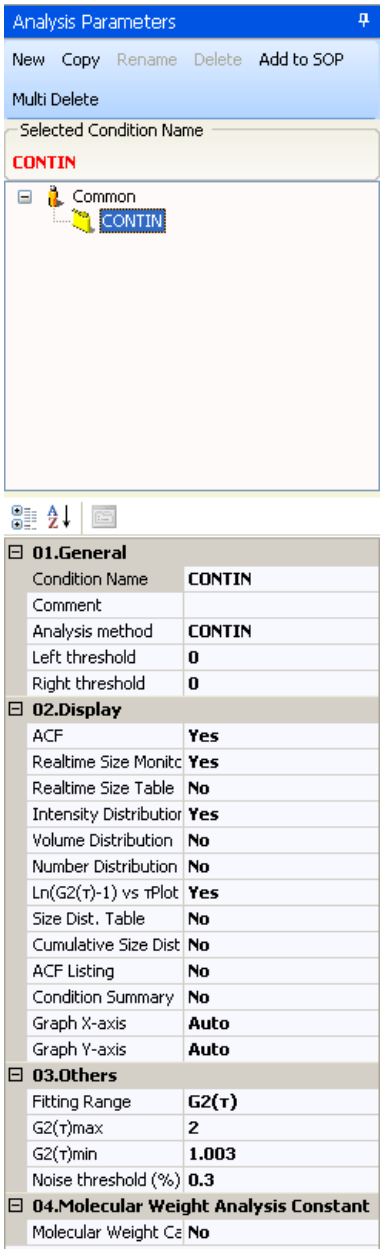


Table 1.8 SOPs for Particle Size Measurements: Analysis Parameters

Parameter Group	Parameter	Description
General	Condition Name	This is a name that identifies the analysis condition. Provide a unique, easily understood name that is different from other condition names. You can enter up to 20 characters.
	Comment	You can enter a comment about the analysis parameters. Enter up to 20 characters.
	Analysis Method	Select the algorithm that will be used in the particle size distribution analysis. There are three different analysis algorithms: CONTIN, Marquardt, NNLS. For information on these analysis algorithms, see the *Introduction*.
	Left Threshold	Enter the specific number of channels to be removed on the left side of particle size distribution. Up to 45 channels can be removed. This is useful for removing unwanted distribution due to noise.
	Right Threshold	Enter the specific number of channels to be removed on the right side of particle size distribution. Up to 45 channels can be removed. This is useful for removing unwanted distribution due to clumps/agglomerates.
Display	ACF	Select Yes to display a second-order autocorrelation function plot.
	Realtime Size Monitor	Select Yes to display a plot of the particle size values in each integration cycle (accumulation times) during a measurement. This makes it possible to confirm the stability of the sample, such as whether or not the particle size changes over time during the measurement. For more information and an example of a Realtime Size Monitor display, see APPENDIX D, *Graphs and Table Displays*.
	Realtime Size Table	Select Yes to display a table of the particle size values in each integration cycle (accumulation times) during a measurement. For more information and an example of a Realtime Size Table display, see APPENDIX D, *Graphs and Table Displays*.
	Intensity Distribution	Select Yes to display the particle size distribution (the intensity distribution) showing both differential and cumulative. For more information and an example of an Intensity Distribution display, see APPENDIX D, *Graphs and Table Displays*.
	Volume Distribution	Select Yes to display the particle size distribution (the volumetric conversion distribution) showing both differential and cumulative. For more information and an example of a Volume Distribution display, see APPENDIX D, *Graphs and Table Displays*.

Table 1.8 SOPs for Particle Size Measurements: Analysis Parameters *(Continued)*

Parameter Group	Parameter	Description
	Number Distribution	Select Yes to display the particle size distribution (the numeric conversion distribution) showing both differential and cumulative. For more information and an example of a Number Distribution display, see APPENDIX D, *Graphs and Table Displays*.
	Ln (G2(τ)-1) vs τ Plot	Select Yes to display a second-order autocorrelation function logarithmic plot.
	Size Dist. Table	Select Yes to display the particle size frequency distribution table.
	Cumulative Size Dist. Table	Select Yes to display the particle size distribution cumulative frequency table.
	ACF Listing	Select Yes to display a second-order autocorrelation function table.
	Condition Summary	Select Yes to display extracts of the measurement parameters, analysis parameters, cell parameters, and diluent properties, including the SOP name and file name.
	Graph X-Axis	This is used to switch the particle size distribution graph X axis (particle size range) between the automatic and manual settings or a pre-selected size range (0.1-1000 nm or 1-10,000 nm). For manual settings, the upper limit (X) and lower limit (X) will be displayed. Input the range, which can be between 0.1–10,000 nm. This is useful if you want to see a size distribution within a specified range.
	Upper Limit (X)	Enter the maximum value for the particle size distribution graph X axis (particle size range). This parameter is visible when Graph X-Axis is set to a fixed scale (10,000 nm).
	Lower Limit (X)	Enter the minimum value for the particle size distribution graph X axis (particle size range). This parameter is visible when Graph X-Axis is set to a fixed scale (0.1 nm).
	Graph Y-Axis	This is used to switch the particle size distribution graph Y axis (frequency) between the automatic and manual settings. For manual settings, the upper limit (Y) will be displayed. Enter the range (1–100).
	Upper Limit (Y)	Enter the maximum value for the particle size distribution graph Y axis (frequency). This parameter is visible when Graph Y-Axis is set to a fixed scale.
Others	Fitting Range	Select the axis fitting range of G2(τ), or select the range of τ. Then, enter the corresponding maximum and minimum values in the two fields below this one.
	G2(τ) max	For fitting range G2(τ), enter the upper limit to be used for calculating the size distribution. The maximum value allowed is 2.

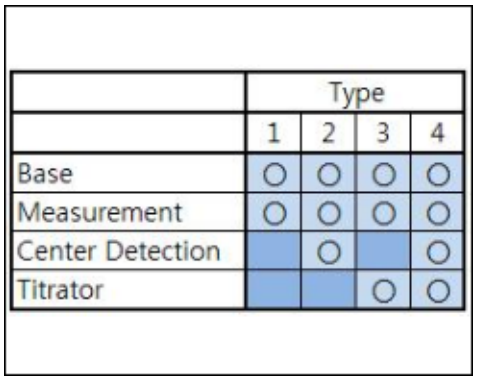
Table 1.8 SOPs for Particle Size Measurements: Analysis Parameters *(Continued)*

Parameter Group	Parameter	Description
	G2(τ) min	For fitting range G2(τ), enter the lower limit to be used for calculating the size distribution. The minimum value allowed is 1.
	τ max	For fitting range τ, enter the maximum value to be used in fitting (maximum 983040 µs).
	τ min	For fitting range τ, enter the minimum value to be used in fitting (minimum 0.1 µs).
	Noise Threshold (%)	Set the lower limit value on the frequency (0–100%) of the peak displayed in the graph in the particle size distribution graph. A peak that is lower than this lower limit value is handled as noise, and is not displayed on the particle size distribution graph. The default value is 0.3%.
Molecular Weight Analysis Constants	Molecular Weight Calculation	This is used to determine the molecular weight of the sample. If you select Yes, you are prompted to enter the a and b constants pertaining to the sample. For more information, see APPENDIX C, *Alpha and Beta Values*.
	α	Enter the a value of the sample ranging from 1E-09 to less than 0.1.
	β	Enter the b value of the sample ranging from 0.3 to 1.

SOPs for Particle Size Measurements and Zeta Potential Measurements: Cell Parameters

In the cell parameters, the parameters that are displayed differ depending on the measurement items and the measurement types. There are several measurement types available for each cell (see Figure 1.20). Each measurement type includes specific routines and/or tests to perform during a measurement.

Figure 1.20 Measurement Types



The parameters that are displayed for each measurement item (particle size and zeta potential) are shown below. Additionally, the types of parameters that are displayed will differ depending on the measurement type, even given the same measurement item, so a numeric value indicating the item that appears in the measurement type is noted after the parameter name.

Figure 1.21 shows the cell parameters that are set in SOPs for Particle Size and Zeta Potential Measurements. Parameters in italics are available in Advanced mode only.

Figure 1.21 SOPs for Particle Size and Zeta Potential Measurements: Cell Parameters

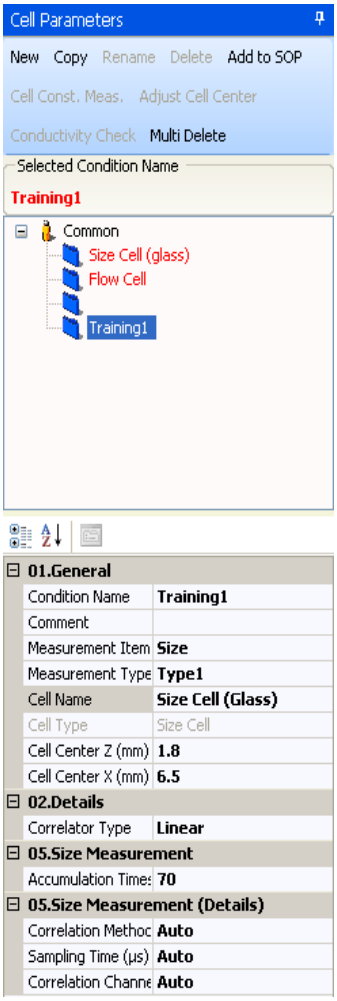


Table 1.9 SOPs for Particle Size Measurements and Zeta Potential Measurements: Cell Parameters

Parameter Group	Parameter	Description
Parameters for both Measurement Items (Particle Size and Zeta Potential)		
General	Condition Name	This name identifies the cell condition. Provide an easily understood name up to 20 characters that can differentiate from other cell condition.
	Comment	You can enter a comment about the cell parameters. Enter up to 20 characters.
	Measurement Item	Select Particle Size or Zeta Potential for the measurement item.

Table 1.9 SOPs for Particle Size Measurements and Zeta Potential Measurements: Cell Parameters

Parameter Group	Parameter	Description
	Cell Name	Select, from the dialog, the cell that is to be used. The registration of cells is performed in the Maintenance section of the software. **⚠ CAUTION** **Before using the High Concentration cell, make sure the sample conductivity is lower than ~3 mS/cm.**
	Cell Type	This displays the type of cell that will be used.
	Cell Center X (mm)	This shows the optimal position of the cell on the X axis. The value will be the default X value for that cell, unless a cell center adjustment has been performed and saved. The saved cell center adjustment will be displayed here. See *Detecting the Optimal Cell Position*. This disappears when either Type 2 or Type 4 is selected.
	Cell Center Z (mm)	This shows the optimal position of the cell on the z axis. The value will be the default Z value for that cell, unless a cell center adjustment has been performed and saved. The saved cell center adjustment will be displayed here. See *Detecting the Optimal Cell Position*. This disappears when either Type 2 or Type 4 is selected.
Parameters if the Measurement Item is “Size”		
General	Measurement Type	Type 1: Measures only the particle size.
		Type 2: Starts the measurement after automatic detection of the optimal cell position at the time of measurement. The optimal position for the cell can also be detected in advance. For details, see *Detecting the Optimal Cell Position*.
		Type 3: Performs the particle size measurement after using the titrator to adjust the pH and other parameters.
		Type 4: Performs the particle size measurement after using the titrator to adjust the pH and other parameters and performing automatic detection of the optimal cell position. **⚠ CAUTION** **Type 3 and Type 4 are available only when the Auto Titrator is configured on the system.**

Table 1.9 SOPs for Particle Size Measurements and Zeta Potential Measurements: Cell Parameters

Parameter Group	Parameter	Description
Details	Correlator Type	Log correlator is the default correlator. Log correlator covers a wide range of decay times and can be used to measure any sample having broad distribution. Linear correlator has high resolution within the limited range of decay time and thus it is suitable for very small particles of narrow distribution. You can use Linear correlator if you have prior knowledge of the sample.
Size Measurement	Accumulation Times	Enter the number of integration cycles for the measurement. The default value is 70. At this value, the software determines 70 ACFs and calculates 70 realtime diameters from each ACF and finally calculates one averaged ACF from 70 ACFs to give the mean diameter. Increase the number of cycles if the intensity of the sample is weak or if the signal is too noisy to obtain a stable autocorrelation function. If the intensity is less than 10,000 cps, use the following formula as a guideline to input the number of integrating cycles. Number of integration cycles = 10,000 (cps)/intensity of the sample (cps) × 10.
Size Measurement (Details)	Correlation Method	Select either Time Domain (TD), Time Interval (TI), or Auto. This parameter is valid only when the Linear correlator is selected. Generally, you select TD for large particles having stronger scattering levels, and you select TI for small particles of less than 10 nm having weaker scattering levels. If you are not sure what to use, select Auto.
	Sampling Time (μs)	Select the sampling time from the list. This parameter is valid only when the Linear correlator is selected. If you don't know what to use, select Auto. The sampling time is the time after which the signal is collected from the sample by the instrument. Appropriate sampling time must be selected to get a smoother ACF and for accurate size measurement.
	Correlation Channel	Select the correlation channel of the correlation calculation from the list. This parameter is valid only when the Linear correlator is selected. If you don't know what to use, select Auto. The correlation channel represents the number of bins into which the size data is distributed. It is the size resolution (the more channels, the more divisions on the size scale, i.e., X-Axis.

Table 1.9 SOPs for Particle Size Measurements and Zeta Potential Measurements: Cell Parameters

Parameter Group	Parameter	Description
Titrator	Titration Mode (3 & 4)	Select this parameter to carry out the pH titration vs. size, diluting/priming, or circulation of the sample. **NOTE** Titration Mode is applicable only when you are using a flow cell or Size cell (Flow).
pH Titration	pH Table (3 & 4)	This parameter is valid when pH titration is selected as the operating mode of the Auto Titrator. The Auto Titrator can titrate from pH 1–13. The pH values can be entered from low to high or from high to low. Set the pH value to be adjusted.
	pH Tolerance (3 & 4)	This parameter is valid when pH titration is selected as the operating mode of the Auto Titrator. Set the tolerance value for the pH adjustment. The pH value is considered to have been completed if the pH is in the range (the pH setting ± the tolerance value). It ranges from 0.05–2.
Pipetting	Titration Volume Table (3 & 4)	This parameter is valid when Pipetting is selected as the operating mode of the Auto Titrator. This sets the volume of the additive to be added. The additive volume range is 1–50,000 ml.
	Circulation Time (min) (3 & 4)	This parameter is valid when Pipetting is selected as the operating mode of the Auto Titrator. This sets the time for the sample to circulate around the flow cell after adding additive. The circulation time range is 1–999 minutes.
Circulation	Circulation Time Table (3 & 4)	This parameter is valid when circulation is selected as the operating mode of the Auto Titrator. This sets the time for the sample to circulate around the flow cell. The circulation time range is 1–999 minutes.
Parameters if the Measurement Item is “Zeta Potential”		
General	Measurement Type	Type 1: Base+Measurement: Performs base measurement first, then actual zeta potential measurement. Base refers to the modulator frequency that acts as a reference. The base measurement is required before measuring zeta potential. The base frequency is dependent on the type of cell used and the concentration of the sample. The base frequency for Flow cell, Flat Surface cell, Disposable Zeta cell, and Low Conductivity cell ranges from 110–140 Hz and from 220–270 Hz for High Concentration cell. It can be affected by other electrical devices near the DelsaNano. For more information and an example of an ACF (Base) graph, see *ACF (Base)*.
		Type 2: Center Detection+Base+Measurement: Performs center detection, base measurement, and sample zeta potential measurement.

Table 1.9 SOPs for Particle Size Measurements and Zeta Potential Measurements: Cell Parameters

Parameter Group	Parameter	Description
		Type 3: Titrator+Base+Measurement: Performs either pH titration, pipetting or circulation, then base measurement and sample zeta potential measurement.
		Type 4: Titrator+Center Detection+Base+Measurement: Performs either pH titration, pipetting or circulation, then center detection, base measurement and sample zeta potential measurement. **⚠ CAUTION** **Type 3 and Type 4 are available only when the Auto Titrator is configured on the system**
	Cell Constant	Displays the cell constant, determined by using an electrical conductivity standard solution. See *Measuring the Cell Constant* for the method of determining the cell constant. Upon cell selection, the default cell constant appears. The cell constant must be measured when the cell is replaced, or when the electrodes are replaced.
Detail	Correlator Type	This is used to select the correlator for zeta potential measurement. The default correlator is Linear.
Zeta Measurement	Accumulation Times	This is the number of times the instrument measures the zeta potential at a particular position. The default value is 10 for flow cell and Flat Surface cell, and 20 for High Concentration cell; however, it can be changed.
	Cell Position	This sets the position of the cell when the measurement is performed. The setting will vary, depending on the cell that is used, and it can be changed. The typical settings are: Flow cell and Disposable Zeta cell: 5 points: 0.7/0.3/0/-0.35/-0.7 High Concentration cell and Low Conductivity cell: 1 point: 0 Flat Surface cell: 7 points: 0.8/0.6/0.3/0/-0.3/-0.6/-0.8 During the measurement, the instrument measures zeta potential up to five different locations to eliminate the effects of Brownian motion. The number of positions can vary from -1 to +1. The points can be in ascending or descending order.
Zeta Measurement Detail	Applied Voltage	Select how you want the applied voltage to be set, automatically or manually. If you don't know what to use, select Auto. When you select Fixed, you are prompted to enter the DC voltage.
	Applied Voltage (Fixed)	This is used for inputting the voltage value if the applied voltage is to be set manually. The DC voltage range is 0–300 V.

Table 1.9 SOPs for Particle Size Measurements and Zeta Potential Measurements: Cell Parameters

Parameter Group	Parameter	Description
	Polarity	This is used to select the polarity of the applied voltage from positive, negative, or automatic on the reference electrode. If you don't know what to use, select Auto.
	Maximum Current (mA)	This allows you to set the maximum amount of current that flows through the sample cell during measurement.
Titrator	Titration Mode (3 & 4)	This is selected to carry out the pH titration vs. size, diluting/priming, or circulation of the sample. This applies only if the cell type is flow cell.
pH Titration	pH Table (3 & 4)	This parameter is valid when pH titration is selected as the operating mode of the Auto Titrator. The Auto Titrator can titrate from pH 1–13. You can enter the pH values from low to high or from high to low. Set the pH value to be adjusted.
	pH Tolerance (3 & 4)	This parameter is valid when pH titration is selected as the operating mode of the Auto Titrator. Set the tolerance value for the pH adjustment. The pH value is considered to have been completed if the pH is in the range (the pH setting ± the tolerance value). It ranges from 0.05–2.
	Circulation Time (min) (3 & 4)	This is used to set the time for the pH adjustment. The adjustment is considered to have been completed if this set time has elapsed, even if the pH value is not within the range of the tolerance value. Moreover, the sample is circulated until the set time has elapsed, without advancing to the next operation, even if the pH value is within the tolerance range when within this time.
Additive Titration	Titration Volume Table (3 & 4)	This parameter is valid when additive titration is selected as the operating mode of the Auto Titrator. This sets the volume of the additive to be titrated.
	Circulation Time (3 & 4)	This parameter is valid when additive titration is selected as the operating mode of the Auto Titrator. This sets the sample circulation time at the time of additive titration.
Circulation	Circulation Time Table (3 & 4)	This parameter is valid when circulation is selected as the operating mode of the Auto Titrator. This sets the circulation time.

SOPs for Particle Size Measurements and Zeta Potential Measurements: Diluent Properties

Table 1.10 describes the parameters that are set in the Diluent Properties section of the SOP Designer windows. Parameters in italics are available in Advanced mode only.

Table 1.10 SOPs for Particle Size Measurements and Zeta Potential Measurements: Diluent Properties

Parameter Group	Parameter	Description
General	Condition Name	This is a name for identifying the diluent. The diluent name can be used as the condition name. Provide a unique, easily understood name up to 20 characters that is different from other condition names.
	Comment	You can enter a comment about the diluent. Enter up to 20 characters.
Properties	Diluent Name	Select the diluent name from the drop-down menu.
	Refractive Index	This displays the Refractive Index of the selected diluent.
	Viscosity	This displays the viscosity of the selected diluent.
	Dielectric Constant	This displays the Dielectric Constant of the selected diluent.

Adding a Diluent to the Diluent List

If the diluent property you want is not in the diluent list, you can add a new diluent by following the steps in this procedure.

To add a new diluent to the diluent list:

1. In the Diluent Properties panel of the Size SOP Designer or Zeta SOP Designer, click **Diluent List**. The Select Diluent List dialog appears.

2. In the Select Diluent List dialog, select **New** on the menu bar, and enter the diluent name in the Diluent Name field.

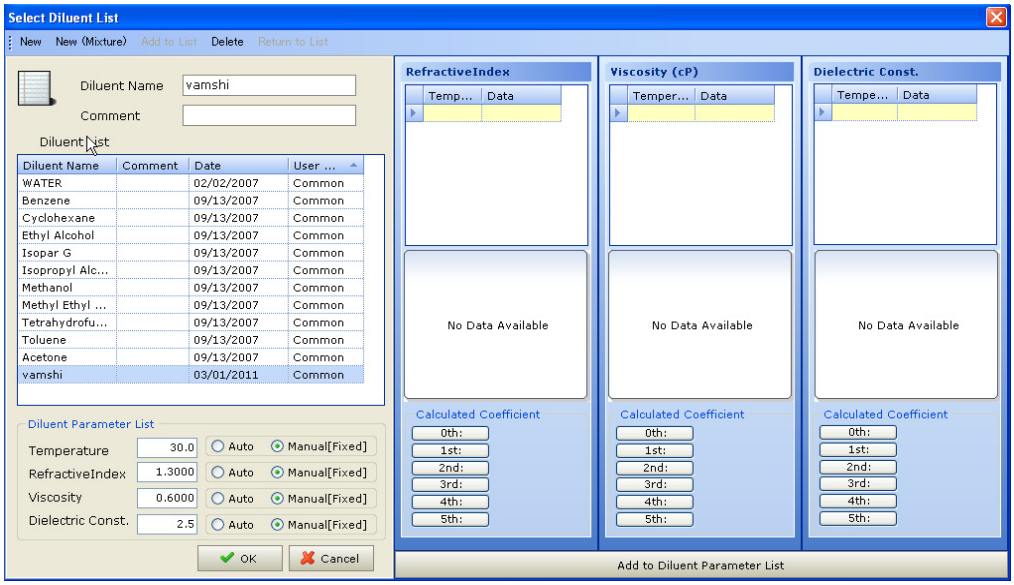
3. Optionally, enter a comment in the Comment field (To return to the diluent list without adding a new diluent, click **Return to List**).

4 To add the Refractive Index, Viscosity, and Dielectric Constant of the diluent to the list, as follows.

- In Case 1 (Figure 1.22):

  If the parameters of the diluent are known only at a certain temperature (for example, 30° C), input the temperature as 30° C and the corresponding Refractive Index, Viscosity, and Dielectric Constant values of the diluent. Select Manual [Fixed] next to each parameter, and select **Add to List** on the menu bar. Click **OK** to close the dialog.

  Figure 1.22 Adding a Diluent, Case 1



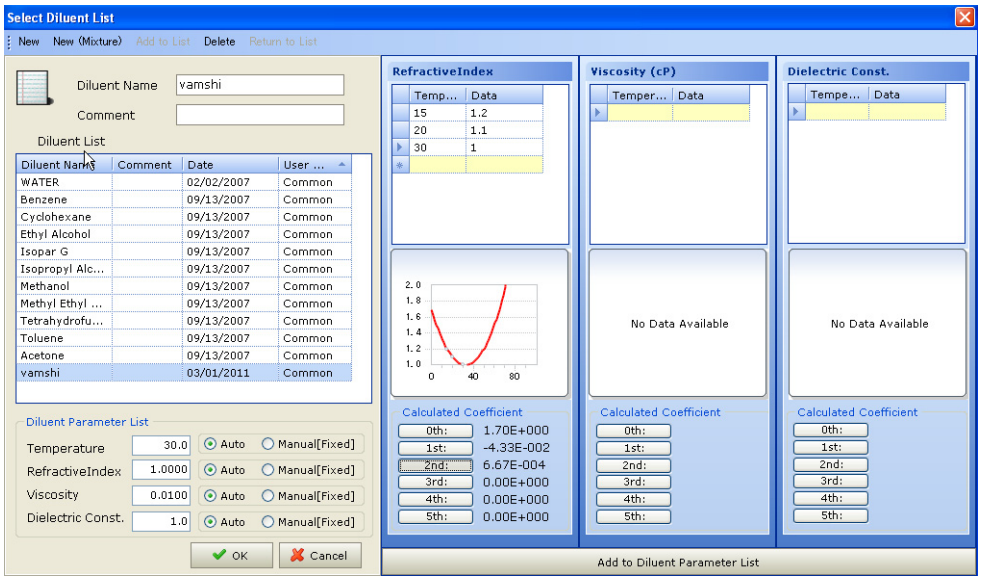
- In Case 2 (Figure 1.23):

  If the parameters of the diluent are known at a different temperature, a table can be built (such that the software can use any of these parameters for the temperatures at which the parameters are not directly available) from the fitting curves to determine the particle size or zeta potential.

  To create the table using Refractive Index as an example, input the Refractive Indices of the diluent for the known temperatures (for example, 15° C, 20° C, and 30° C).

  To obtain the Refractive Index of the diluent at a different temperature (for example, 25° C, which is not listed in the table), input the temperature as 25° C in the Temperature column, and select Auto next to the parameter. Use one of the Calculated Coefficients that best fits the Refractive Index, and click **Add to Diluent Parameter List**. Select **Add to List** on the menu bar, and click **OK** to close the dialog.

Figure 1.23 Adding a Diluent, Case 2



NOTE The software automatically picks the values of the Diluent Refractive Index, Viscosity, and Dielectric Constant from the table for the temperature at which these values are not available, provided the table is built for that diluent.

Calculating the Diluent Properties of a Mixture

To calculate the diluent properties of a mixture:

1. In the Diluent Properties panel of the Size SOP Designer or Zeta SOP Designer, click **Diluent List**. The Select Diluent List dialog appears.

2. In the Select Diluent List dialog, select **New** (Mixture) on the menu bar, and enter the diluent name in the Diluent Name field.

3. Select Diluent 1 (Water or others). If "others" is selected, highlight the diluent in Diluent 1 table. Similarly highlight the diluent in Diluent 2 table.

4. Enter Diluent 2 Contents (wt% or mM) and Temperature (°C).

5. Click Calculation.

Deleting a Diluent

To delete a diluent:

1 In the Diluent Properties panel of the Size SOP Designer or Zeta SOP Designer, click **Diluent List**. The Select Diluent List dialog appears.

2 Select the name of the diluent you want to delete from the diluent list.

3 Select **Delete** on the menu bar.

⚠ CAUTION

You cannot recover a diluent that has been deleted. Default diluents cannot be deleted.

SOPs for Zeta Potential Measurements: Measurement Parameters

Figure 1.24 shows the measurement parameters that are set in SOPs for Zeta Potential Measurements. Parameters in italics are available in Advanced mode only.

Figure 1.24 SOPs for Zeta Potential Measurements: Measurement Parameters

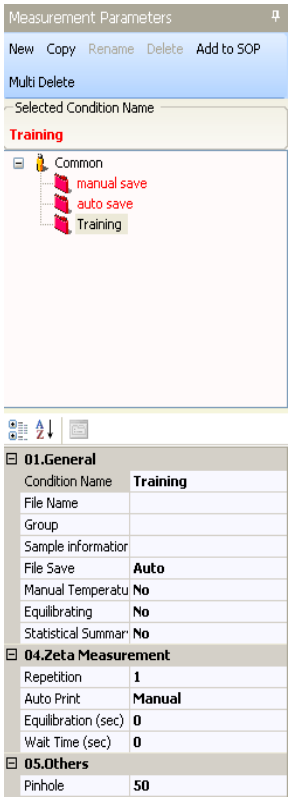


Table 1.11 SOPs for Zeta Potential Measurements: Measurement Parameters

Parameter Group	Parameter	Description
General	Condition Name	This is a name for identifying the measurement condition. Provide a unique, easily understood name up to 20 characters that is different from other condition names.
	File Name	This is the file name for storing the measured data. Enter a file name up to 40 characters. Spaces are not allowed.
	Group	This is used to identify samples. In the analysis, the data lists can be sequenced by group. Enter a group name up to 20 characters.
	Sample Information	This is used to describe sample information and the measurement parameters. Enter a description up to 40 characters.
	File Save	Select Auto to save the data automatically or Manual to save the data manually after the measurements are completed.
	Manual Temperature Setting	This is used to select the measurement temperature. Select Yes, and enter the desired temperature in the Temperature (° C) field, which appears automatically. The temperature range allowed is 5–90° C. If you select No, the measurement is performed at the temperature currently set in the Intensity Monitor. **⚠ CAUTION** **Select No to measure at the same temperature as the current temperature setting. If you select Yes, the temperature setting is performed again at the beginning of the measurement, even if the temperature setting is the same as the current temperature, and some time will elapse before temperature stabilization.**
	Equilibrating	If you select Yes, the measurement will not be started until the set temperature is reached. Select Yes when measuring at the measurement temperature in the “Manual Temperature Setting” field.
	Statistical Summary	Select Yes to save the measurement data in a statistical summary table, then enter the name of the file you want in the Statistical File Name field, which appears automatically. You can enter up to 20 characters.
	Statistical File Name	Enter the name of the statistical file. If an identical statistical file name exists, the data is appended to the end of that file.

Table 1.11 SOPs for Zeta Potential Measurements: Measurement Parameters *(Continued)*

Parameter Group	Parameter	Description
Zeta Measurement	Repetition	This is the number of times that the measurement is repeated. The maximum value allowed is 10,000.
	Auto Print	If you want the results printed automatically after the measurement is completed, click the button next to "Auto." This opens the Print dialog, where you select the items you want to print. For more information, see *Printing Zeta Potential Analysis Results*. If you do not want the results printed automatically, leave the options in the Print dialog blank.
	Equilibration (sec)	Enter the delay time (in seconds, up to a maximum of 100,000) that elapses prior to starting a measurement. The equilibration time starts when the run begins, and measurement will start when the equilibration time has elapsed.
	Wait Time (sec)	Enter the wait time (in seconds, up to a maximum of 10,000) between repeated measurements.
	Pinhole (μm)	This sets the pinhole size. The default value is 50 μm, which normally is used without modification. The sizes that can be set for the pinhole are 20, 50, and 100 μm. If it is not possible to obtain an adequate intensity because the sample is small and the concentration is low, set the pinhole to 100 μm. If the intensity is too high at the lowest aperture (ND filter), set to 20 μm. **⚠ CAUTION** **If the pinhole setting is large, the intensity (scattering strength) that can be obtained is increased; however, the state of the coherence will be worse, reducing the signal-to-noise ratio.**

SOPs for Zeta Potential Measurements: Analysis Parameters

Figure 1.25 shows the analysis parameters that are set in SOPs for Zeta Potential Measurements. Parameters in italics are available in Advanced mode only.

Figure 1.25 SOPs for Zeta Potential Measurements: Analysis Parameters

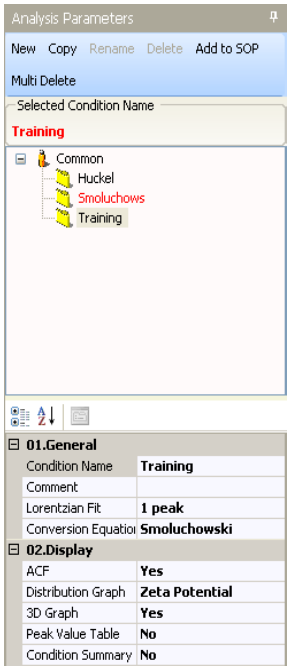


Table 1.12 SOPs for Zeta Potential Measurements: Analysis Parameters

Parameter Group	Parameter	Description
General	Condition Name	This is a name for identifying the analysis condition. Provide a unique, easily understood name up to 20 characters that is different from other condition names.
	Comment	You can enter a comment about the analysis parameters. Enter up to 20 characters.
	Lorentzian Fit	This is used to set the number of peaks when making a Lorentzian fit with the zeta potential distribution. *In the distribution graph, the Brownian motion of the particles is characterized by a Lorentzian peak centered at a frequency shift that characterizes Electrophoretic mobility of the particles.* If the sample is a mixture of particles of different mobility, e.g., 2, then 2 peaks can be selected for the Lorentzian Fit. The default selection is 1 peak that corresponds to the fitted data. When None is selected, the distribution graph displays the raw data; i.e., intensity zeta potential/mobility distribution.

Table 1.12 SOPs for Zeta Potential Measurements: Analysis Parameters *(Continued)*

Parameter Group	Parameter	Description
	Conversion Equation	This is used to select the equation for calculating the zeta potential from the mobility. You may select Smoluchowski, Huckel, or Other. Generally, Smoluchowski is used for aqueous samples and Huckel is used for organic samples. A coefficient must be input when you select Other. This coefficient is substituted for the “k” in the following equation: $Z = k\frac{\pi\eta}{\varepsilon}U$ Z: Zeta potential, η: Viscosity of the solution, ε: Dielectric constant of the solution, U: Mobility
Display	ACF	Select Yes to display a second-order autocorrelation function plot. For more information, see the *Introduction*.
	Distribution Graph	Select Yes to display a graph of the zeta potential measurement results. It is possible to select whether the display will display using the zeta potential or using the mobility. For more information and an example of a Distribution Graph, see APPENDIX D, *Graphs and Table Displays*.
	3D Graph	Select Yes to display the profile for the velocity of movement of the particles, including the electrosmosis. The 3D Graph can be viewed for mobility of the particles. For more information and an example of a 3D Graph, see APPENDIX D, *Graphs and Table Displays*.
	Peak Value Table	Select Yes to display a table of the apparent mobility and zeta potential results. For more information and an example of a Peak Value Table, see APPENDIX D, *Graphs and Table Displays*.
	Condition Summary	Select Yes to display extracts of the measurement parameters, analysis parameters, cell parameters, and diluent properties, including the SOP name and file name.

Other Functions of the SOP Designer

This section describes how to detect the optimal cell position, how to measure the cell constant, and how to measure the electrical conductivity.

Detecting the Optimal Cell Position

The measurement cell can be positioned to obtain an optimum intensity for measurement. This value is saved in the cell parameter.

Perform Cell Center adjustment under the following conditions:

- when a new cell parameter is created
- the first time a cell is used in a measurement
- if the concentration or sample type changes

You can repeat the Cell Center adjustment if the intensity monitor shows the sample concentration is too low.

To detect the optimal cell position:

---

1 In the Cell Parameters, click **Adjust Cell Center**. The Cell Center Detection dialog opens.

Figure 1.26 Cell Center Detection Dialog

2 Click **Start**. The optimal position for measurement is detected automatically, depending on the selected cell type. After the detection has been completed, the results appear in the Result pane.

3 Click **OK** to store the calculated cell position in the cell parameters. If you click **Cancel**, the previous center (the cell position from the previous time) remains stored as is, and the cell moves to the previous location.

You can see the following:

- The variation of intensity of the cell position
- The attenuator percentage
- The previous and new cell centers

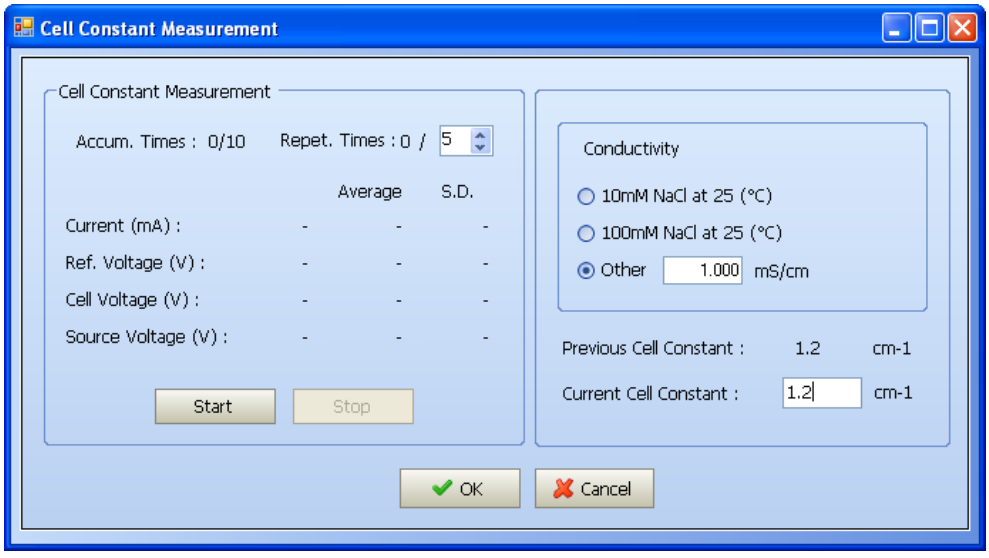
Measuring the Cell Constant

When you click **Cell Const. Meas.** in the cell parameters, the Cell Constant Measurement screen opens. The number of measurement cycles is set by Rept. Times, and the conductivity standard used can be selected on the right-hand side of the display under Conductivity. When measuring the cell constant using a solution other than the 10 mM or 100 mM NaCl solution, place a checkmark next to Other and input the conductivity of the solution used.

The cell constant measurement begins when you click **Start** (to stop the measurement, click **Stop**). When the measurement has been completed, the cell constant appears as the Current Cell Constant, and the previous cell constant is also displayed. The cell constant is saved in the cell parameters when you click **OK**. If canceled, the cell constant is not saved, and the previous cell constant remains as-is.

Perform a cell constant when you create a new cell parameter, when a cell is used for the first time, or when the electrodes have been replaced.

Figure 1.27 Cell Constant Measurement Screen

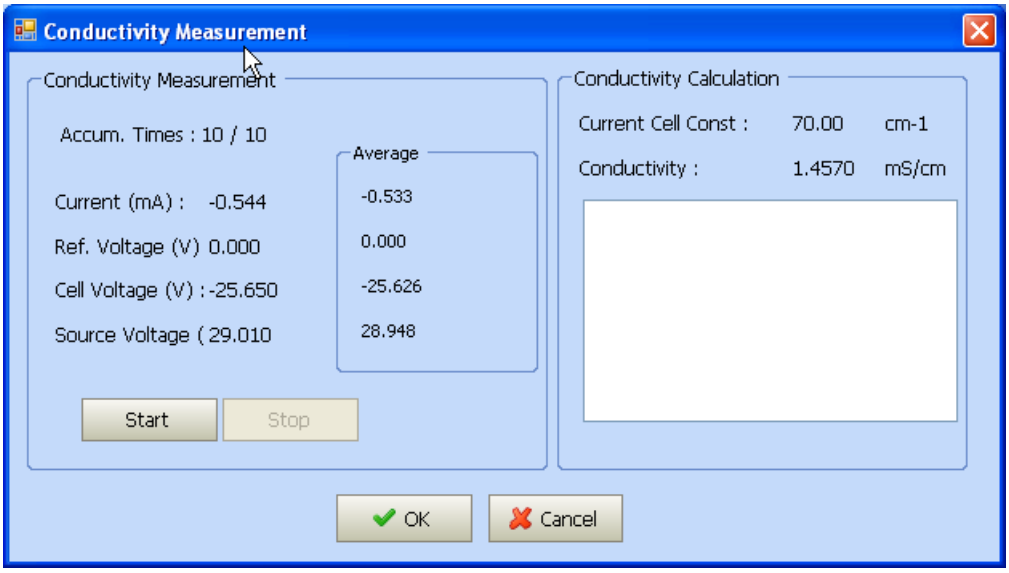


Measuring the Electrical Conductivity

The following dialog opens when you click **Conductivity Check** in the cell parameters. Click **Start** to start the measurement (to stop it, click **Stop**), and the measurement results for the conductivity of the sample will be displayed. To cancel the operation prior to completion, click **Cancel**.

Perform the conductivity check when the concentration of your sample changes or the sample type is unknown.

Figure 1.28 Conductivity Measurement Dialog



Saving to Favorite SOPs

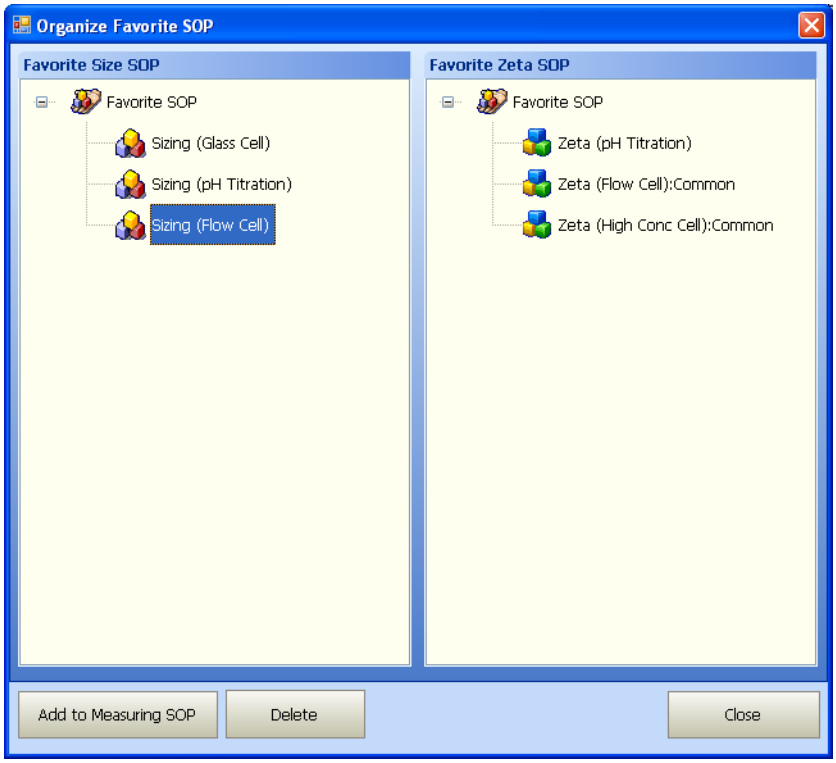
You can save regularly-used SOP as a “Favorite SOP” or “Favorite SOP Series.”

Saving an SOP to Your Favorite SOP List

To save an SOP to your Favorite SOP list:

Select the SOP from either Measuring SOP, Particle SOP listing, or Zeta SOP listing, and select **SOP > Favorite SOP > Add to Favorite SOP** on the Main menu bar.

Figure 1.29 Favorite SOP List Example

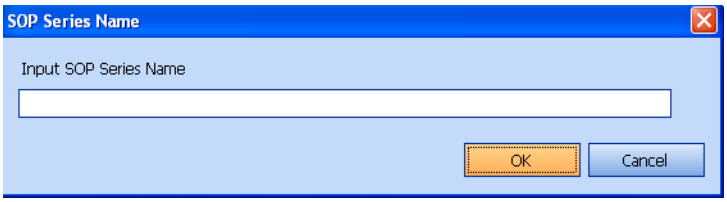


Saving an SOP to Your Favorite SOP Series

To save an SOP to your Favorite SOP Series:

1 Select the SOP from either Measuring SOP, Particle SOP listing, or Zeta SOP listing, and select **SOP > Favorite SOP > Add to Favorite SOP Series** on the Main menu bar.

SOP Series Name window opens.



2 Enter an SOP Series Name and Click **OK**.

Calling a Favorite SOP

If multiple SOPs are generated and saved, the SOP can be read out at the time of the next measurement and saved as a Selected SOP.

To call an SOP from your Favorite SOP list:

1. Select **SOP > Favorite SOP > Organize Favorite SOP** on the Main menu bar. Organize Favorite SOP dialog box appears.

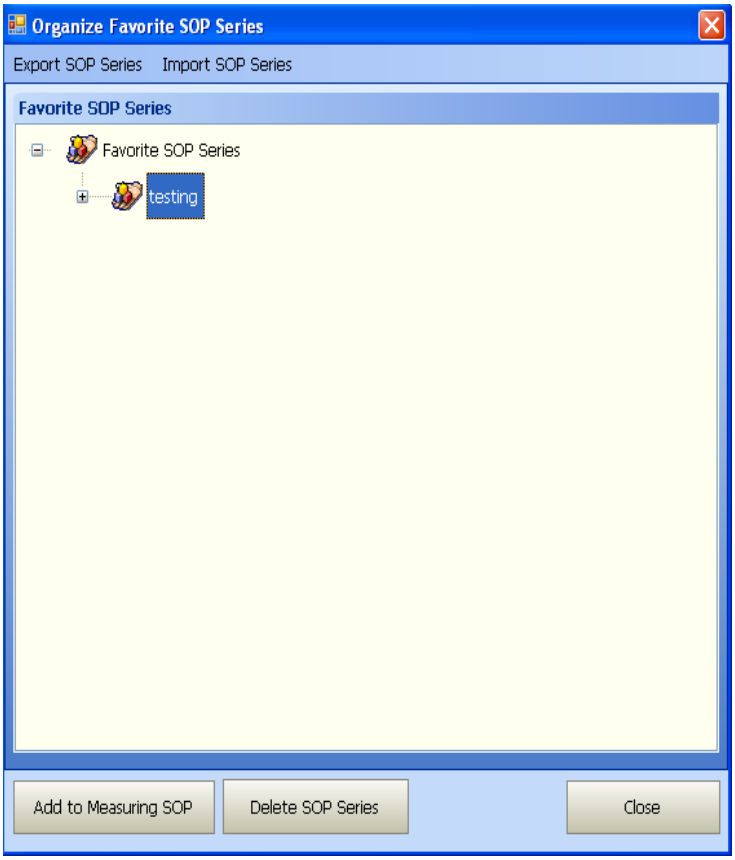
2. Select an SOP, and click **Add to Measuring SOP** to store that SOP as a Selected SOP.

Calling and Deleting a Favorite SOP Series

To call or delete an SOP from your Favorite SOP Series:

1. Select **SOP > Favorite SOP > Organize Favorite SOP Series** on the Main menu bar.

   Organize Favorite SOP Series dialog box appears.



2 Select an SOP Series, and click **Add to Measuring SOP** to store the Selected SOP Series or click **Delete SOP Series** to delete the Selected SOP Series.

3 Click **Close** to close the Organize Favorite SOP Series window.

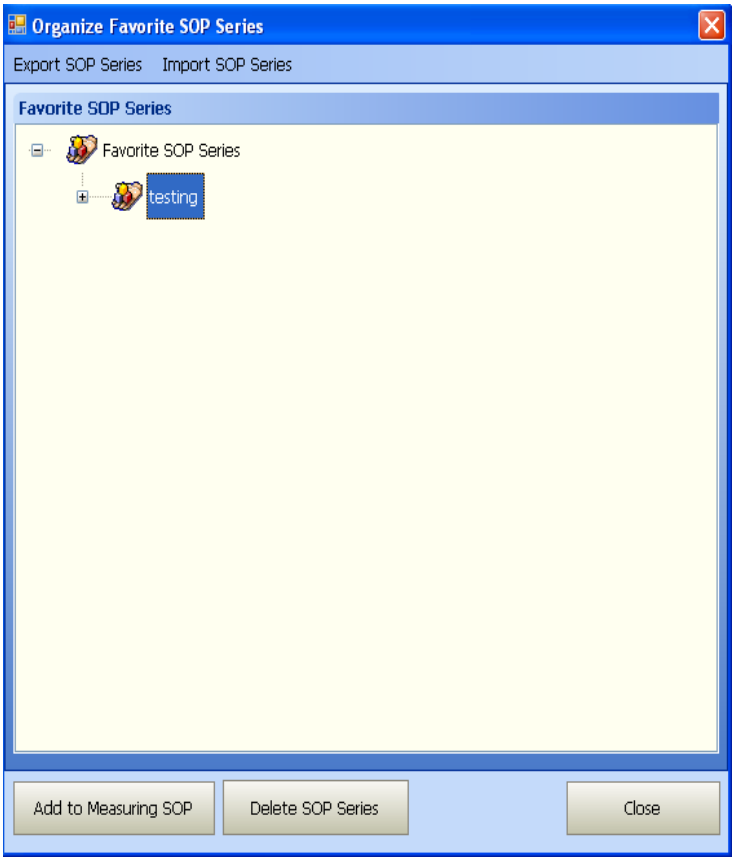
Exporting Favorite SOP Series Files

You can export SOP files from your Favorite SOP Series to a folder or external memory.

To export SOP files from your Favorite SOP Series:

1 Select **SOP > Favorite SOP > Organize Favorite SOP Series** on the Main menu bar.

Organize Favorite SOP Series window appears.



2 Click **Export SOP Series.** The Export SOP Series window opens.

3 Click **Browse** next to the Export File field to open the Save As dialog.

4 Select the destination, enter a file name in the Save As dialog, and click **Save**.

5 In the Export SOP Series window, click **Export File**. A status message at the bottom of the window indicates the completion of the export.

6 Click **Close** to close the Export SOP Series window.

7 Click **Close** to close the Organize Favorite SOP Series window.

Importing Favorite SOP Series Files

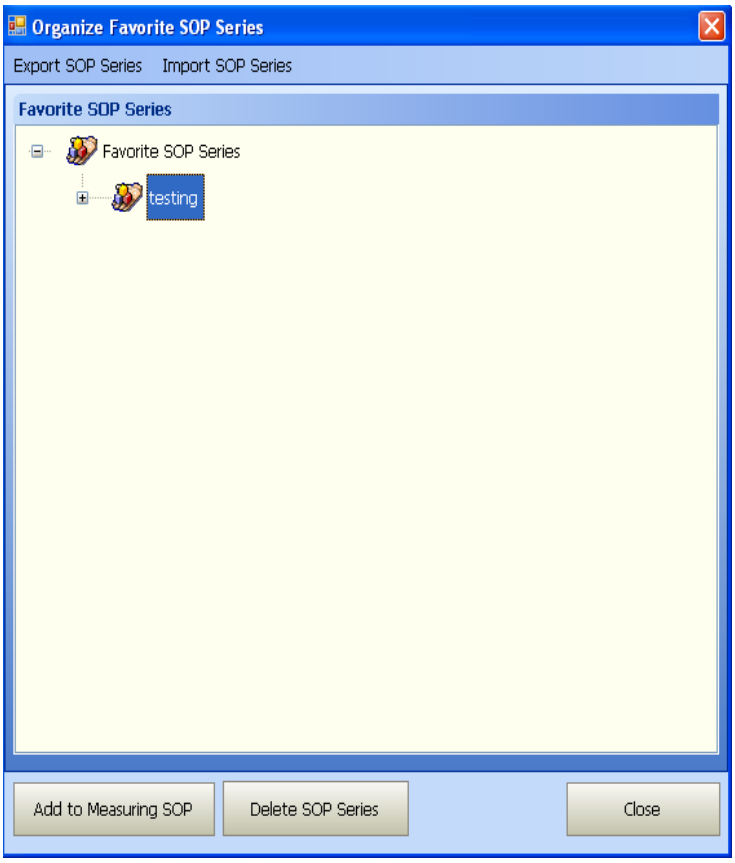
Use the Import function to include exported SOP Series data in a data file list.

You can export SOP files from your Favorite SOP Series to a folder or external memory.

To import SOP files from your Favorite SOP Series:

1 Select **SOP > Favorite SOP > Organize Favorite SOP Series** on the Main menu bar.

Organize Favorite SOP Series window appears.



2 Click **Import SOP Series**. The Import SOP Series window opens.

3 Click **Browse** next to the Import File field to open the Open dialog.

4 Select a file name in the Open dialog, and click **Open**.

5 In the Import SOP Series window, click **Import File**. A status message at the bottom of the window indicates the completion of the export.

6 Click **Close** to close the Import SOP Series window.

7 Click **Close** to close the Organize Favorite SOP Series window.

Measuring Particle Size and Zeta Potential

This section describes how to prepare for the measurement, how to start and stop the measurement, and how to display and print measurement results.

Figure 1.30 shows the first screen that appears when you start the software (and log in if necessary).

Figure 1.30 Data Acquisition Measurement Screen

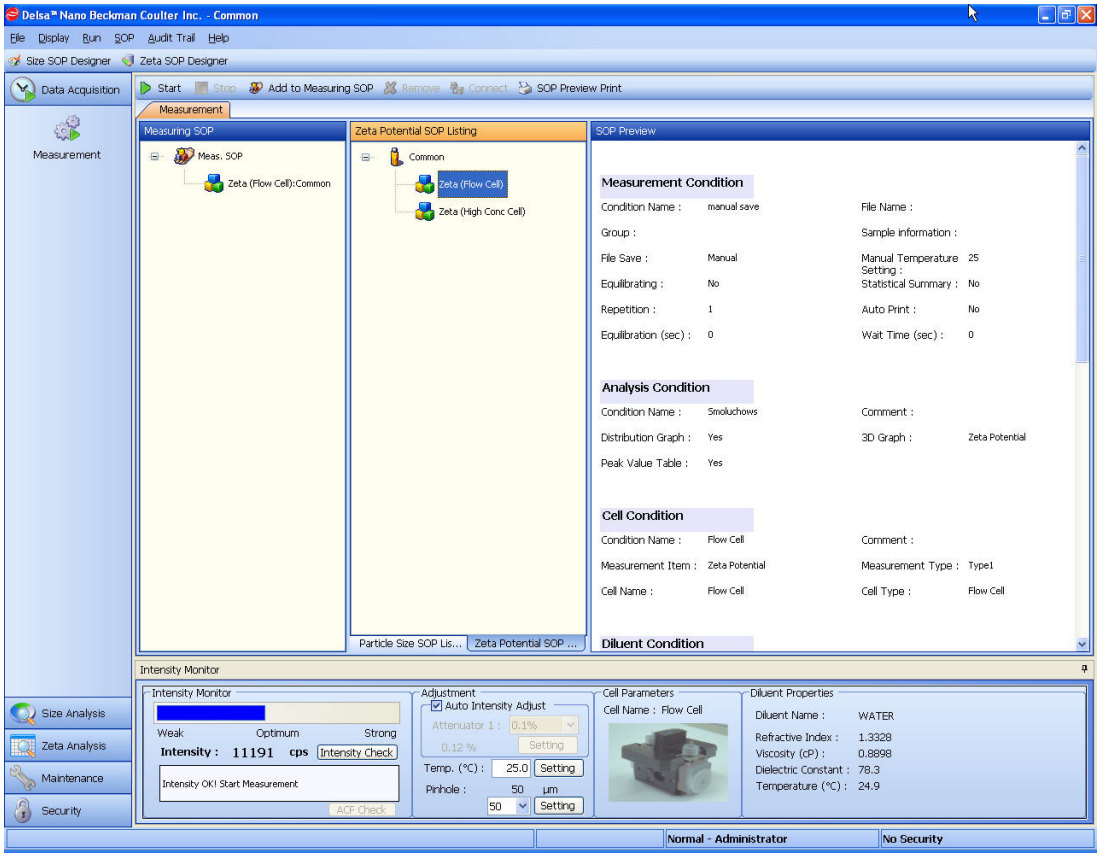


Table 1.13 Data Acquisition Measurement Screen Elements

Screen Element	Description
Data Acquisition function icons	Displays the selected icon; in this case, Measurement.
Selected Measuring SOP Listing	Displays the SOP that will be used in the measurement.
Particle Size SOP Listing	Displays the particle size measurement SOPs that have been registered.
Zeta Potential SOP Listing	Displays the zeta potential SOPs that have been registered.

Table 1.13 Data Acquisition Measurement Screen Elements *(Continued)*

Screen Element	Description
SOP Preview	Displays the parameters within the SOPs. When you select an SOP in the SOP listing or in the Measuring SOP, the parameters for that SOP are displayed.
Intensity Monitor	Displays the parameters that must be verified prior to measurement, such as the cell type, the intensity monitor (sample's intensity), and the diluent properties conditions within the SOP conditions that have been registered for the selected SOP.

Selecting the Measurement SOP

To select the measurement SOP:

1 In the Measurement screen, select the desired SOP from the SOP listing.

2 Select **SOP > Add to Measuring SOP** on the Main menu bar, or click **Add to Measuring SOP**. (You can also drag and drop the SOP into the Measuring SOP.) This registers the selected SOP.

NOTE Up to a maximum of 20 SOPs can be registered to the Selected SOPs. When multiple SOPs are registered, execution will proceed from the top of the list.

⚠ CAUTION

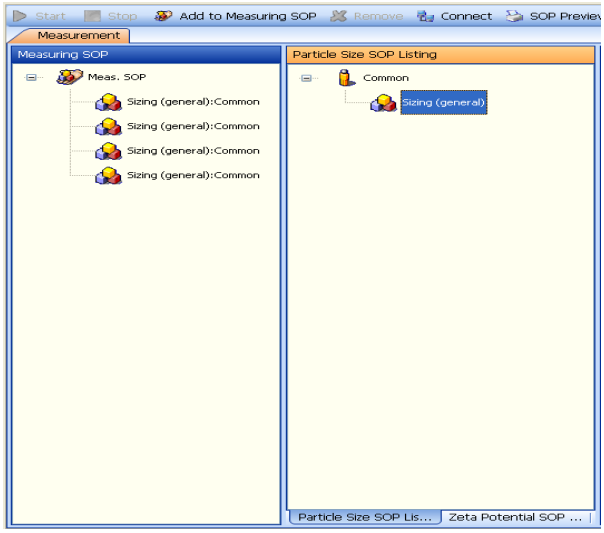
When multiple SOPs are registered, the registered SOPs must all use the same type of cell and diluent.

One-By-One Analysis of Size and Zeta Potential

The instrument can measure particle size/zeta potential (or vice versa) one after the other when the same cell type and diluent are used to measure both size and zeta potential of the sample.

The instrument uses the first SOP to measure either size or zeta potential, then moves to the next SOP, and so on.

Figure 1.31 Measurement SOP



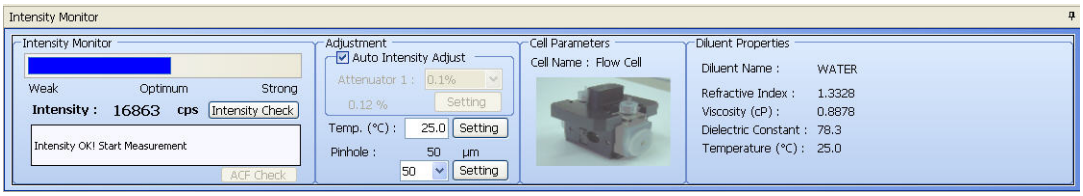
Checking the Cell Type, Intensity (Scattering Strength), and Diluent Properties

To check the cell type, intensity (scattering strength), and diluent properties:

1 Check the SOP parameters in the SOP Preview screen.

2 Perform the minimum checks in the Intensity Monitor at the bottom of the window.

Figure 1.32 Intensity Monitor

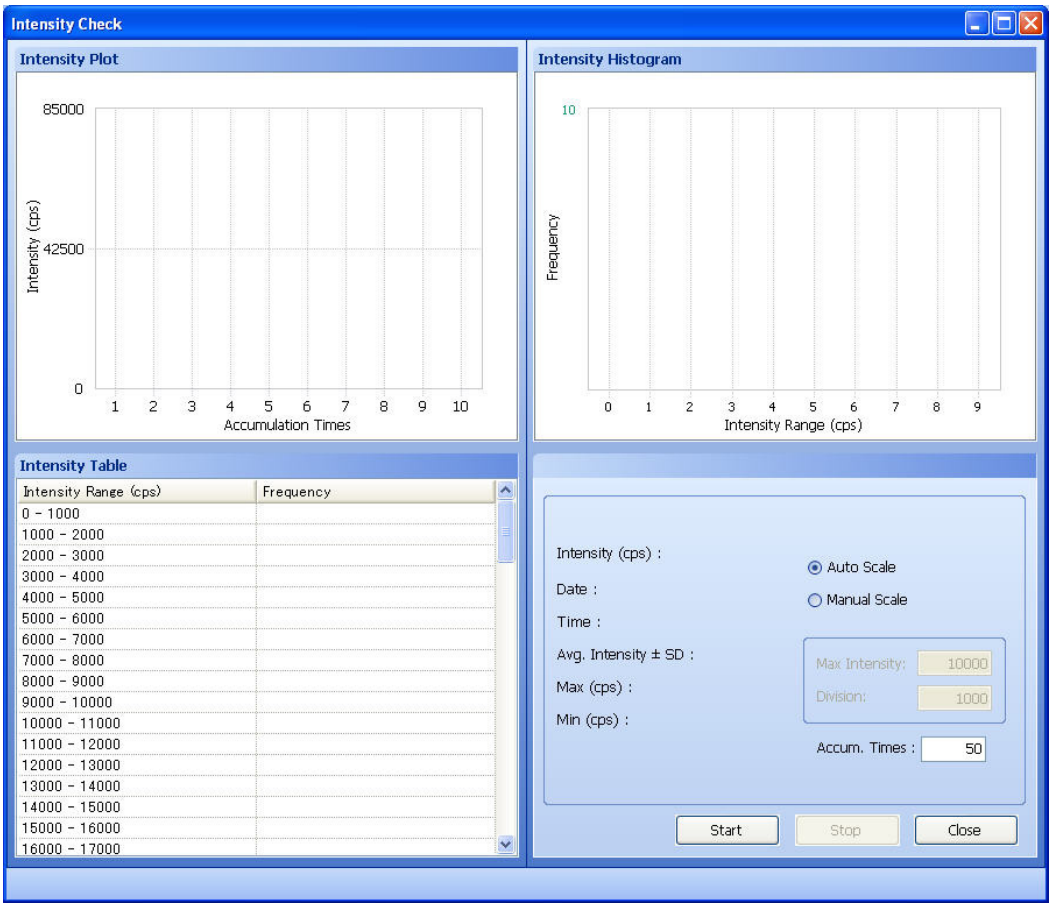


Checking the Intensity (Scattering Strength) Stability

To check the intensity (scattering strength) stability:

1 Click **Intensity Check** in the Intensity Monitor Form. The Intensity Check screen opens.

Figure 1.33 Intensity Check Screen



2 Enter the Accum Times and click **Start** to perform the intensity measurement. When the measurement is complete, the Intensity Plot, Intensity Histogram, and Intensity Table value (±SD), Max (cps), and Min (cps) for the intensity appear. You can select either Auto Scale or Manual Scale.

Starting the Measurement

To start the measurement:

1 Select the Measurement icon from the Data Acquisition function panel.

2 Click **Start** in the upper left of the Measurement screen, or select **Run > Start** on the Main menu bar.

NOTE The **Start** button is not displayed in other software screens, such as SOP Designer. Select the Measurement icon in the Data Acquisition function panel to display **Start**.

During the measurement, the screens that are selected in the Analysis Condition in the SOP are displayed. To display a graph (or graphs), click the name of the graph you want at the top of the screen. You can do this during a measurement. Additionally, a summary of the measurement parameters and conditions appears in the Results panel above the graphs.

Figure 1.34 Example Screen Display During Measurement of Particle Size

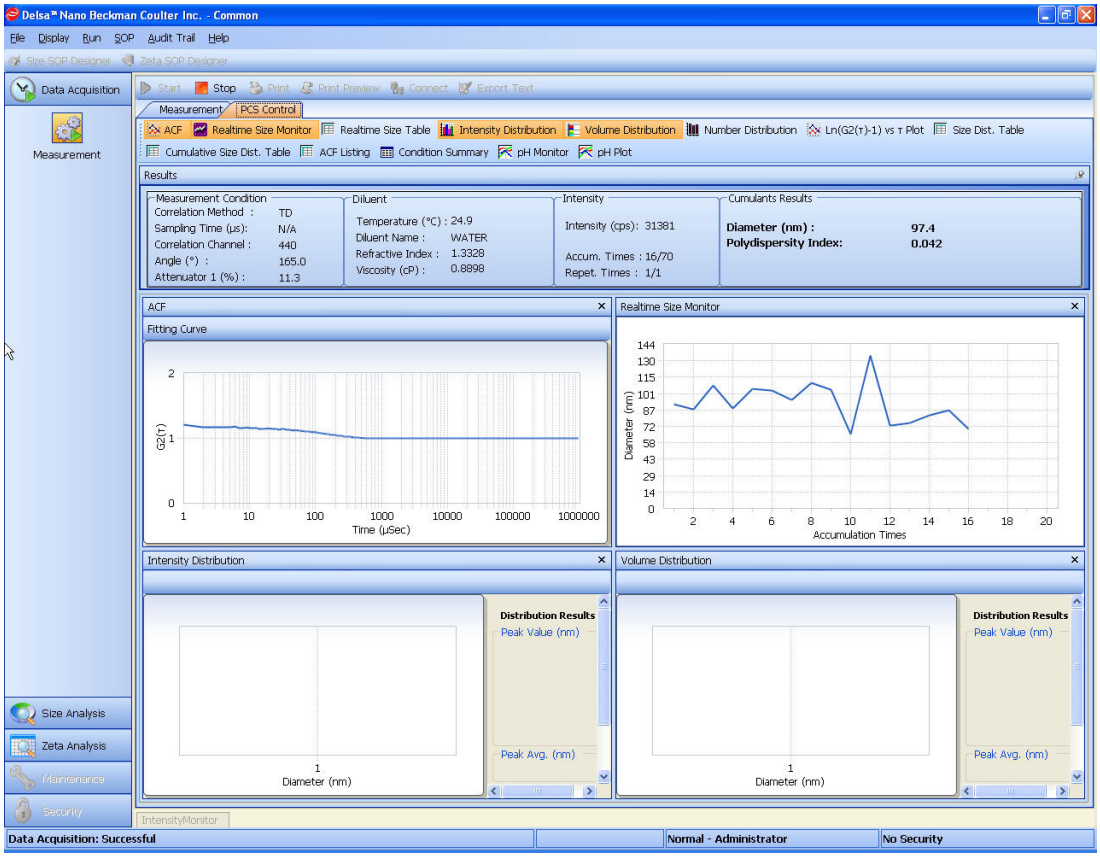


Table 1.14 Data Acquisition Measurement Screen Elements

Screen Element	Description
Display Item Icons	Select an icon to display or remove the desired graph or table.
Overview of Measurement Parameters and Measurement Status	Displays the measurement parameters and diluent properties set in the SOP; the intensity, cumulative number of measurement cycles, and the number of repeats during the measurement; and the real-time measurement results during the measurement are displayed.
Measurement Results Display Screen	Displays the graphs and tables selected in the SOP.

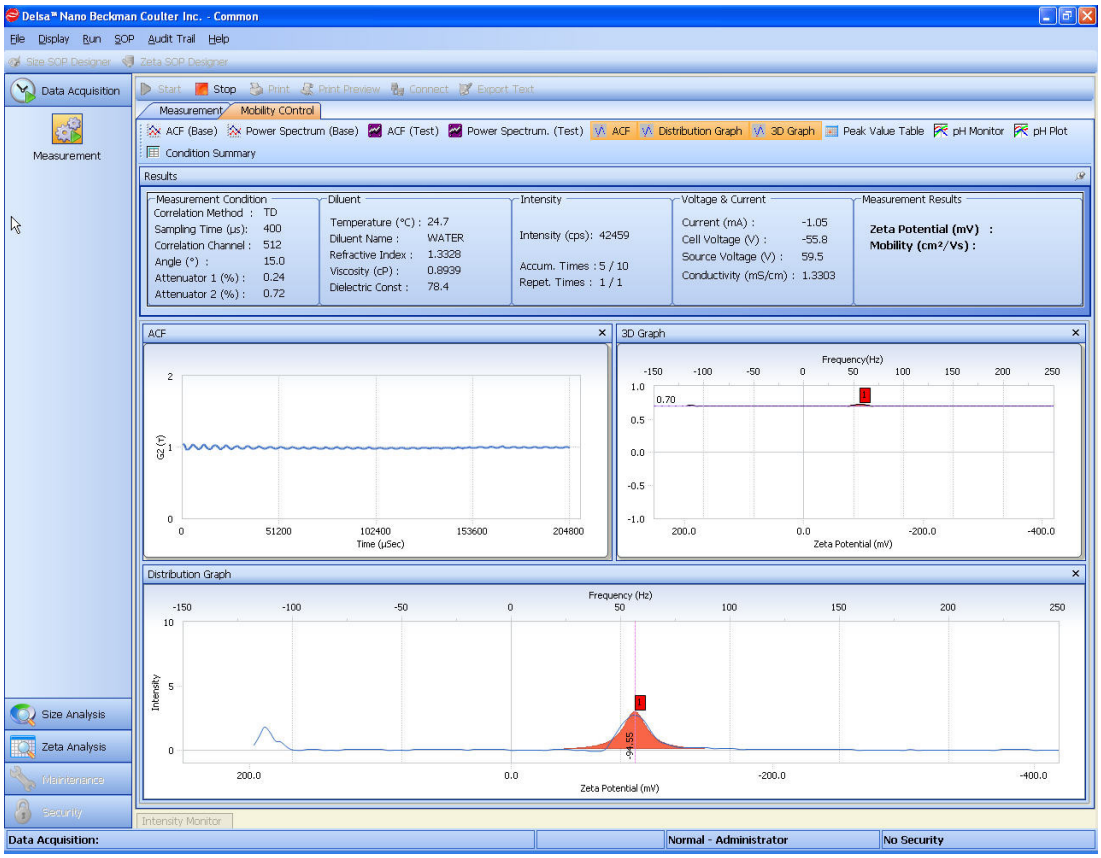
Stopping the Measurement

To stop the measurement, click **Stop** in the upper left of the Measurement screen, or select **Run > Stop** on the Main menu bar.

Displaying and Printing Measurement Results

After the measurement is complete, the measurement results are displayed according to the analysis parameters. To change the screen display, click the buttons at the top of the screen. SOP settings are unaffected.

Figure 1.35 Example Zeta Potential Analysis Measurement Results



When Auto Print is set to YES in the SOP, the selected results print automatically upon conclusion of the measurement.

To print the measurement results manually:

1 Select **File > Print** on the Main menu bar, or click **Print** at the top of the screen. A Print dialog opens.

2 Select the items you want to print, and click **OK**.

Analyzing Particle Size

Accessing Particle Size Analysis Data File Operations

To access particle size analysis data file operations, select the Size Analysis function panel. The Particle Size Analysis screen opens.

Figure 1.36 Particle Size Analysis Screen

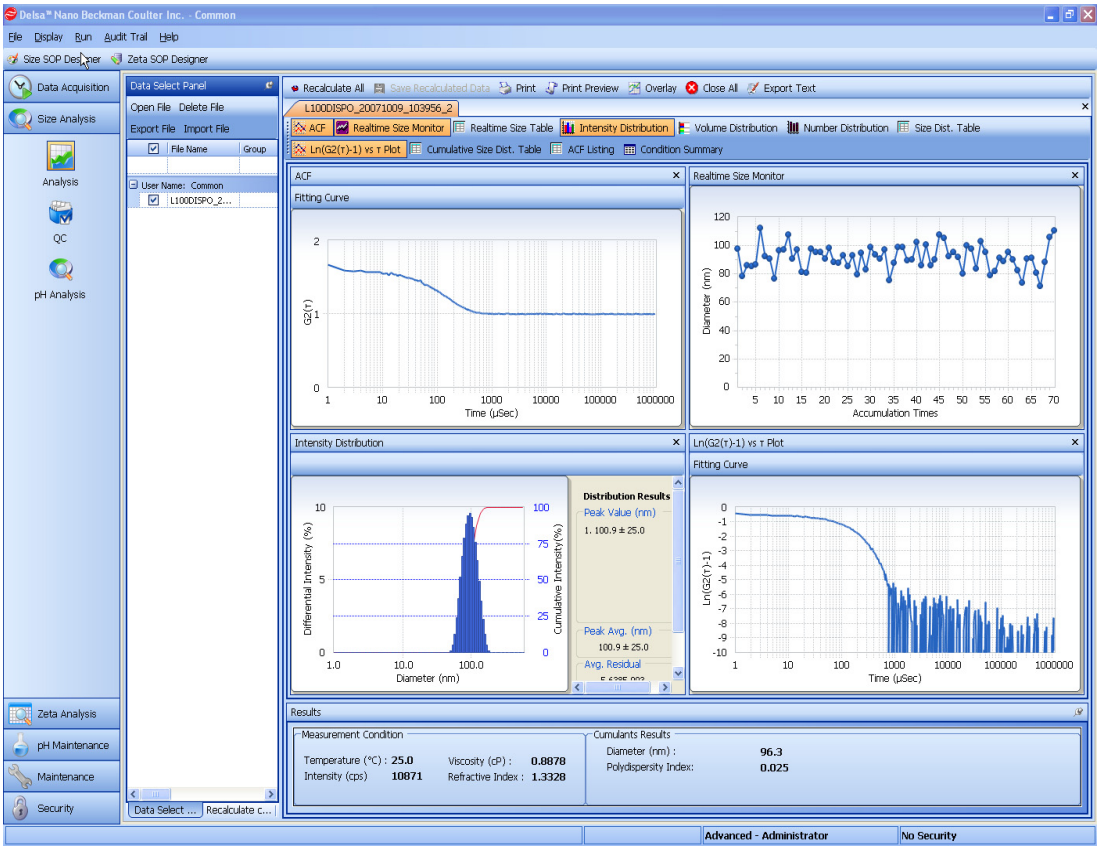


Table 1.15 Particle Size Analysis Screen Elements

Screen Element	Description
Particle Size Analysis Data Select Panel	Lists the data files with these details: File Name, Group, Repet,No, Date&Time, User Name, Sample Info, SOP Name, pH setting and Cell Name.
Recalculate Condition Panel	Displays particle size files for recalculation.
Data Select Panel Button Bar	Use these buttons to perform data file operations: • **Open File** • **Delete File** • **Export File** • **Import File**
Analysis Results Panel	Displays the details of a selected data file. For more information, see *Opening a Particle Size Analysis Data File*.

Opening a Particle Size Analysis Data File

To open a particle size analysis data file:

1 In the Particle Size Analysis Data Select panel, place a checkmark next to the data file you want to open.

2 Click **Open File**. The analysis results are displayed in graphs and tables in the Analysis Results panel (see Figure 1.37).

Figure 1.37 Particle Size Analysis Data File Details

Deleting a Particle Size Analysis Data File

To delete a particle size analysis data file:

1. In the Particle Size Analysis Data Select panel, place a checkmark next to the data file you want to delete.
2. Click **Delete File**. A confirmation message appears.
3. Click **OK** to delete the file.

⚠ CAUTION

Deleted data cannot be recovered.

Exporting a Particle Size Analysis Data File

You can export particle size analysis data files to a folder or external memory.

To export a particle size analysis data file:

1. In the Particle Size Analysis Data Select panel, place a checkmark next to the data file to export.
2. Click **Export File**. The Export File window opens.
3. Click **Browse** next to the Export File field to open the Save As dialog.
4. Select the destination, enter a file name in the Save As dialog, and click **Save**.
5. In the Export File window, click **Export File**. A status message at the bottom of the window indicates the completion of the export.
6. Click **Close** to close the Export File window.

Importing a Particle Size Analysis Data File

Use the Import function to include exported particle size data in a data file list.

To import a particle size analysis data file:

1. Click **Import File** above the Particle Size Analysis Data Select panel. The Import File window opens.
2. Click **Browse** next to the Import File field to open the File Selection dialog.
3. Select the file name you want to import, and click **Open**.
4. In the Import File window, click **Imported Selected Files**. A status message at the bottom of the window indicates the completion of the imported.
5. Click **Close** to close the Import File window.

⚠ CAUTION

A file cannot be imported if it has the same name as a data file already in the list. Data highlighted in red text indicates that data with the same name already exists in the data file.

Modifying Particle Size Analysis Parameters

To modify particle size analysis parameters:

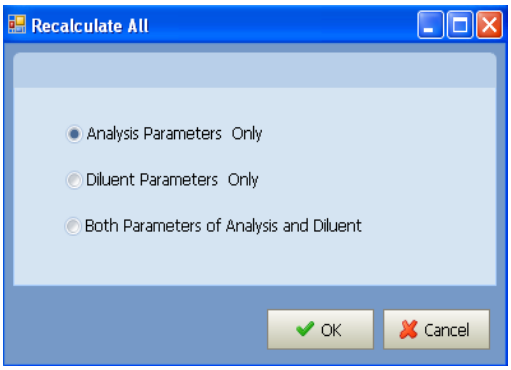
1. Click **Recalculate Condition**.
2. Select the set of parameters to be modified. Each of the parameters within the selected set of parameters appears at the bottom of the window, and the necessary parameters will be modified.
3. After the modification, click **Recalculate** to analyze the data again. The analysis results using the new parameters are displayed.

Modifying All Particle Size Analysis Parameters

To modify all particle size analysis parameters:

1 Select and open the desired files and click **Recalculate Condition**.

2 Make the necessary changes to the condition analysis and click **Recalculate All.**Recalculate All dialog box appears.



3 Select the parameters and click **OK**.

Saving the Particle Size Reanalysis Data

To save particle size reanalysis data:

1. Click **Save Recalculated Data** in the Particle Size Analysis Results panel. The Save Recalculated Data dialog opens.

2. Select either Manual Input to enter the file name, group name, and sample information, or select Add Numbering to append a number to the current file name or overwrite the existing file.

3. Click **OK** to save and close the dialog.

4. In 21 CFR Part 11 security mode, after recalculation and saving the data, signature, and so on, a Signature dialog appears. Enter the password and the reason for the changes (Comment), and click **OK** to save and close the dialog.

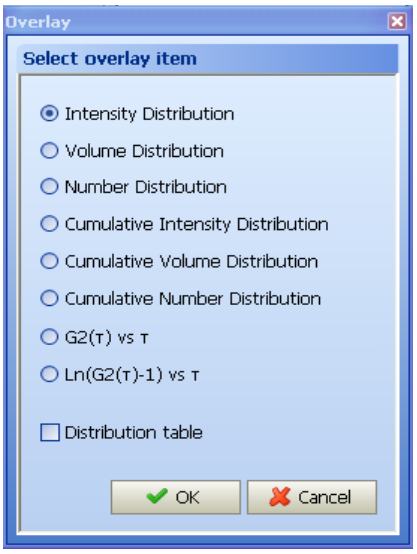
Overlaying Particle Size Analysis Data

You can select multiple data in the Particle Size Analysis Data Select Panel, analyze them, and overlay the results.

To overlay particle size analysis data:

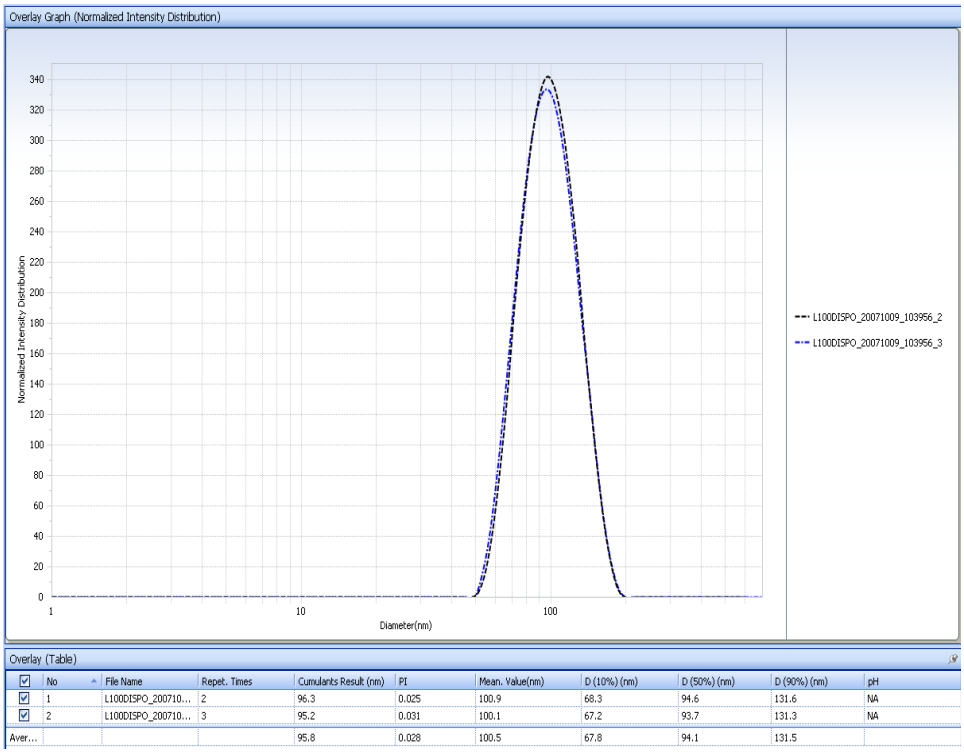
1. Click **Overlay** in the Particle Size Analysis Results panel. The Particle Size Analysis Data Overlay dialog opens.

Figure 1.38 Particle Size Analysis Data Overlay Dialog



2 Select the items to overlay, and click **OK**. The overlay in graph form appears.

Figure 1.39 Example Particle Size Analysis Overlays

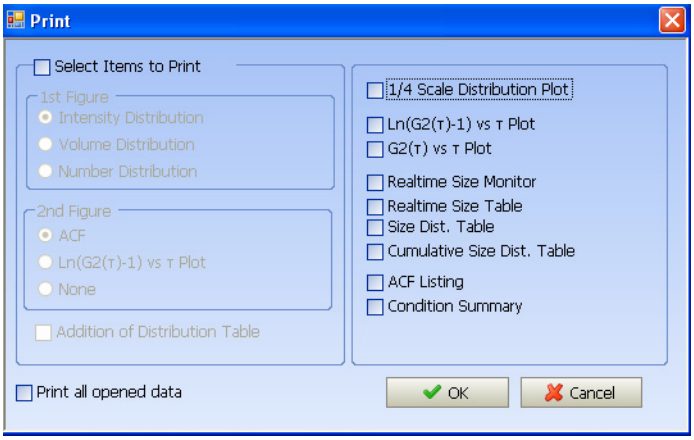


Printing Particle Size Analysis Data

To print the particle size analysis results:

1 Click **Print** in the upper-left of the Measurement screen, or select **File > Print** on the Main menu bar.

Figure 1.40 Data Print Dialog



2 Select the options for printing, and click **OK**.

Table 1.16 Data Print Dialog Options

Selection	Description
Select Print Item	Activates these options: 1st Figure, 2nd Figure, and Addition of Distribution Table.
1st Figure	Select from Intensity Distribution, Volume Distribution, and Number Distribution. The item selected for the 1st figure is printed on the same sheet of paper with the 2nd figure and the Distribution Table (if selected).
2nd Figure	Select ACF or LN(G1(τ)) vs τ Plot. The item selected for the 2nd figure is printed on the same sheet of paper with the 1st figure and the Distribution Table (if selected).
Distribution Table	Prints the population distribution data selected in the 1st figure, together with the 1st figure and a 2nd figure.
1/4 Scale Distribution Plot	Prints on a single sheet of paper the Intensity Distribution, the Volume Distribution, the Number Distribution, and LN(G1(τ)) vs τ Plot.
LN(G1(τ)) vs τ Plot	Prints a first-order autocorrelation logarithmic display.
G2(τ) vs τ Plot	Prints a second-order autocorrelation function.
Realtime Size Monitor	Prints a plot of the particle size values for each integration cycle being calculated.
Realtime Size Table	Prints a table of the particle size values for each integration cycle being calculated.

Table 1.16 Data Print Dialog Options *(Continued)*

Selection	Description
Size Dist. Table	Prints distribution frequency tables for the intensity distribution, volume distribution, and number distribution.
Cumulative Size Dist. Table	Prints cumulative frequency tables for the intensity distribution, volume distribution, and number distribution.
ACF Listing	Prints tables of the first-order and second-order autocorrelation functions.
Condition Summary	Displays the measurement parameters, analysis parameters, cell parameters, and diluent properties extracted from the SOP parameters.
Print All Open Data	Prints all data that is opened.

Displaying the Particle Size Statistical Summary

To display the particle size measurement statistical summary:

1. Set the Statistical Summary in the Measurement Parameters to Yes. This saves all measurement data in the statistical summary file. This applies to new measurements using the measurement parameter; any previous data will not appear in the statistical summary.

2. To display the statistical summary, select the QC icon in the Size Analysis panel.

3. From the list of statistical summary files, select the files you want to open, and click **Open** above the list. In the panel on the right, a list of the measurement dates, file names, and typical data (typical values for the average particle sizes, multidisperse indices, and distributions) for each data file included in the statistical summary will be displayed.

4. To display details, select the data you want, and click **Details** above the summary list.

Analyzing Zeta Potential

Accessing Zeta Potential Analysis Data File Operations

To access zeta potential data file operations, select the Zeta Analysis function panel. The Zeta Potential Analysis screen opens.

Figure 1.41 Zeta Potential Analysis Screen

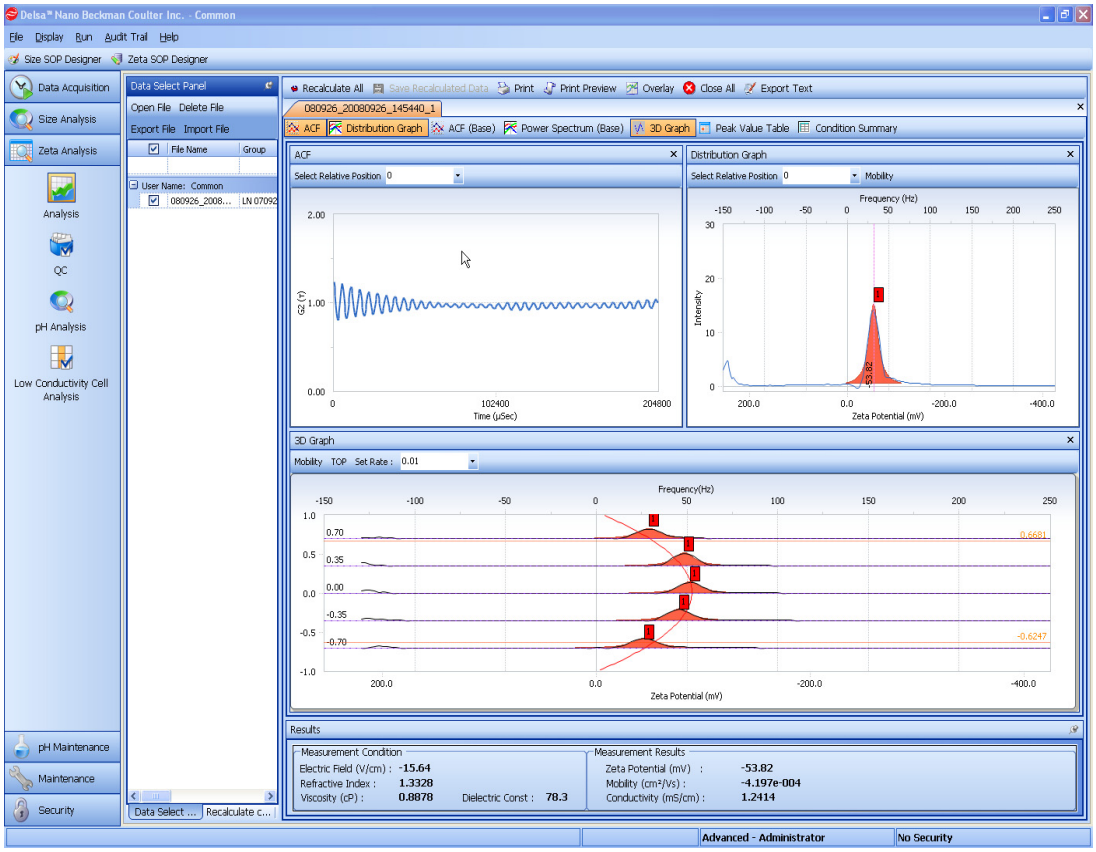


Table 1.17 Zeta Potential Size Analysis Screen Elements

Screen Element	Description
Zeta Potential Analysis Data Select Panel	Lists the data files with these details: File Name, Repet. No, Date & Time, User Name, Sample Info, Group, and pH setting and Cell Name.
Recalculate Condition Panel	Displays particle size files for recalculation.
Data Select Panel Button Bar	Performs the following data file operations: • **Open File** • **Delete File** • **Export File** • **Import File**
Analysis Results Panel	Displays the details of a selected data file. For more information, see *Opening a Particle Size Analysis Data File*.

Opening a Zeta Potential Analysis Data File

To open a zeta potential analysis data file:

1 In the Zeta Potential Analysis Data Select panel, place a checkmark next to the data file you want to open.

2 Click **Open**. The analysis results are displayed in graphs and tables (see Figure 1.42).

Figure 1.42 Zeta Potential Analysis Data File Details

Deleting a Zeta Potential Analysis Data File

To delete a zeta potential analysis data file:

1 In the Zeta Potential Analysis Data Select panel, place a checkmark next to the data file you want to delete.

2 Click **Delete File**. A confirmation message appears.

3 Click **OK** to delete the file.

Exporting a Zeta Potential Analysis Data File

You can export zeta potential analysis data files to a folder or external memory.

To export zeta potential analysis data files:

1. In the Zeta Potential Analysis Data Select panel, place a checkmark next to the data files to export.
2. Click **Export File**. The Export File window opens.
3. Click **Browse** next to the Export File field to open the Save As dialog.
4. Select the destination, enter a file name in the Save As dialog, and click **Save**.
5. In the Export File window, click **Export File**. A status message at the bottom of the window indicates the completion of the export.
6. Click **Close** to close the Export File window.

Importing a Zeta Potential Analysis Data File

Use the Import function to include exported zeta potential analysis data in a data file list.

To import zeta potential analysis data files:

1. Click **Import File** above the Zeta Potential Analysis Data Select panel. The Import File window opens.
2. Click **Browse** next to the Import File field to open the File Selection dialog.
3. Select the file name you want to import, and click **Open**.
4. In the Import File window, click **Imported Selected Files**. A status message at the bottom of the window indicates the completion of the import.

5 Click **Close** to close the Import File window.

⚠ CAUTION

A file cannot be imported if it has the same name as a data file already in the list. Data highlighted in red text indicates that data with the same name already exists in the data file.

Modifying Zeta Potential Analysis Parameters

To modify zeta potential analysis parameters:

1 Click **Recalculate Condition**.

2 Select the set of parameters to be modified. Each of the parameters within the selected set of parameters appears at the bottom of the window, and the necessary parameters are modified.

3 After the modification, click **Recalculate** to analyze the data again. The analysis results using the new parameters are displayed.

Modifying All Zeta Potential Analysis Parameters

To modify all zeta potential analysis parameters:

1 Select and open the desired files and click **Recalculate Condition**.

2 Make the necessary changes to the condition analysis and click **Recalculate All.** The Recalculate All window opens.

3 Select the parameters and click **OK**.

Saving the Zeta Potential Reanalysis Data

To save zeta potential reanalysis data:

1 Click **Save Recalculate Data** in the Zeta Potential Analysis Results panel. The Save Recalculated Data dialog opens.

2 Select **Overwrite** to overwrite the data or Add Numbering to append a number extension to the current file name or overwrite the existing file.

3 Click **OK** to save and close the dialog.

In 21 CFR Part 11 security mode, after the data is recalculated and saved, a Signature dialog appears. Enter the password and the reason for the changes (Comment), and click **OK** to save and close the dialog.

Overlaying Zeta Potential Analysis Data

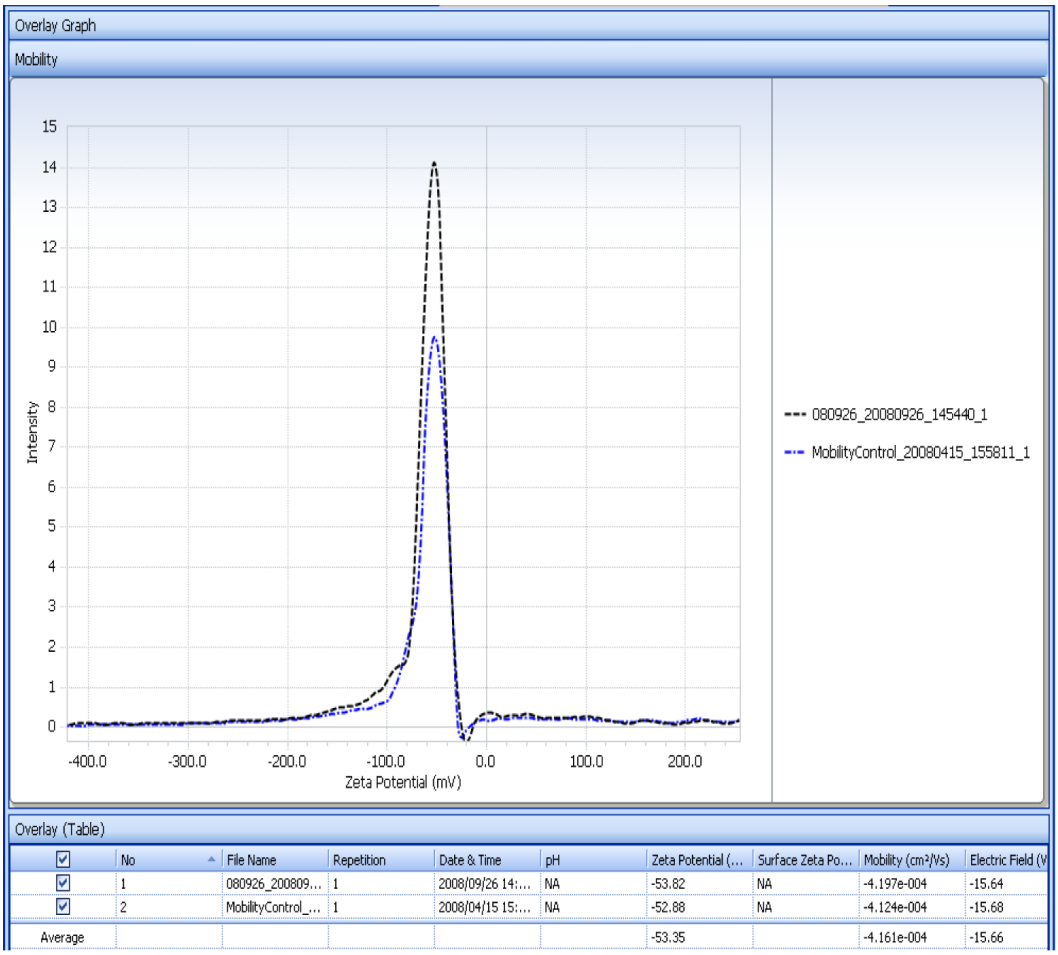
You can select multiple data in the Zeta Potential Analysis Data Select Panel, analyze them, and overlay the results.

To overlay zeta potential analysis data:

1 Click **Overlay** in the Zeta Potential Analysis Results panel. The Zeta Potential Analysis Overlay dialog opens.

2 Select the items to overlay, and click **OK**. The overlay in graph form appears.

Figure 1.43 Example Zeta Potential Analysis Overlays



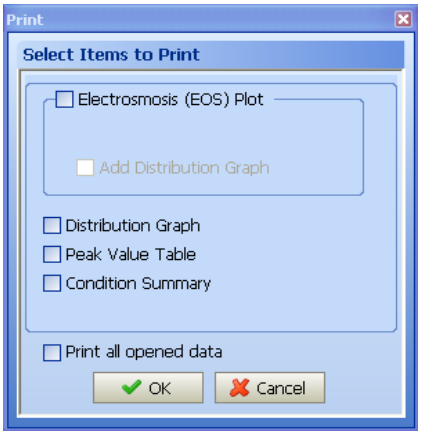
	No	File Name	Repetition	Date & Time	pH	Zeta Potential (...	Surface Zeta Po...	Mobility (cm²/Vs)	Electric Field (V
☑	1	080926_200809...	1	2008/09/26 14:...	NA	-53.82	NA	-4.197e-004	-15.64
☑	2	MobilityControl_...	1	2008/04/15 15:...	NA	-52.88	NA	-4.124e-004	-15.68
Average						-53.35		-4.161e-004	-15.66

Printing Zeta Potential Analysis Results

To print the results of a zeta potential analysis:

1 Click **Print** in the upper left of the Measurement screen, or select **File > Print** on the Main menu bar. The Print dialog opens.

Figure 1.44 Zeta Potential Analysis Data Print Dialog



2 Select the options for printing, and click **OK**.

Table 1.18 Zeta Potential Analysis Data Print Dialog Selections

Selection	Description
Electrosmosis (EOS) Plot	Displays a table of the speed of movement for all particles within the cell, including an electroosmotic profile. When you select the Distribution Graph check box, a graph of the true zeta potential (mobility) of the particles corresponding to the speed of movement in the static layer position is printed on the same sheet. See APPENDIX D, *Graphs and Table Displays*.
Distribution Graph	Prints a graph of the true zeta potential (mobility) of the particles corresponding to the speed of movement in the static layer position. See APPENDIX D, *Graphs and Table Displays*.
Peak Value Table	Prints a table of zeta potentials, mobilities, and electric fields. See APPENDIX D, *Graphs and Table Displays*.
Condition Summary	Displays the measurement parameters, analysis parameters, cell parameters, and diluent properties extracted from the SOP parameters.
Print All Open Data	Prints all data that is opened.

Displaying the Zeta Potential Analysis Statistical Summary

To display the zeta potential statistical summary:

1. Set Statistical Summary in the Measurement Parameters to Yes. This saves all measurement data in the statistical summary file. This applies to new measurements using the measurement parameter; any previous data will not appear in the statistical summary.

2. To display the statistical summary, select the QC icon in the Zeta Analysis panel.

3. From the list of statistical summary files, select the files you want to open, and click **Open** above the list. In the panel on the right, a list of the measurement dates, file names, and typical data (zeta potentials and electrophoretic mobilities) for each data file included in the statistical summary is displayed.

4. To display details, select the data you want, and click **Details** above the summary list.

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CHAPTER 2

Troubleshooting

Introduction

This sections lists possible malfunctions, together with probable causes and corrective actions. Maintenance procedures are described in CHAPTER 4, *Maintenance*.

Diagnostic Conditions

Diagnostic conditions appear during:

- Instrument startup
- Self-Diagnosis
- Door operations
- Communications
- Measurements

Table 2.1 DelsaNano Diagnostics

Symptom	Cause	Corrective Action
Instrument does not turn on	AC cable on the rear panel of the instrument is not connected.	Connect power cable. Verify power cable is plugged in securely.
The Logotype on the front panel does not light up	Blown fuse.	Replace fuse. If the fuse continues to blow after being replaced, contact Beckman Coulter Field Service.
The power can be switched on, but the Power LED does not light up	CPU does not start up.	Contact Beckman Coulter Field Service.
Power LED does not change from orange to green	CPU does not start up.	Contact Beckman Coulter Field Service.
Shutter 1 does not move correctly	Shutter movement error.	Contact Beckman Coulter Field Service.
Shutter 2 does not move correctly	Shutter movement error.	Contact Beckman Coulter Field Service.

Table 2.1 DelsaNano Diagnostics *(Continued)*

Symptom	Cause	Corrective Action
Shutter 3 does not move correctly	Shutter movement error.	Contact Beckman Coulter Field Service.
Shutter 4 does not move correctly	Shutter movement error.	Contact Beckman Coulter Field Service.
Error reading temperature	Error in setting temperature constant.	Verify temperature setting in SOP. If the problem persists, contact Beckman Coulter Field Service.
	Malfunction of temperature sensor.	Contact Beckman Coulter Field Service.
	Malfunction of temperature control part on main board.	Contact Beckman Coulter Field Service.
Error message, dark error detected	Error in shutter movement.	Contact Beckman Coulter Field Service.
	Malfunction of Discriminator board.	Contact Beckman Coulter Field Service.
	Malfunction of Detector (PMT).	Contact Beckman Coulter Field Service.
	Malfunction of Detector (PMT).	Contact Beckman Coulter Field Service.
The version of DSP is not correct	The slave version does not conform with the host.	Contact Beckman Coulter Field Service.
Instrument does not sense that the door is closed	The cover is open during operation of the instrument.	Close the cover door.
	The sensor is not pushed down fully even though the cover is closed.	Verify that there are no obstructions to the door sensor and door latch. Close the door.
	Malfunction of cell door sensor.	Contact Beckman Coulter Field Service.
Communication failure between the instrument and the computer	Power to the instrument is not switched on.	Verify instrument is turned on.
	USB cable is not connected.	Reconnect USB cable at the DelsaNano and at the rear of the controller.
	Instrument CPU failure.	Contact Beckman Coulter Field Service.
Light intensity is too low (the light count is too low and the indicator is red at sample check)	Setting of ND filter is not correct.	Check SOP for Intensity adjustment parameter. Set parameter to Yes so that the ND filter automatically adjusts for optimum intensity. If the problem persists, contact Beckman Coulter Field Service.
	The type of cell block is wrong.	Check SOP parameter.
	The cell block is not set correctly.	Reseat measuring cell holder.
	The concentration of sample is too low (at measurement in the cell for low concentration).	Increase concentration of sample.
	The concentration of sample is too low (at measurement in the cell for high concentration).	Decrease concentration of sample.
	The cell center is not adjusted correctly.	Check SOP parameter. Verify cell type. Readjust cell center.

Table 2.1 DelsaNano Diagnostics *(Continued)*

Symptom	Cause	Corrective Action
	Movement error of the shutter.	Contact Beckman Coulter Field Service.
	Movement error of rotating concentration filters on the light pass of reference light/ light for Zeta Potential.	Contact Beckman Coulter Field Service.
Light intensity is too low (the light count is too low and the indicator is red at sample check)	Malfunction of laser.	Contact Beckman Coulter Field Service.
	Light axis has shifted.	Contact Beckman Coulter Field Service.
	Malfunction of Discriminator board.	Contact Beckman Coulter Field Service.
	Malfunction of HV.	Contact Beckman Coulter Field Service.
Light intensity is insufficient (an error message at the start of measurement)	Light intensity was not confirmed before measurement.	Confirm light intensity and adjust sample concentration accordingly.
	"Adjustment of Light Intensity" has not been set to be automatic at setting of conditions described in SOP.	Check SOP.
Cell center adjustment has failed	The cell block is not set correctly.	Reseat cell block into cell compartment.
	The type of cell block is wrong.	Check SOP.
	Error in assembly of cell block.	Verify cell block assembly.
	Debris or dirt on the cell.	Clean cell block.
	Scratches on the cell	Replace cell block.
	Concentration of sample is too low.	Increase concentration of sample.
	Movement error of rotating concentration filter	Contact Beckman Coulter Field Service.
	Movement error of cell stage	Contact Beckman Coulter Field Service.
Temperature indicated on the display does not reach the set temperature	The set temperature is out of the specification range.	Check SOP parameter. DelsaNano specification is -15° C below ambient.
	Contact of connector of temperature sensor is not secure.	Contact Beckman Coulter Field Service.
	Movement of fan for heat radiation/ cooling is not correct.	Contact Beckman Coulter Field Service.
	Malfunction of temperature sensor.	Contact Beckman Coulter Field Service.
	Malfunction of Peltier device.	Contact Beckman Coulter Field Service.
	Malfunction of heater.	Contact Beckman Coulter Field Service.
	Malfunction of temperature control part on the main board.	Contact Beckman Coulter Field Service.
Temperature indicated on the display fluctuates	Malfunction of temperature sensor.	Contact Beckman Coulter Field Service.
Automatic adjustment of light intensity has failed	The concentration of the sample is too low.	Increase concentration of sample.
	Range of intensity for automatic adjustment of light intensity is too narrow.	Confirm the range of intensity for automatic adjustment of light intensity in SOP parameters.

Table 2.1 DelsaNano Diagnostics *(Continued)*

Symptom	Cause	Corrective Action
	Movement error of rotating concentration filter.	Contact Beckman Coulter Field Service.
No voltage detected	Applied voltage of SOP parameter is “0V”.	Check SOP.
	Cell door is opened.	Close cell door.
	No voltage.	Contact Beckman Coulter Field Service.
Measurement values of standard samples do not fall within the permissible range	Shelf life of the samples has expired.	Replace sample.
	The type of cell block is wrong.	Check SOP.
	The cell block is not installed correctly.	Reinstall cell block.
	There are bubbles in the cell.	Remove bubbles.
	The cell center is not adjusted correctly.	Check SOP. Verify cell type. Readjust cell center.
	Error in diluent properties.	Check SOP. Verify diluent properties parameters.
	Voltage applied is too high.	Confirm the voltage and current values on the display for measurement. Measure again at 60V for standard Concentration cell and at 20V for High Concentration cell.
	Cell is not clean.	Clean cell.
	Instrument is affected by vibration of the work surface.	Remove or turn off the device that is causing the work surface to vibrate.
The base frequency cannot be stabilized	Instrument is affected by vibration of the work surface.	Remove or turn off the device that is causing the work surface to vibrate.
	Malfunction of modulator.	Contact Beckman Coulter Field Service.
Appearance of side peaks	Problem with sample.	Check purity of diluent and sample.
	The instrument has been affected by vibration.	Remove or turn off the device that is causing the work surface to vibrate.
	Light axis of scattered light has shifted.	Contact Beckman Coulter Field Service.
	Light axis of reference light has shifted.	Contact Beckman Coulter Field Service.
	Malfunction of modulator.	Contact Beckman Coulter Field Service.
Peaks become small	Light axis of scattered light has shifted.	Contact Beckman Coulter Field Service.
	Light axis of reference light has shifted.	Contact Beckman Coulter Field Service.
A communication error with Auto Titrator	USB cable not connected.	Reconnect USB cable from the Auto Titrator to the USB port on the back of the controller labeled Titrator.
	Error of the communication port.	Contact Beckman Coulter Field Service.

Table 2.1 DelsaNano Diagnostics *(Continued)*

Symptom	Cause	Corrective Action
Sensitivity of pH Electrode has diminished	Lowering of sensitivity of pH electrode. When sensitivity is less than 90%, a warning appears.	• Confirm the calibration history of pH electrode. • Exchange the inner solution (KCL solution), and calibrate the electrode. • If sensitivity does not improve, exchange the electrode.
Dissymmetric potential of pH electrode has exceeded the limit (when using the Titrator/ when calibrating the pH sensor)	Dissymmetric potential of pH electrode has exceeded the limit.	• Confirm the calibration history of pH electrode. • Exchange the inner solution (KCL solution), and calibrate the electrode. • If sensitivity does not improve, exchange the electrode.
Competence of pH electrode has exceeded the limit (when using the Titrator/ when calibrating the pH sensor)	Competence of pH electrode has exceeded the limit.	• Confirm the calibration history of pH electrode. • Exchange the inner solution (KCL solution), and calibrate the electrode. • If sensitivity does not improve, exchange the electrode.
pH cannot be measured/ Titration volume cannot be calculated (when using the Titrator)	Titration volume cannot be calculated.	• Confirm whether the concentration of titration solution has been set correctly. Set up the concentration of titration solution again. • Confirm whether the volume of sample has been entered correctly. Re-enter the sample volume.
Titration volume will exceed the limit	It was judged that the total titration volume would exceed the volume of sample.	• Confirm whether the concentration of titration solution is consistent with that has been set by the software. Enter the concentration of titration solution to be the same as that has set by the software. • Confirm the concentration of titration solution. Use a titration solution of higher concentration. • Confirm whether the volume of sample entered to the software was correct. Enter the volume of sample to be entered to the software.

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CHAPTER 3

Regulatory Compliance

This chapter describes the relevant portions of the 21 CFR Part 11 regulations and their implementation using the DelsaNano software.

Electronic Signatures (21 CFR Part 11)

The Electronic Records and Electronic Signatures Rule (21 CFR Part 11) was established by the FDA to define the requirements for submitting documentation in electronic form and the criteria for approved electronic signatures. Organizations that choose to use electronic records to meet record keeping requirements must comply with 21 CFR Part 11. Because analytical instrument systems such as the DelsaNano generate electronic records, these systems must comply with the Electronic Records Rule.

NOTE It is important to realize that implementation and compliance of the rule remains the responsibility of the organization or entity creating and signing the electronic records in question. Proper procedures and practices, such as GLP and cGMP, are as much a part of overall compliance with these regulations as are the features of the DelsaNano software.

Security Controls

A DelsaNano installation follows a procedure designed to ensure proper operation, maintenance and administration for system security and data integrity. Users interacting with the system must follow these procedures. The responsibility is with the organization generating electronic records and signatures. The DelsaNano software is a component, albeit a vital one, of the overall process.

The controls to be applied to a closed system are specified in Subpart B, Section 11.10.

System Validation

Other controls are addressed either by the DelsaNano software itself, or in combination with end user procedures. Most important is the validation of systems to ensure accuracy, reliability, consistent intended performance, and the ability to discern invalid or altered records. The complete and overall validation of the system, as developed by the organization, ensures the integrity of the system and its data. The features of the DelsaNano software are designed to comply with the specifications of these regulations.

The DelsaNano software employs a system of usernames and passwords, to ensure that only authorized individuals can use the system, electronically sign a record, access the operation or computer system input or output device, alter a record, or perform the operation at hand.

File History and Tracking

File History is enabled for any file created by the DelsaNano software in 21 CFR Security mode. Under these conditions, all changes made to a file are automatically recorded. These changes consist of computer-generated, time-stamped audit trails to independently record the data and time of operator entries and actions that create, modify, or delete electronic records.

When a change to a file is detected, the DelsaNano software automatically records the identity of the user making the change, the date and timestamp of the change, the parameter that has changed, the old value and the new value. The user is also required to "sign" the record electronically and enter a reason for the change.

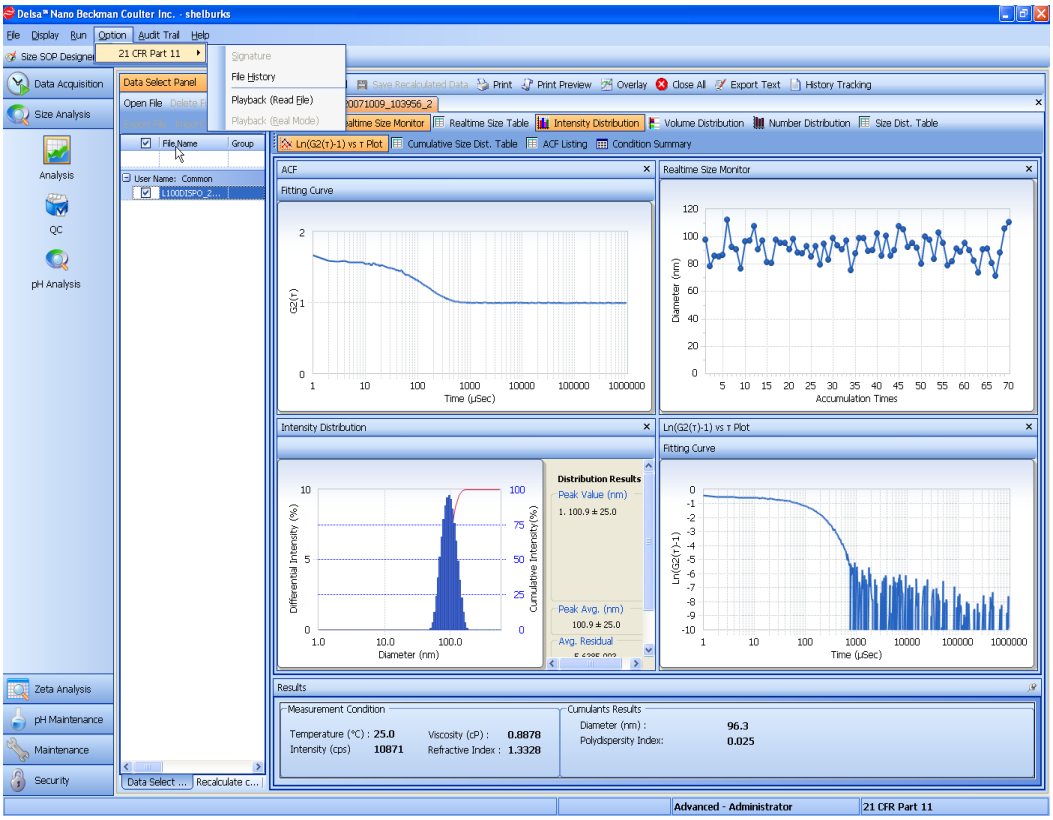
The audit trail is stored as History Tracking inside the file itself, so that record changes do not obscure previously recorded information and the file is suitable for inspection, review, and copying by the agency. This ensures that a complete and continuous record of all changes to the file is maintained.

Accessing File History

To access file history:

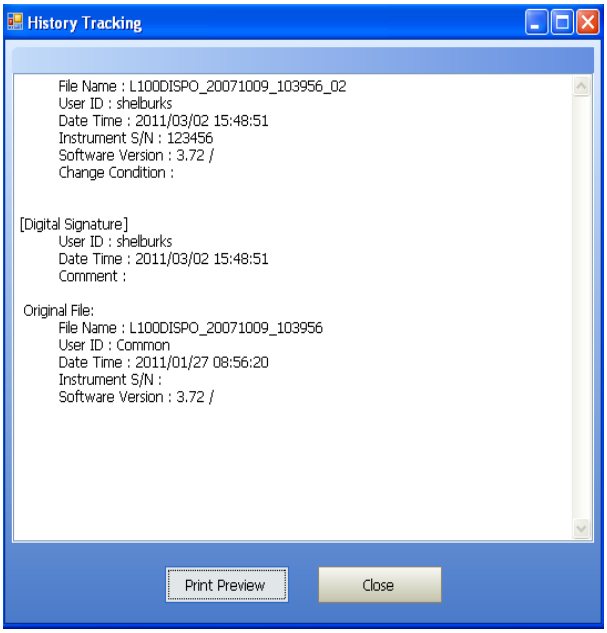
1 Select **Option > 21 CFR Part 11 > File History** on the Main Menu bar. Or, after you open a Size or Zeta Potential file, click **File History** in the upper right of the window. Figure 3.1 shows the location of both options.

Figure 3.1 File History Menu Selection and File History Button



2 The File History (History Tracking) dialog opens.

Figure 3.2 History Tracking Dialog



3 To print a copy of the file history information, click **Print and Close**. To close the dialog, click **Close**.

Playback

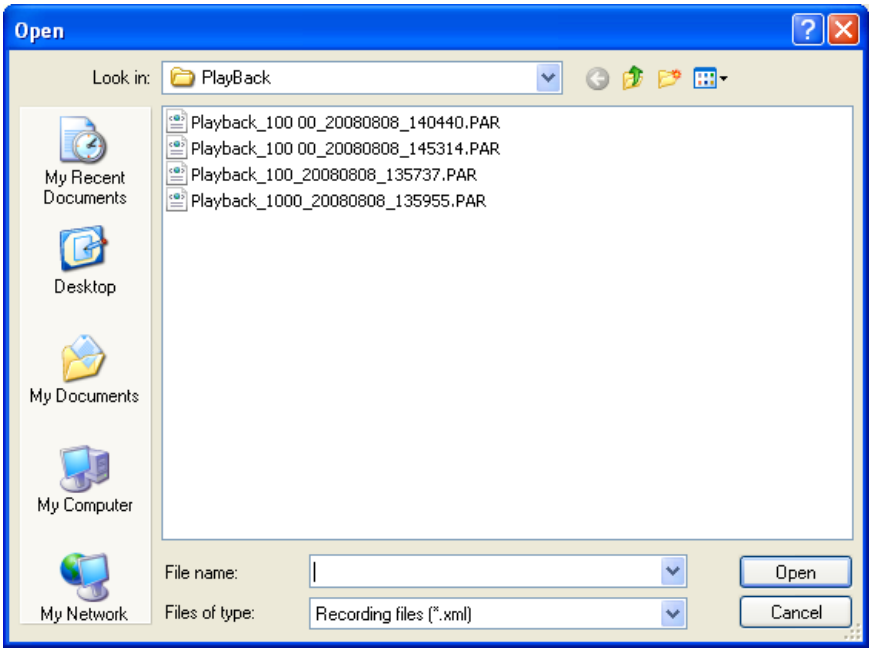
Playback is available when 21 CFR Part 11 is enabled. When selected, Playback causes the instrument to use Size/Zeta Potential SOP settings that were previously used to analyze the sample.

To select Playback mode:

1 Verify that 21 CFR Part 11 security is enabled.

2 Select **Option > 21 CFR Part 11 > Playback** on the Main menu bar. The Playback File Selection dialog opens.

Figure 3.3 Playback File Selection Dialog



3 Select the name of the Playback file you want, and click **Open**. The selected Size/Zeta Potential SOP for analysis is recalled. The Playback file is saved with the same sample name used during measurement. File extension ".PAR" indicates size SOP, and file extension ".ZETA" indicates zeta SOP.

4 To return the instrument to Real mode, select **Option > 21 CFR Part 11 > Playback (Real Mode)**.

Audit Trails

In addition to the auditing associated with the electronic file itself, the DelsaNano offers four additional levels of auditing, as follows. This section describes how to display the audit trails, change audit trail options, and export audit trail files.

- Error audit trail stores and records errors associated with the system; for example, date/time, user name, communication errors, details, and so on.
- pH Calibration audit trail stores and records pH calibration history; for example, date/time, user name, coefficients of acids and bases, temperature, asymmetric potential, response and sensitivity, and so on.
- Operation audit trail records and stores information at a system level; for example, who logged in when, when users were added to the system, all operational steps, and so on.
- Parameter audit trail stores and records parameter change history; for example, date/time, user name, parameter group, parameter, changed value, and so on.

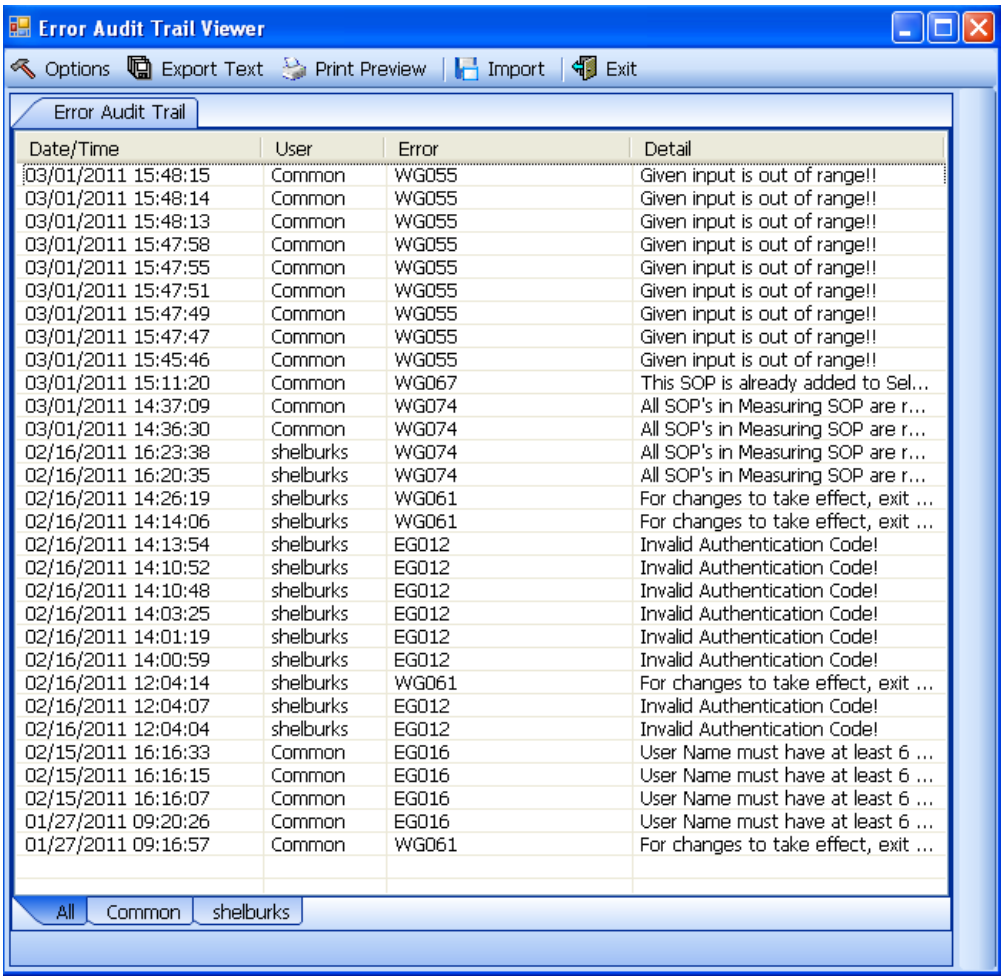
Displaying the Audit Trails

To display audit trail information:

1 Select **Audit Trail** on the Main Menu bar. The list of audit trails appears on the drop-down menu.

2 Select the name of the audit trail to display. This opens a viewer that provides details on the activities for the type of audit trail you select. See Figure 3.4 for an example of the Error audit trail.

Figure 3.4 Error Audit Trail



3 You can view the errors in the Error audit trail in one of two modes: *All* displays all errors, *Individual User* displays the errors of a particular user. To change modes, select the appropriate tab at the bottom of the viewer window.

4 To close the viewer window, click **Exit**.

Changing Audit Trail Options

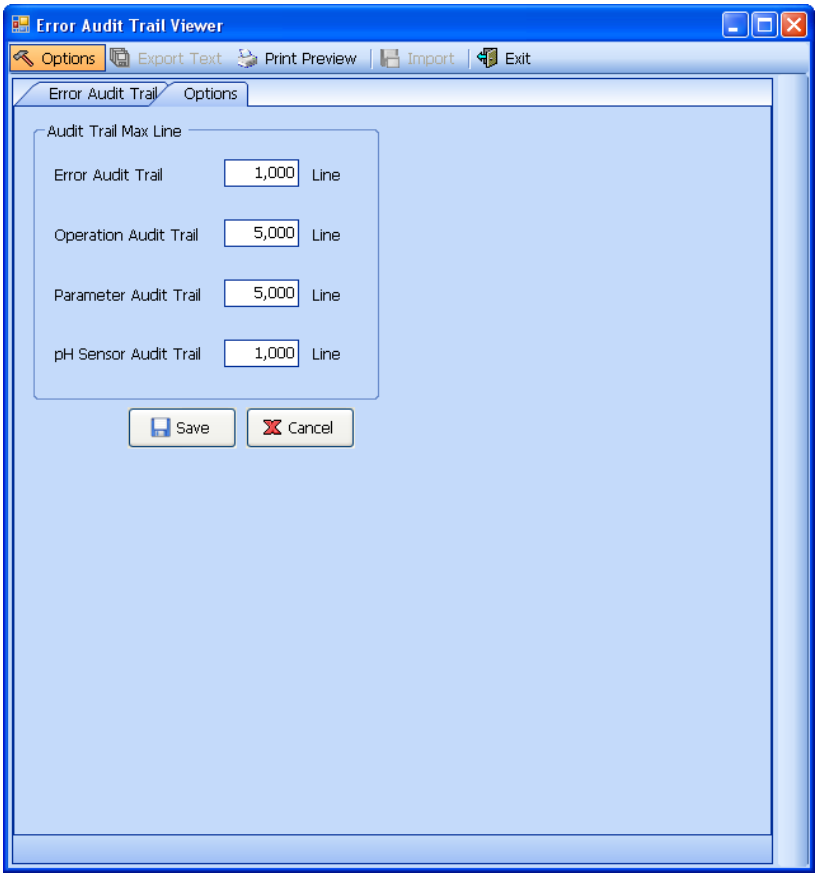
To change audit trail options:

1 Select **Audit Trail** on the Main Menu bar. The list of audit trails appears on the drop-down menu.

2 Select the name of the audit trail to display.

3 In the viewer window, click **Options**. The Options tab displays the maximum number of lines that have been set for each audit trail (Figure 3.5).

Figure 3.5 Audit Trail Options



4 Change the values in the fields provided, as appropriate.

5 Click **Save**.

Exporting Audit Trail Files

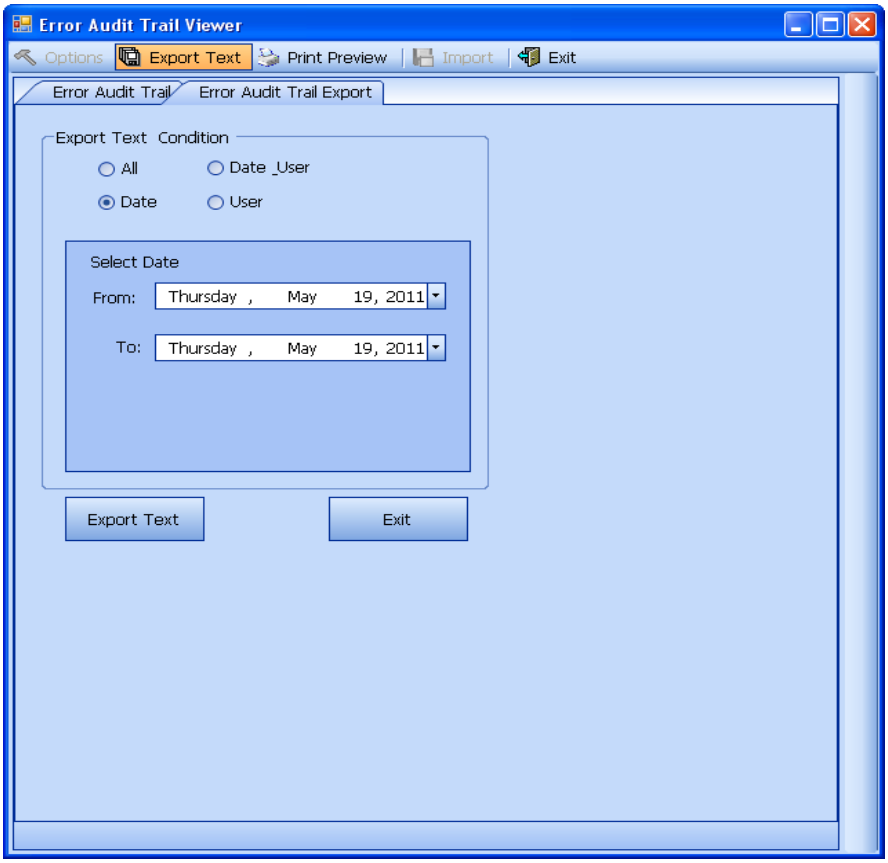
To export audit trail files:

1 Select **Audit Trail** on the Main Menu bar. The list of audit trails appears on the drop-down menu.

2 Select the name of the audit trail to display.

3 In the viewer window, click **Export Text**. The Export tab displays fields for setting export options.

Figure 3.6 Audit Trail Export Text Options



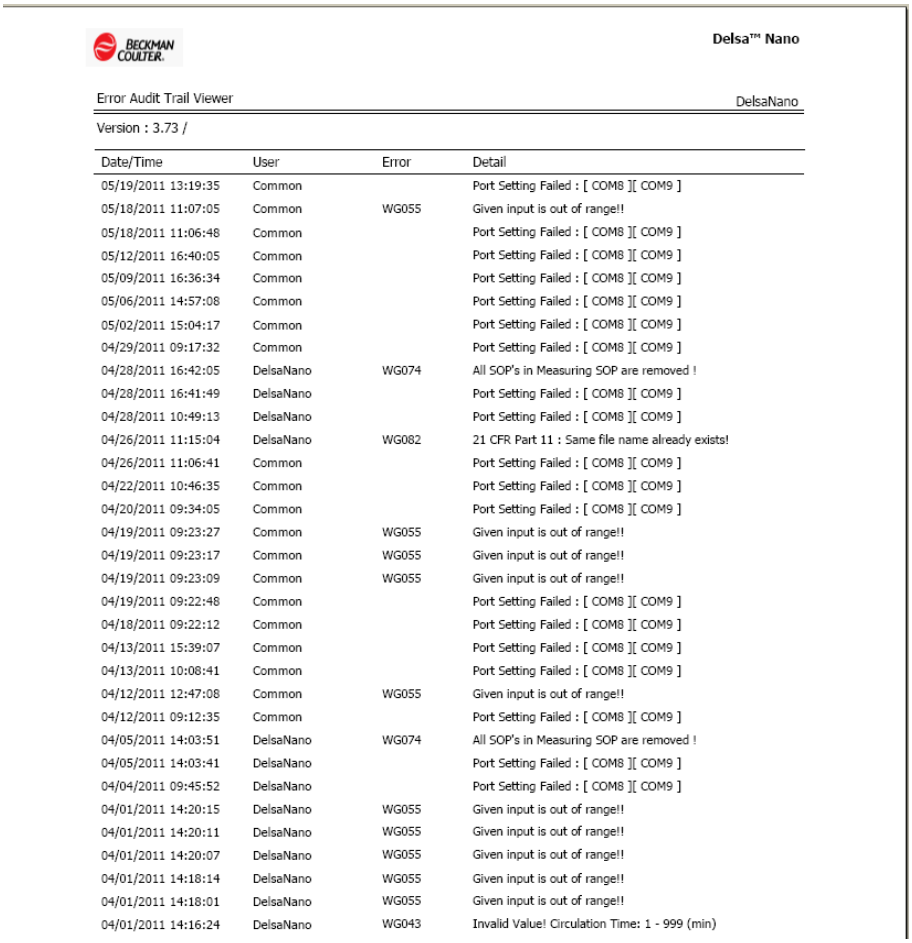
4 Select the condition and the from and to dates as appropriate.

5 Click **Export Text**. A Windows dialog opens to allow you to select an export location and provide a name for the text file.

6 Click **Save** to export the text file. The dialog closes.

7 Click **Print Preview** to print the text file. A Print Preview window opens (Figure 3.7) to show the print results prior to printing.

Figure 3.7 Print Preview



Delsa™ Nano

Error Audit Trail Viewer — DelsaNano

Version : 3.73 /

Date/Time	User	Error	Detail
05/19/2011 13:19:35	Common		Port Setting Failed : [COM8][COM9]
05/18/2011 11:07:05	Common	WG055	Given input is out of range!!
05/18/2011 11:06:48	Common		Port Setting Failed : [COM8][COM9]
05/12/2011 16:40:05	Common		Port Setting Failed : [COM8][COM9]
05/09/2011 16:36:34	Common		Port Setting Failed : [COM8][COM9]
05/06/2011 14:57:08	Common		Port Setting Failed : [COM8][COM9]
05/02/2011 15:04:17	Common		Port Setting Failed : [COM8][COM9]
04/29/2011 09:17:32	Common		Port Setting Failed : [COM8][COM9]
04/28/2011 16:42:05	DelsaNano	WG074	All SOP's in Measuring SOP are removed !
04/28/2011 16:41:49	DelsaNano		Port Setting Failed : [COM8][COM9]
04/28/2011 10:49:13	DelsaNano		Port Setting Failed : [COM8][COM9]
04/26/2011 11:15:04	DelsaNano	WG082	21 CFR Part 11 : Same file name already exists!
04/26/2011 11:06:41	Common		Port Setting Failed : [COM8][COM9]
04/22/2011 10:46:35	Common		Port Setting Failed : [COM8][COM9]
04/20/2011 09:34:05	Common		Port Setting Failed : [COM8][COM9]
04/19/2011 09:23:27	Common	WG055	Given input is out of range!!
04/19/2011 09:23:17	Common	WG055	Given input is out of range!!
04/19/2011 09:23:09	Common	WG055	Given input is out of range!!
04/19/2011 09:22:48	Common		Port Setting Failed : [COM8][COM9]
04/18/2011 09:22:12	Common		Port Setting Failed : [COM8][COM9]
04/13/2011 15:39:07	Common		Port Setting Failed : [COM8][COM9]
04/13/2011 10:08:41	Common		Port Setting Failed : [COM8][COM9]
04/12/2011 12:47:08	Common	WG055	Given input is out of range!!
04/12/2011 09:12:35	Common		Port Setting Failed : [COM8][COM9]
04/05/2011 14:03:51	DelsaNano	WG074	All SOP's in Measuring SOP are removed !
04/05/2011 14:03:41	DelsaNano		Port Setting Failed : [COM8][COM9]
04/04/2011 09:45:52	DelsaNano		Port Setting Failed : [COM8][COM9]
04/01/2011 14:20:15	DelsaNano	WG055	Given input is out of range!!
04/01/2011 14:20:11	DelsaNano	WG055	Given input is out of range!!
04/01/2011 14:20:07	DelsaNano	WG055	Given input is out of range!!
04/01/2011 14:18:14	DelsaNano	WG055	Given input is out of range!!
04/01/2011 14:18:01	DelsaNano	WG055	Given input is out of range!!
04/01/2011 14:16:24	DelsaNano	WG043	Invalid Value! Circulation Time: 1 - 999 (min)

8 To print, click the Print icon.

Maintaining Electronic Signatures

DelsaNano software supports non-biometric signatures. Non-biometric signatures are computer generated and employ at least two distinct identification components such as an identification code and password.

Applying Electronic Signatures

There are several requirements for the control of electronic signatures. Through the application of DelsaNano user and password configuration procedures, the system can be configured to ensure that use of these identifiers by someone other than the owner can be performed only with the owner's permission.

The DelsaNano software uses the application of the username and password to authenticate the user making and saving the changes, in conjunction with file history and audit trails, to independently record the date and time of operator entries and actions that create, modify, or delete electronic records.

Generating Electronic Signatures

The DelsaNano software employs User IDs and passwords to verify the identify of each user logging into the system.

To generate an electronic signature:

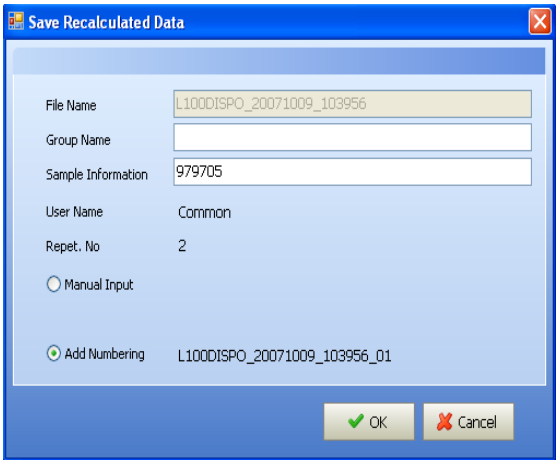
NOTE The following procedure is an example of how to generate an electronic signature. Any instance of data modification (for example, changing the name of a file) generates an electronic signature.

1 Select **File > Open** on the Main Menu bar, and select any Size or Zeta Potential data file run under 21 CFR Part 11 security mode from the Data Select Panel.

2 Recalculate the data.

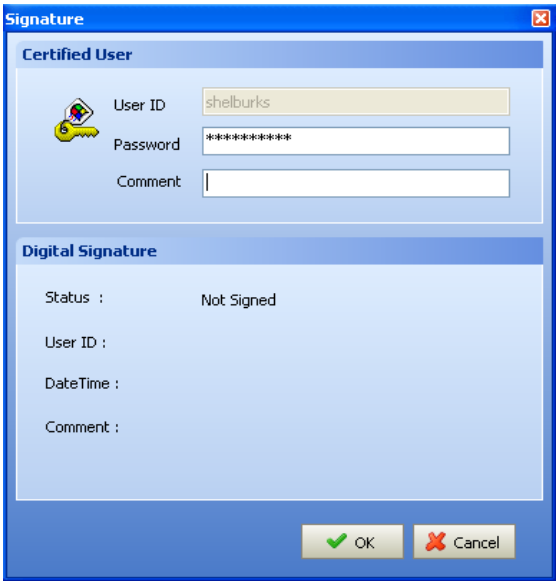
3 Click **Save Recalculated Data**. The Save Recalculated Data dialog opens.

Figure 3.8 Save Recalculated Data Dialog



4 Fill in the fields provided, and click **OK**. The Signature dialog opens. Before a signature is entered, the Status field in the Digital Signature field shows “Not Signed.”

Figure 3.9 Signature Dialog

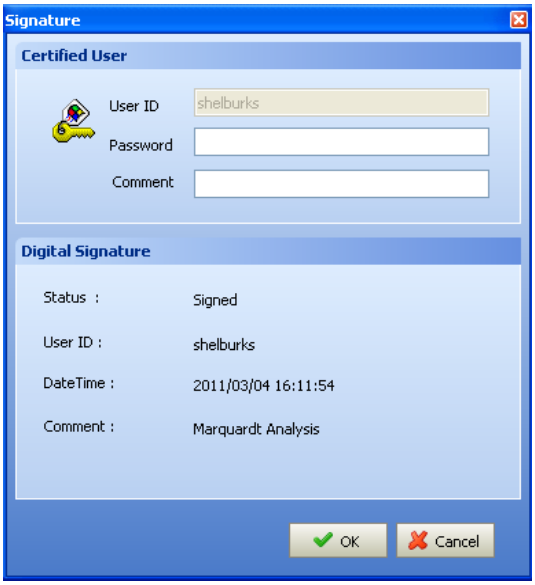


5 Enter your Password, a Comment (required), and click **OK**.

To view an added electronic signature:

1 Select **Option > 21 CFR Part 11 > Signature** on the Main Menu bar.

Figure 3.10 Signature Dialog Showing Digital Signature



The status (Signed), User ID, Date and Time stamp, and Comment appear in the Digital Signature fields.

2 Click **OK** to close the dialog.

Additional Security Features

The DelsaNano software offers an important additional level of security that makes defining and implementing system policies easier.

Data Mirroring allows you to store files securely in a separate location. This feature is available to users at the Administrator or Supervisor level.

For information on configuring Data Mirroring, see CHAPTER 1, *Operation*

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CHAPTER 4

Maintenance

This chapter describes the maintenance procedures that should be performed regularly.

DelsaNano Care

Keep the DelsaNano clean and periodically check for spills. Be sure to power off and unplug the DelsaNano prior to cleaning.

Constantly monitor the equipment for the presence of foreign materials. If you discover foreign materials in the equipment, take appropriate measures, such as following warning labels, when attempting to stop the equipment.

Cleaning the Exterior of the DelsaNano

If the exterior of the equipment or the inside of the cover becomes dirty, clean by wiping with a soft cloth dampened with water or a neutral solvent.

Cleaning the Cell Block

If the cell block becomes dirty, clean by wiping with a soft cloth dampened with water or a neutral solvent. Severe buildup of dirt from fluid spills may interfere with measurements. Stop using the equipment if you suspect that it is not functioning normally.

Replacing the DelsaNano and Auto Titrator Fuses

The DelsaNano instrument and DelsaNano Auto Titrator each are equipped with two fuses in the power socket on the rear panel. Follow these steps to replace the fuses in either device.

To change the fuses:

1. Turn the power off and unplug the power cord.

2. Using a small, flathead screwdriver, pry open the fuse holder cover from the AC power input module.

3 Carefully remove the fuse holder from the AC power input module.

4 Using your hands, gently remove the blown fuses and replace with two properly rated fuses (per the fuse rating table below).

Figure 4.1 Replacing the DelsaNano Fuses

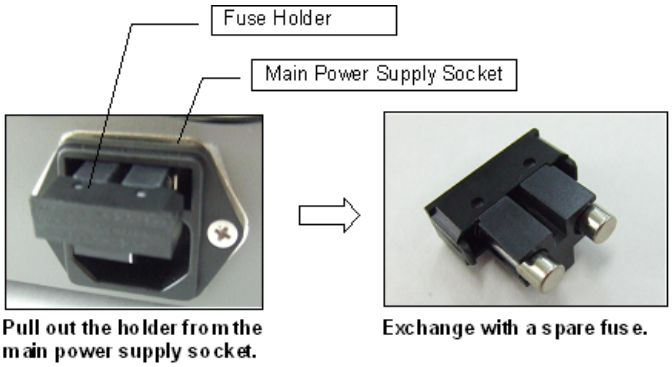


Table 4.1 Fuse Rating Table

Type	Current	Voltage
100–120V	T4A	125V
220–240V	T2A	250V

DelsaNano Auto Titrator Care

⚠ CAUTION

The outside of the instrument is coated with a synthetic resin. Wipe spills immediately.

Cleaning the Auto Titrator

If the instrument surfaces or inside cover are dirty, clean by wiping with a soft cloth dampened with water or a neutral detergent.

Replacing the Auto Titrator Fuses

The DelsaNano Auto Titrator is equipped with two fuses in the power socket on the rear panel. For instructions on replacing the Auto Titrator fuses, see APPENDIX A, *Auto Titrator*.

pH Electrode Maintenance

This section describes how to maintain the pH electrode.

Precautions for Daily Use

The inner solution for the reference electrode must be 3.33 mol/L KCl solution.

Use the electrode after immersion in purified water for 12 hours or more if the responding glass membrane of the electrode has been dried.

Do not touch the electrode connector or allow fluids to contact it.

When the inner solution does not come down to the inside of the responding glass membrane part, shake down the electrode two or three times, holding the cap part of the electrode.

Precautions for First-Time Use or After Long-Term Storage

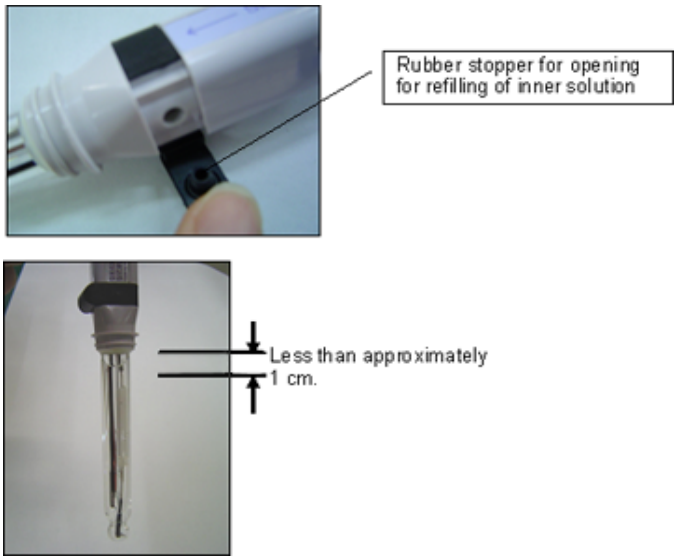
For information on storing the pH electrode, see pH Electrode Storage Conditions.

1. Remove the protective cap.

2. Remove the rubber stopper for opening for draining off the inner solution with a Pasteur pipette.

3. Refill the inner solution.

Figure 4.2 Refilling the pH Electrode



4 Wash the tip of the electrode with purified water, and wipe with a soft cloth.

NOTE If the inside of the protective cap becomes dried, wash the protective cap, then refill with distilled water until the sponge is immersed.

NOTE There may be white crystals of KCL attached to the protective cap and around the opening for refilling the inner solution. This is not harmful to performance. Wash the crystals off with purified water, and use the electrode.

pH Electrode Storage Conditions

To store the pH electrode for 2-3 weeks:

1 Wash off the sample solution well with purified water.

2 Put the rubber stopper on tightly.

3 Remove the plastic vial.

4 Replace the protective cap.

NOTE If the inside of the protective cap becomes dried, wash the protective cap, then refill with distilled water until the sponge is immersed.

To store the pH electrode for longer than one month:

1. Wash off the sample solution well with purified water.
2. Put the rubber stopper on tightly.
3. Remove the plastic vial.
4. Wash the inside of the protective cap with purified water, wipe out the water, and refill with distilled water until the sponge is immersed.
5. Replace the protective cap.

pH Electrode Daily Maintenance

When the electrode has been used for a long time, the sample solution may contaminate the inside of the reference electrode, or its inner solution may become diluted. In this case, follow the steps in Precautions for First-Time Use or After Long-Term Storage.

Improving Response Time

If the response time or reproducibility is decreasing, follow the steps below to improve response time.

To improve response time:

1. Remove the rubber stopper, and suction out all the solution.
2. Reinstall the stopper and refill with new solution. Confirm that the inner solution exudes from the opening.
3. Repeat steps 1 and 2 several times. If performance does not improve, do one of the following, as appropriate:
   - To remove general dirt, wipe off with a soft cloth soaked with neutral detergent.
   - To remove oil residue, wipe off with a soft cloth soaked with an appropriate organic solvent, such as acetone or alcohol.
   - To remove inorganic substances, rinse with approximately 1-Normal HCI. Do not immerse the electrode in a concentrated acid for a long time.

Cell Maintenance

This section contains maintenance instructions for the zeta potential measurement cells and the size measurement cells. The steps required for assembly, disassembly, and cleaning, where applicable, are included.

The zeta potential measurement cells in the DelsaNano cell listing are as follows:

- Disposable Zeta Cell (see Disposable Zeta Cell Maintenance)
- Flat Surface Cell (see Flat Surface Cell Maintenance)
- High Concentration Cell (see High Concentration Cell Maintenance)
- Flow Cell (see Flow Cell Maintenance)
- Low Conductivity Cell (see Low Conductivity Cell Maintenance)

The size measurement cells in the DelsaNano cell listing are as follows:

- Size Cell (Glass); see Figure 4.3.
  This is a glass cuvette (minimum 0.9 mL sample required).

Figure 4.3 Size Measurement Glass Cell

glass cell

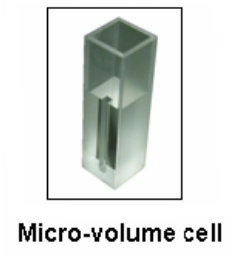
- Size Cell (Disposable); see Figure 4.4.
  This is a plastic cuvette (minimum 0.9 mL sample required).

Figure 4.4 Size Measurement Disposable Cell

disposable cell

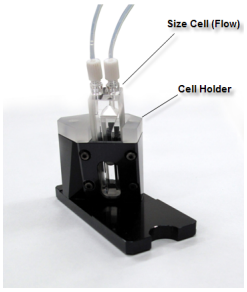
- Size Cell (Micro); see Figure 4.5.
  This is a glass cuvette (minimum 0.03 mL sample required).

  Figure 4.5 Size Measurement Micro Cell



- Size Cell (Flow); see Figure 4.6.
  This is a quartz cuvette (sample volume is 1.8 ml)

  Figure 4.6 Measurement Size Cell (Flow)



- Flow Cell

Measuring with Size Flow Through Cell (using pH Titrator)

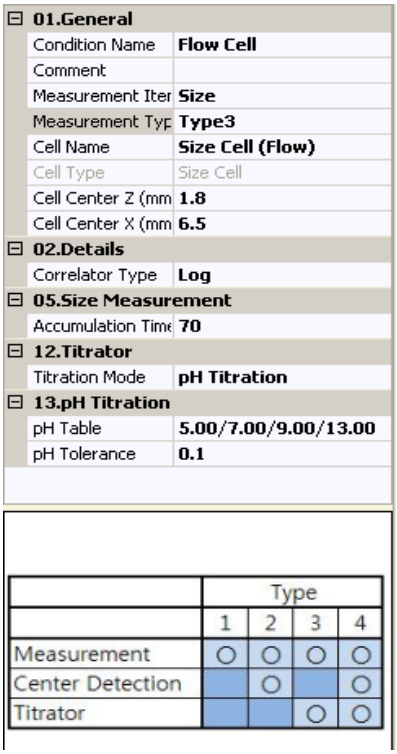
This section describes how to measure with the Size Flow Cell using a pH Titrator.

1 Insert the Cell into the Cell Holder and set it to the instrument.

2 Start the DelsaNano software.

3 Click **Size SOP Designer** and select **Size Cell (Flow)** under Cell Parameters.

4 Select **Type3** or **Type4** under Measurement Size Cell (Flow) Type.



01.General	
Condition Name	**Flow Cell**
Comment	
Measurement Iter	**Size**
Measurement Typ	**Type3**
Cell Name	**Size Cell (Flow)**
Cell Type	Size Cell
Cell Center Z (mm	**1.8**
Cell Center X (mm	**6.5**
02.Details	
Correlator Type	**Log**
05.Size Measurement	
Accumulation Time	**70**
12.Titrator	
Titration Mode	**pH Titration**
13.pH Titration	
pH Table	**5.00/7.00/9.00/13.00**
pH Tolerance	**0.1**

	Type			
	1	2	3	4
Measurement	○	○	○	○
Center Detection		○		○
Titrator			○	○

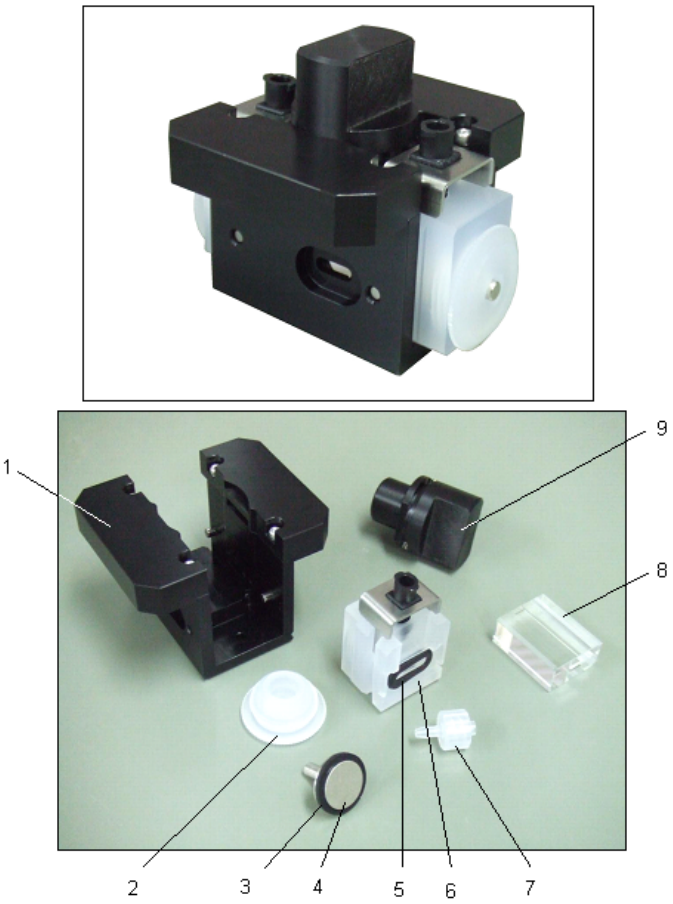
5 Begin the measurement (See Measurement).

6 Analyze the measured data. (See pH Analysis).

Flow Cell Maintenance

This section describes how to disassemble and assemble the Flow Cell, as well as how to wash the glass cell.

Figure 4.7 Flow Cell Components



1. Cell Holder
2. Clamping Knob (2)
3. Packing (8); spare packing (6)
4. Electrode (2)
5. Sealing Rubber (2); spare seal (6)
6. Cell Guide (2)
7. Luer Fitting (2)
8. Glass Cell
9. Cell Stopper

⚠ CAUTION

Be careful not to touch the optical surfaces of the glass cell. The width of the optical surface is 1 mm.

Disassembling the Flow Cell

⚠ CAUTION

Use caution when disassembling the Flow Cell. There may be some residual acidic/alkaline sample solution remaining in the Flow Cell. Wear protective gloves.

To disassemble the Flow Cell:

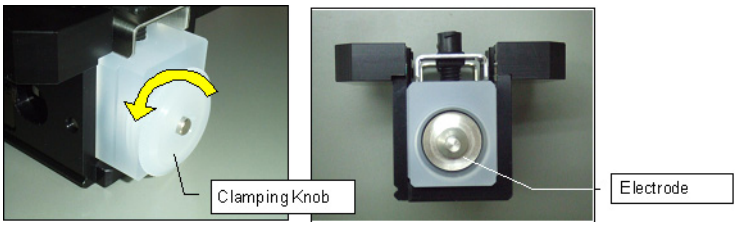
1 Drain the sample solution in the Flow Cell.

2 Remove the flow cell stopper by turning it clockwise.

3 Remove the luer fitting by turning it counter-clockwise.

4 Remove the electrode by turning the clamping knob counter-clockwise (Figure 4.8).

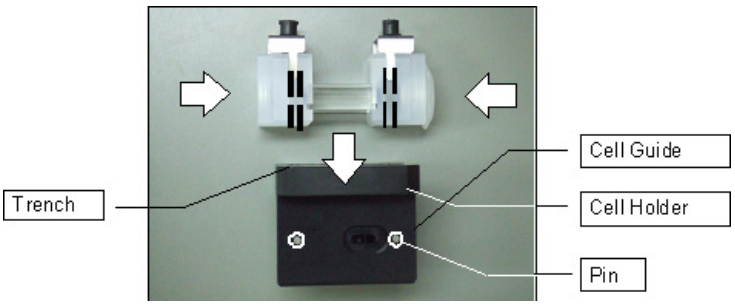
Figure 4.8 Removing the Electrode



5 Repeat steps 1–4 for the opposite side.

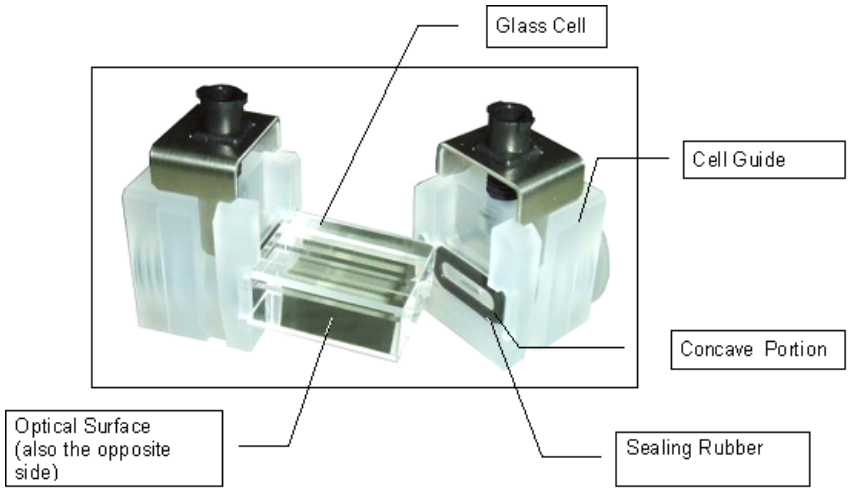
6 Holding the Flow Cell, remove the cell guides from the cell holder. Push the cell guides in while lifting the cell guide out of the cell holder (Figure 4.9).

Figure 4.9 Removing the Cell Guides



7 Remove the glass from the cell guide, and remove the sealing rubber from the gutter of the cell guide.

Figure 4.10 Removing the Glass Cell and Sealing Rubber



Assembling the Flow Cell

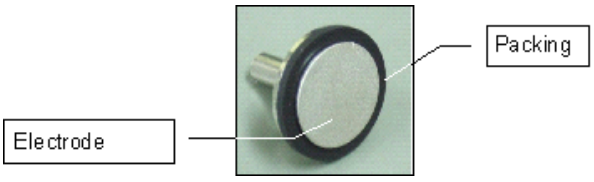
⚠ CAUTION

Be careful not to touch the optical surfaces of the glass cell. The width of the optical surface is 1 mm.

To assemble the Flow Cell:

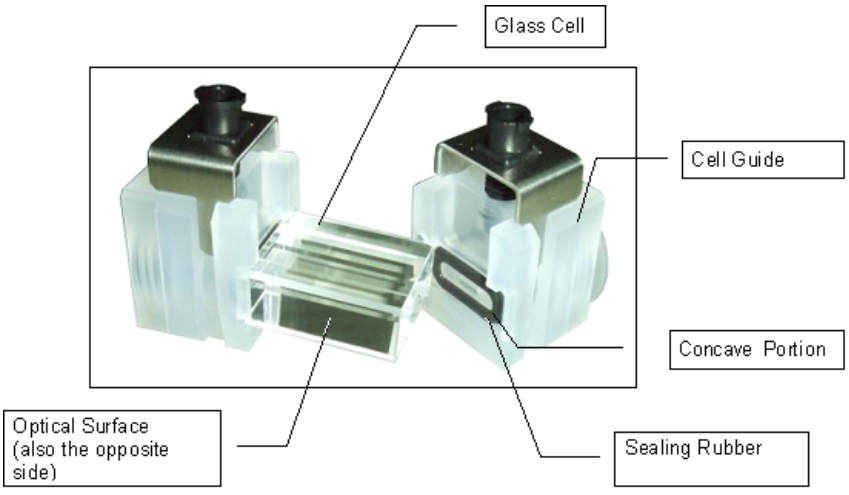
1 Install the electrode packing.

Figure 4.11 Installing the Electrode Packing



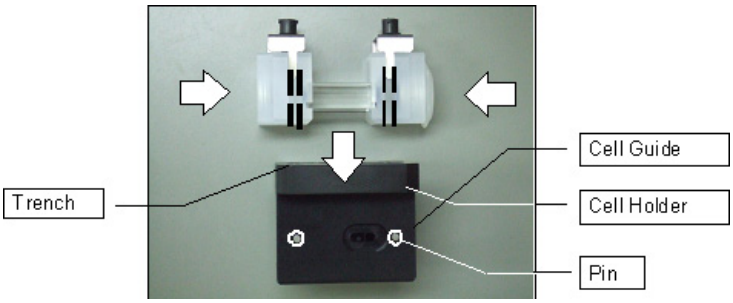
2 Install the sealing rubber to the gutter of the cell guide, and insert the glass cell into the concave portion of the cell guide.

Figure 4.12 Installing the Sealing Rubber



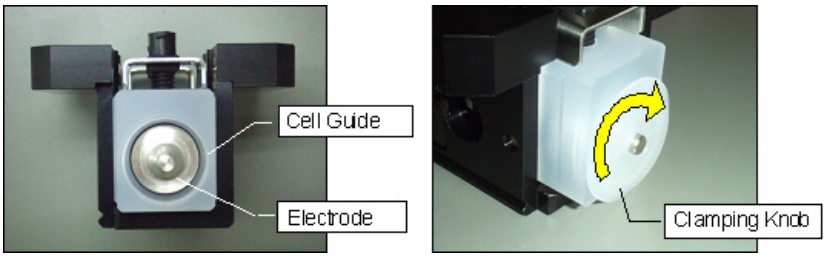
3 Insert the cell guides (holding the Flow Cell) into the cell holder firmly to the bottom of the cell holder, pushing the cell guides against the glass cell and fitting the trenches on the cell guides to the pins on the cell holder.

Figure 4.13 Inserting the Cell Guides



4 Insert the electrode into one of the cell guides, and then set the clamping knob, turning it clockwise.

Figure 4.14 Inserting the Electrode

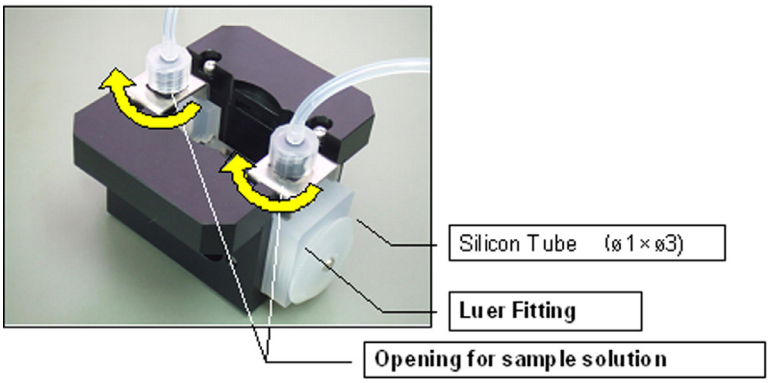


5 Repeat steps 1–4 for the opposite side.

6 Set the luer fitting to the opening of the Flow Cell.

7 Turn the luer fitting clockwise to tighten it.

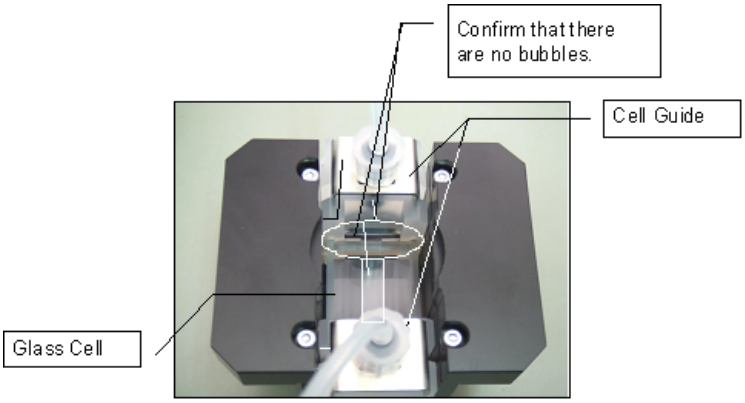
Figure 4.15 Turning the Luer Fitting



8 Fill the cell with sample solution.

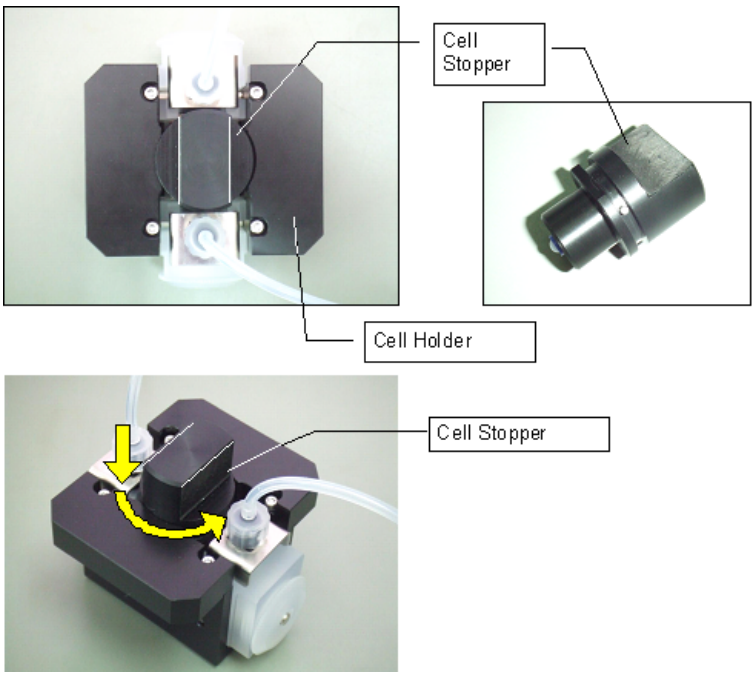
9 Confirm that there are no bubbles in the glass cell and between the cell holder and the glass cell.

Figure 4.16 Checking for Bubbles inside the Glass Cell



10 Set the cell stopper in the direction shown in Figure 4.17, and turn it counter-clockwise, pushing lightly until it stops.

Figure 4.17 Setting the Cell Stopper



Washing the Glass Cell

Be careful not to scratch or damage the glass cell during handling.

Do not wash the cell in an ultrasonic cleaner. Wash the cell as described below.

To wash the glass cell:

1 Wash the glass cell (especially inside the cell) with purified water.

a. When the dirt inside the cell is severe, clean the inside using lens paper.

b. Dip the lens paper in acetone and wrap the lens paper around a thin wire.

2 Rinse with water again, and confirm that no lens paper fibers are remaining in the glass cell.

⚠ CAUTION

The glass cell should be immersed in concentrated sulfuric or hydrochloric acid for a few hours when the dirt is especially severe. Then, wash it well with purified water. (When you use concentrated sulfuric or hydrochloric acid, take appropriate safety precautions according to your laboratory safety officer. Handling these acids is very dangerous.)

3 Wipe off water drops around the glass cell with lens paper.

4 Dry the glass cell in N_2 flow or using a dryer. When you use N_2 flow, it should be passed through a gas filter to reduce spots after drying. When an organic solvent is used for washing, rinse with acetone, and dry the cell.

5 When washing parts other than the glass cell, such as the electrode or packings, immerse the parts in a neutral detergent and insert into an ultrasonic washer. Rinse them well with purified water to remove the detergent completely.

High Concentration Cell Maintenance

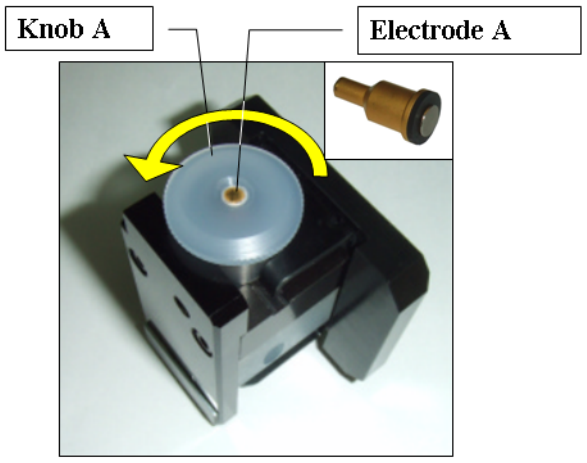
This section describes how to maintain the High Concentration cell.

Disassembling the High Concentration Cell

To disassemble the High Concentration Cell:

1 Turn knob A counter-clockwise to remove electrode A.

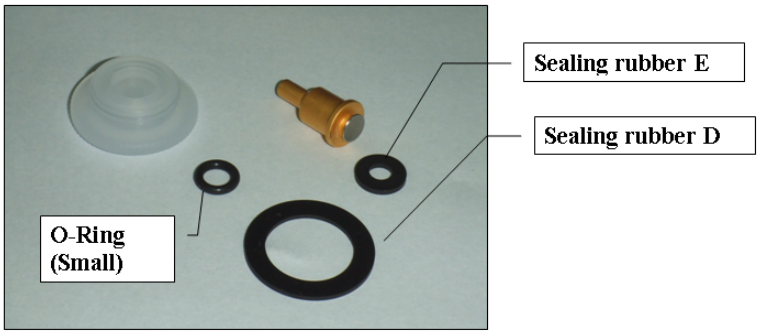
Figure 4.18 Removing the Electrode



2 If applicable, drain the sample solution from the cell.

3 Remove the small O-ring and sealing rubber D from knob A. Remove sealing rubber E from electrode A.

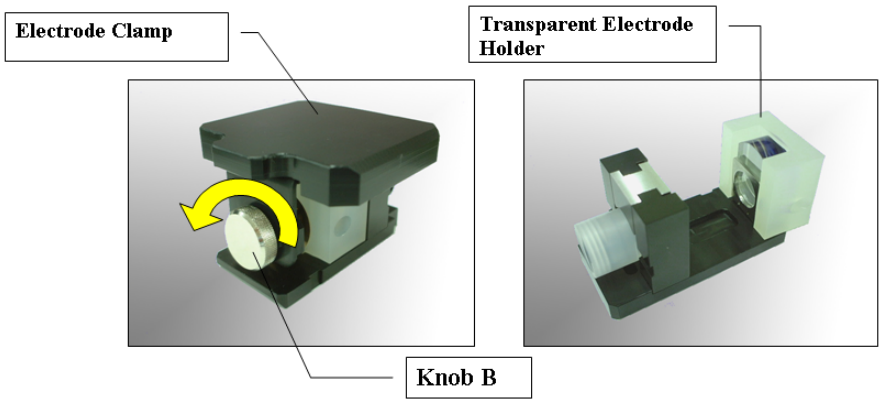
Figure 4.19 Removing the Small O-Ring and the Sealing Rubbers



4 Turn knob B counter-clockwise and lift up the electrode clamp.

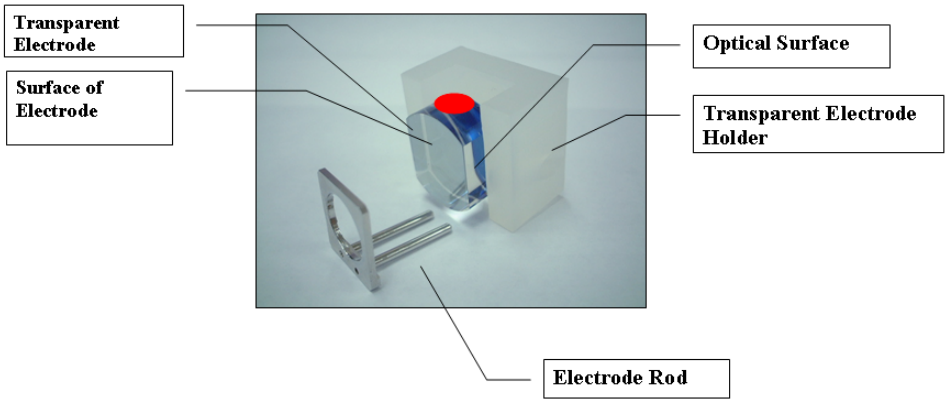
5 Remove the transparent electrode holder.

Figure 4.20 Removing the Transparent Electrode Holder



6 Pull the electrode rod from the transparent electrode holder, and remove the transparent electrode.

Figure 4.21 Removing the Transparent Electrode

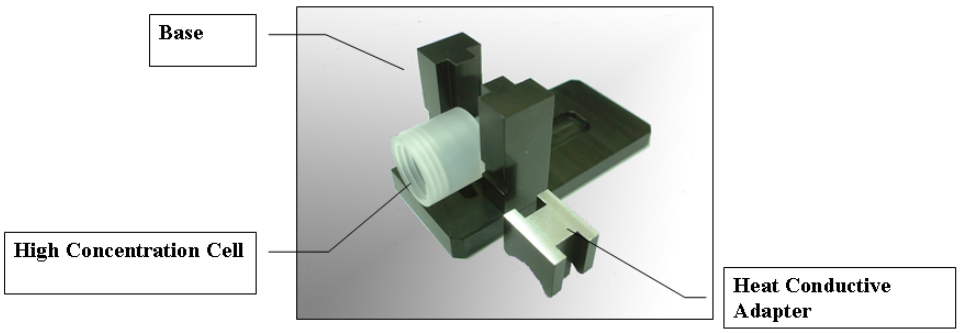


⚠ CAUTION

Do not touch the surface of the electrode and the optical surface. Do not drop the transparent electrode. The transparent electrode is solid glass coated with platinum.

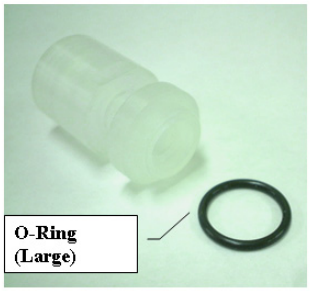
7 Remove the heat conductive adapter upward, and remove the high concentration cell.

Figure 4.22 Removing the High Concentration Cell



8 Remove the large O-ring large from the high concentration cell.

Figure 4.23 Removing the Large O-Ring



9 Rinse the High Concentration Cell.

10 If the High Concentration Cell will not be used from more than a day, store the transparent electrode in a safe place.

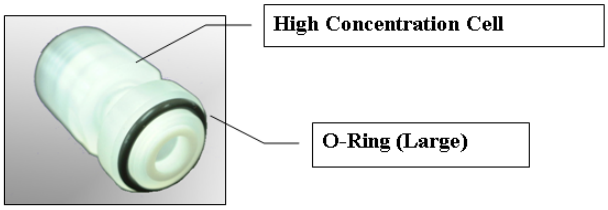
11 Reassemble the High Concentration Cell without the transparent electrode.

Assembling the High Concentration Cell

To assemble the High Concentration Cell:

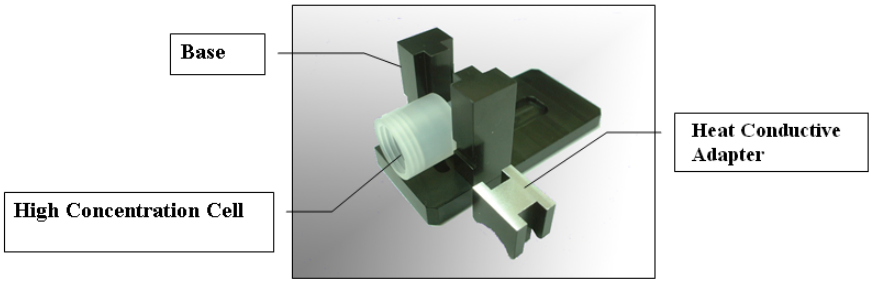
1 Attach the large O-ring.

Figure 4.24 Attaching the Large O-Ring



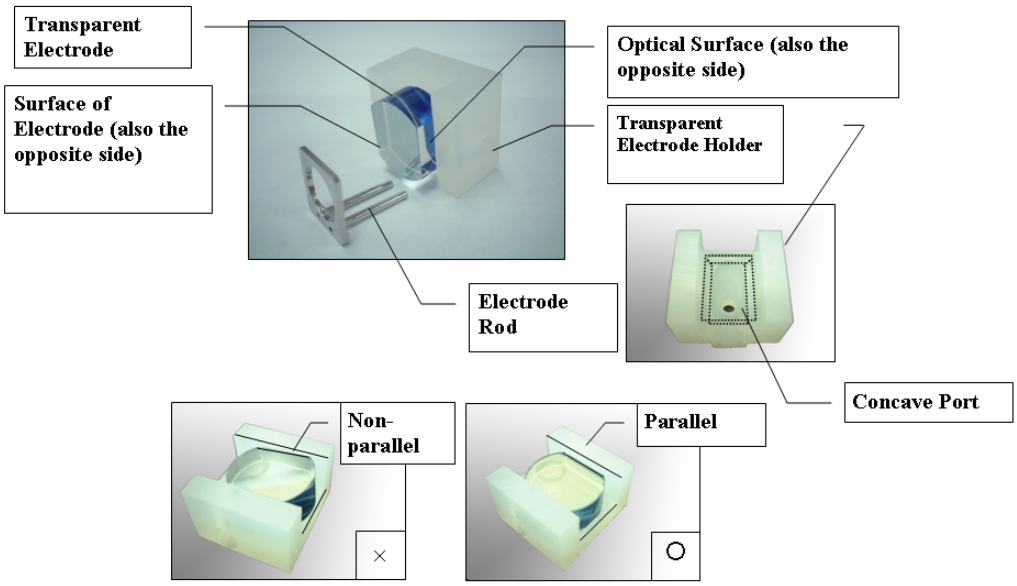
2 Attach the High Concentration Cell to the base, and then attach the heat conductive adapter.

Figure 4.25 Attaching the High Concentration Cell to the Base



3 Attach the transparent electrode to the transparent electrode holder, fitting it into the concave port, and then insert the electrode rod.

Figure 4.26 Attaching the Transparent Electrode to the Holder

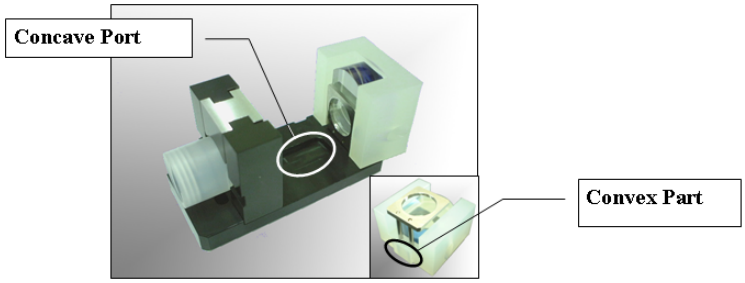


⚠ CAUTION

Do not touch the surface of the transparent electrode and the optical surface. Confirm that the transparent electrode is parallel to the transparent cell holder sides.

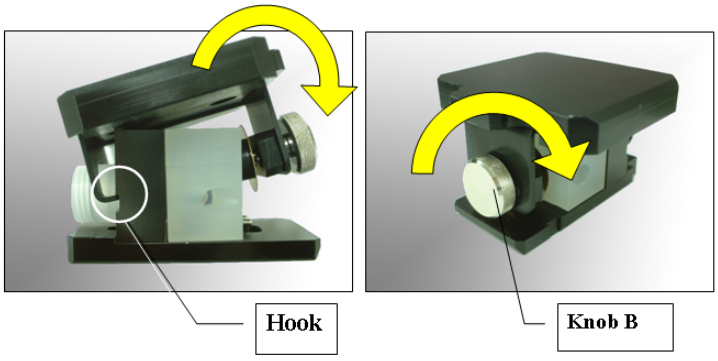
4 Set the transparent electrode holder, lining up the convex part with the concave port in the base.

Figure 4.27 Setting the Transparent Electrode Holder



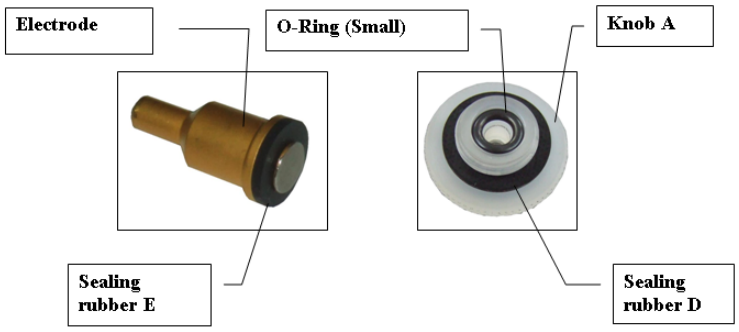
5 Insert the hooked clamp to the concave port in the base. Then, tighten the clamp by turning knob B clockwise.

Figure 4.28 Attaching the Electrode Clamp to the Base



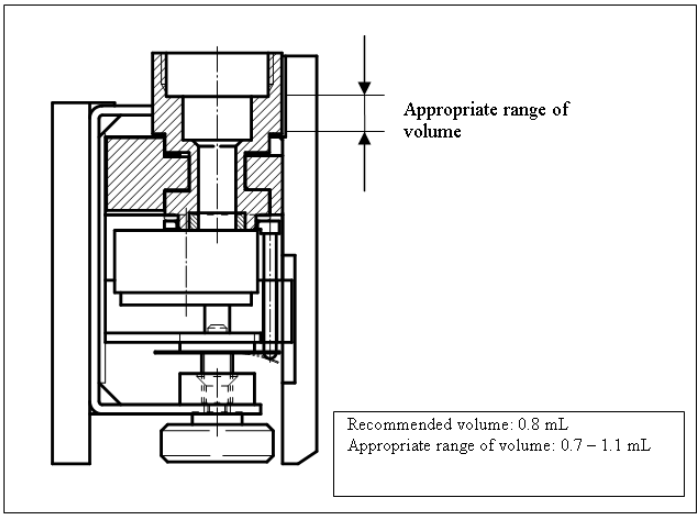
6 Attach sealing rubber E to electrode A. Attach the small O-ring and sealing rubber D to knob A.

Figure 4.29 Attaching the Sealing Rubber and Small O-Ring



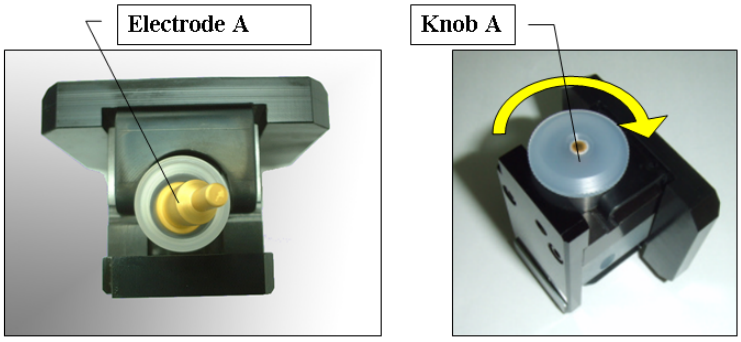
7 Inject the sample solution into the High Concentration Cell.

Figure 4.30 Filling the High Concentration Cell with Sample Solution



8 Insert electrode A into the cell, tilting the cell slightly so that air bubbles do not enter the cell, and then turn knob A clockwise to install electrode A.

Figure 4.31 Inserting the Electrode into the Cell



⚠ CAUTION

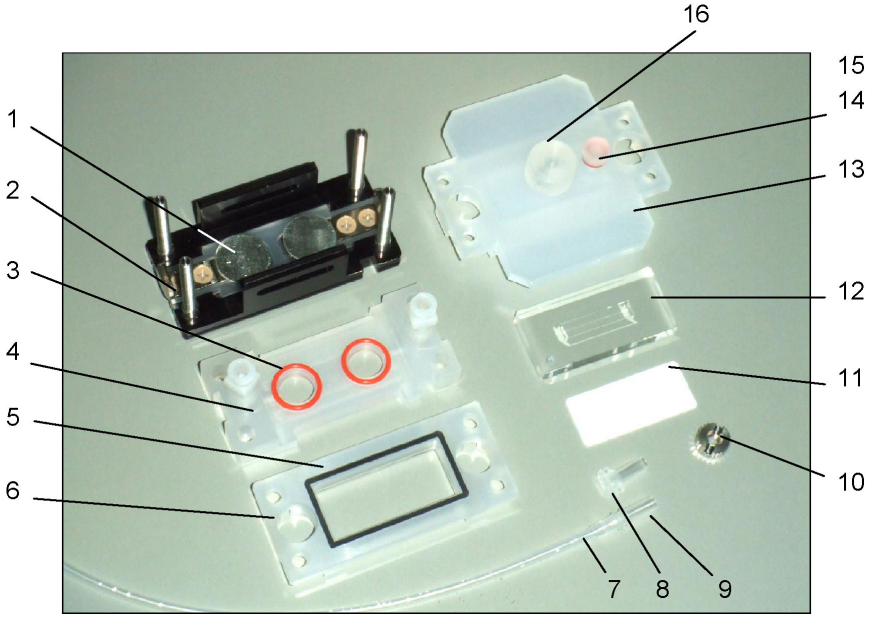
Do not confuse these sealing rubbers with those used for the other measuring cells.

Flat Surface Cell Maintenance

This section describes how to maintain the Flat Surface Cell for Zeta Potential.

Figure 4.32 Flat Surface Cell for Zeta Potential

Figure 4.33 Flat Surface Cell Components



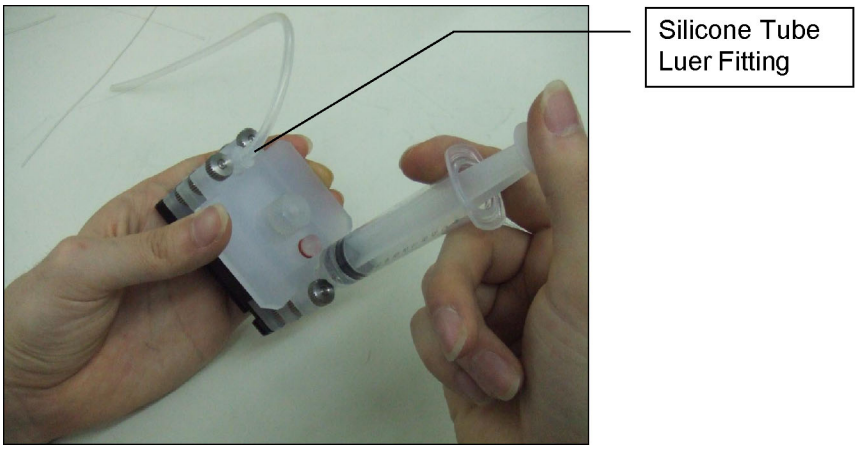
1. Electrode (2)
2. Cell Holder
3. O-Ring (4)
4. Cell Block
5. Cell Seal, translucent (2)
6. Sample Sealing Block
7. Silicone Tube
8. Plug (2)
9. Luer Fitting
10. Fixing Nut (12)
11. Teflon Sheet (and Silicone Sheet)
12. Flat Surface Cell
13. Sample Fixing Block
14. Decompression Cap
15. O-Ring
16. Clamping Knob

Disassembling the Flat Surface Cell

To disassemble the Flat Surface Cell:

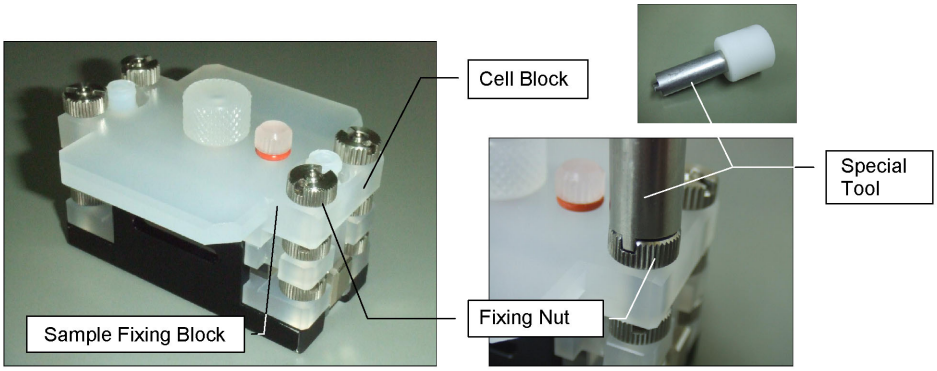
1 Drain the sample solution from the cell. To do this, attach the silicone tube to one of the inlets and a syringe to the other inlet. Inject air into the cell.

Figure 4.34 Removing Solution



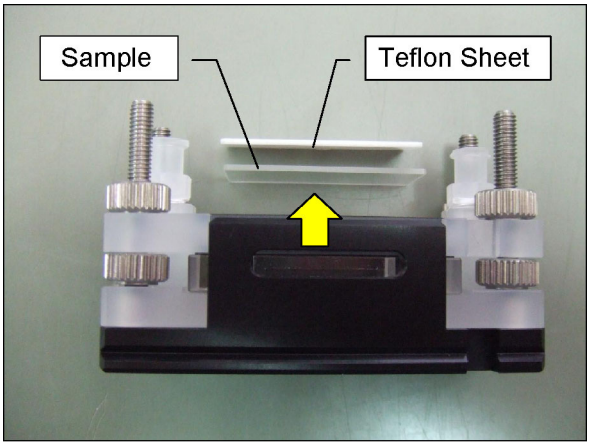
2 Using the special tool, remove the fixing nuts, and then remove the sample fixing block.

Figure 4.35 Removing the Sample Fixing Block



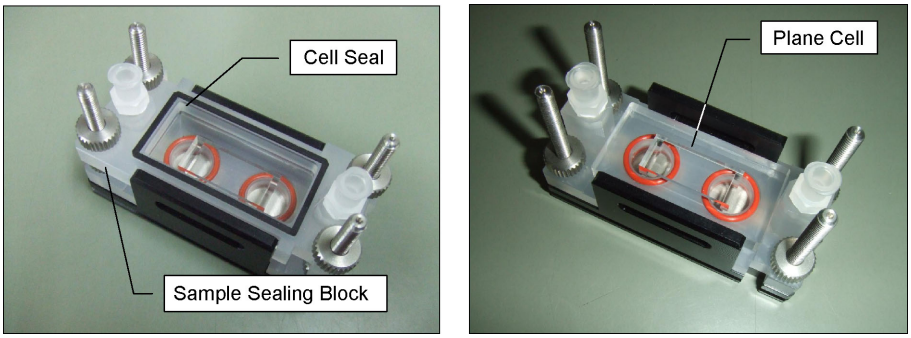
3 Remove the fixing nuts, and then remove teflon sheet or silicone sheet.

Figure 4.36 Removing the Teflon Sheet



4 Remove the fixing nuts, then remove the sample sealing block and cell seals on both sides.

Figure 4.37 Removing the Sample Sealing Block and Cell Seals

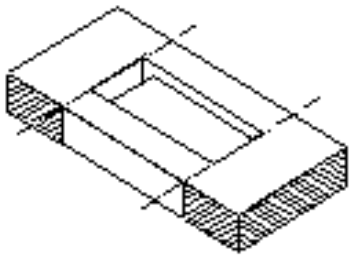


5 Take out the Flat Surface Cell, wash it, wrap it in lens paper, and store it in purified water. Do not dry the Plane Cell.

⚠ CAUTION

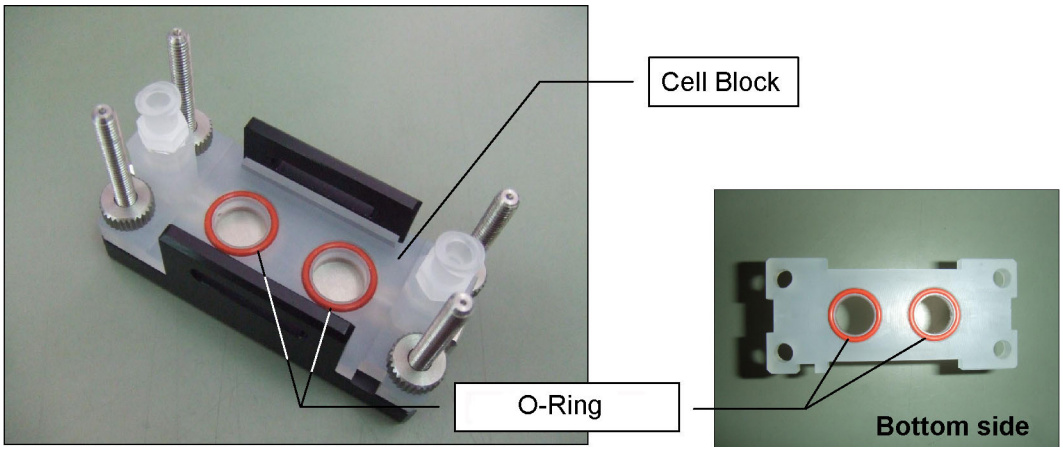
Handle the Plane Cell by the rough surfaces (indicated by the shaded areas in Figure 4.38). Exact measurements may not be possible if other surfaces contain fingerprints or smears. Do not dry the Plane Cell.

Figure 4.38 Plane Cell Rough Surfaces (Shaded)



6 Remove the cell block and O-rings on both sides.

Figure 4.39 Removing the Cell Block and O-Rings



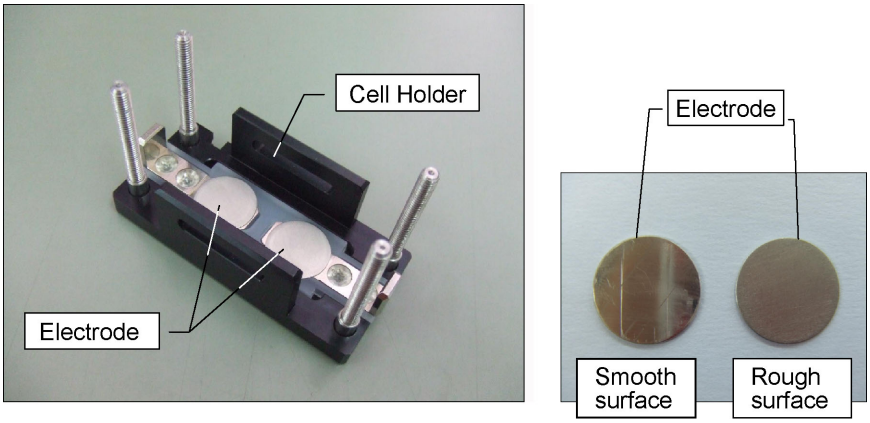
Assembling the Flat Surface Cell

All components of the Flat Surface Cell are symmetrical. Therefore, orientation of the components is not important, unless otherwise indicated.

To assemble the Flat Surface Cell:

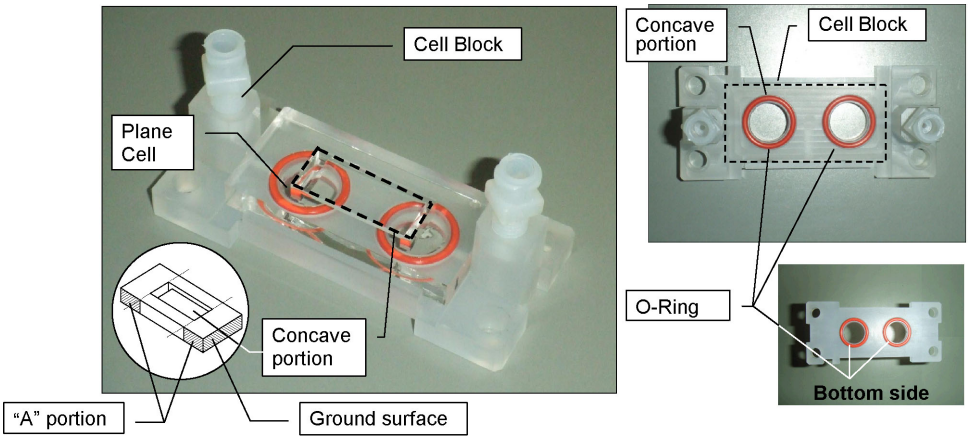
1 Set up the electrode with the rough surface upward to the round concave portion of the cell holder.

Figure 4.40 Setting Up the Electrode



2 Install the two O-rings on both sides of the cell block, and set the Flat Surface Cell with the concave portion upward to the concave portion of the cell block.

Figure 4.41 Installing the O-Rings

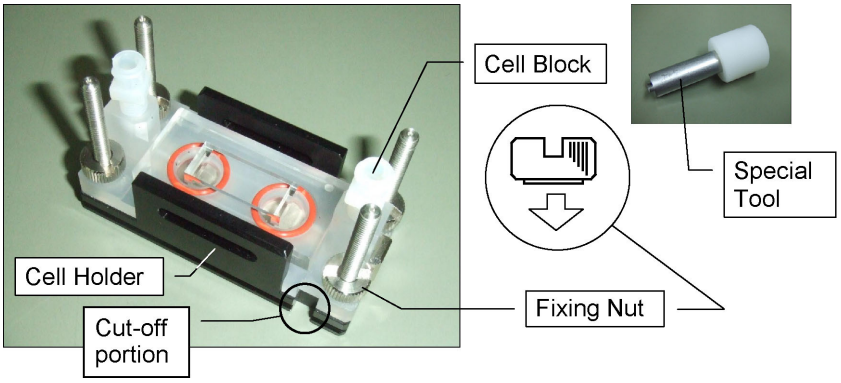


3 Mount the cell block on the cell holder, fitting the cut-off portions together. Then, fasten the fixing nut with the trenched face upward.

⚠ CAUTION

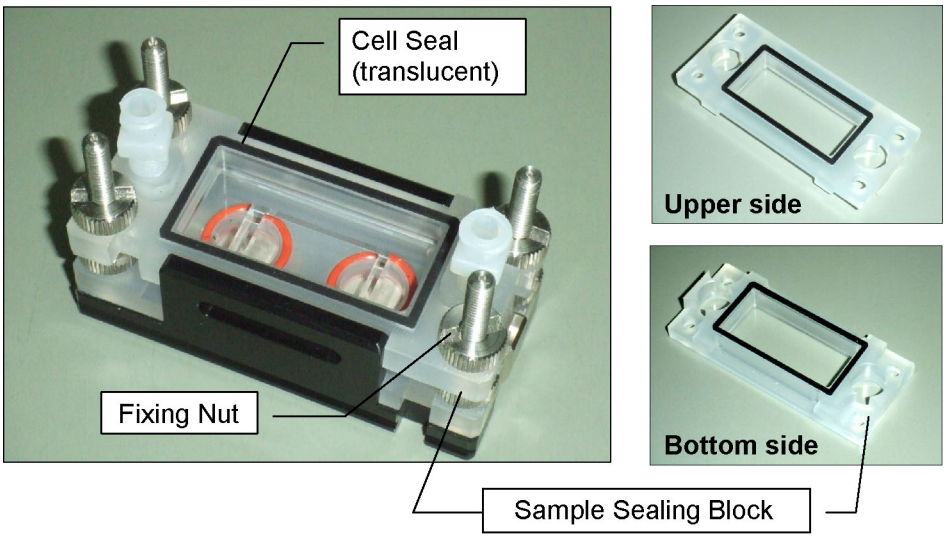
When using the special tool, do not fasten the nut too tightly. This may damage some parts.

Figure 4.42 Mounting the Cell Block



4 Attach a cell seal to each side of the sample sealing block. Mount the sample sealing block with the upper side facing up, and secure with the fixing nut.

Figure 4.43 Attaching the Sample Sealing Block



5 Set the sample and teflon sheet (or silicone sheet) on the Flat Surface Cell.

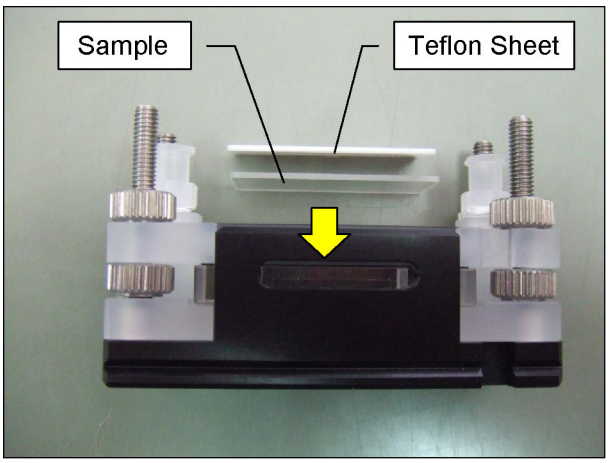
Sample size:

- Recommended (maximum) size: 37´16´5 mm
- Minimum size: 33´14 mm

Sheet selection:

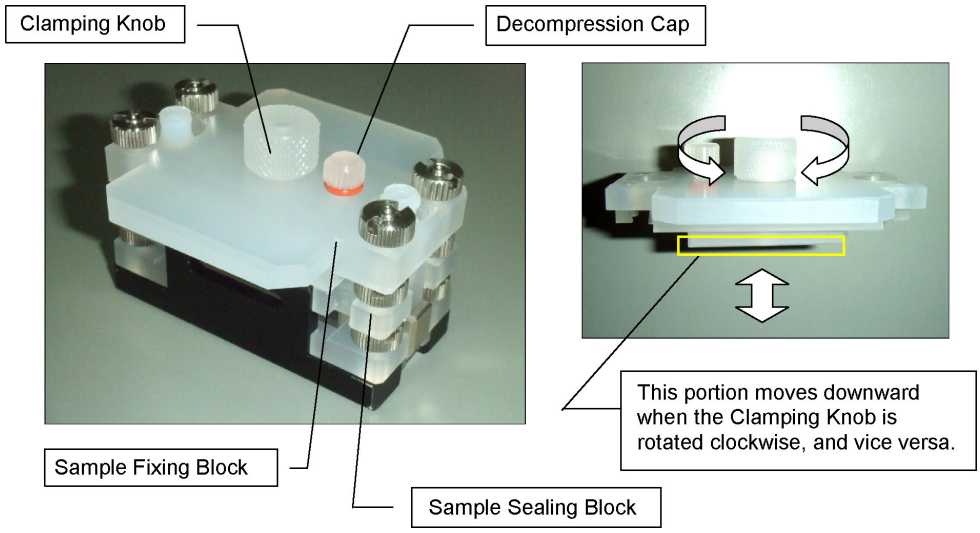
- Teflon sheet: normal samples
- Silicone sheet: samples of low strength

Figure 4.44 Setting the Samples



6 Rotate the clamping knob of the sample fixing block counter-clockwise until it stops, and mount it on the sample sealing block. Fix the sample fixing block with the fixing nut. Then, rotate the clamping knob clockwise to make the sample contact closely with the Flat Surface Cell. Loosen the decompression cap to let the air out.

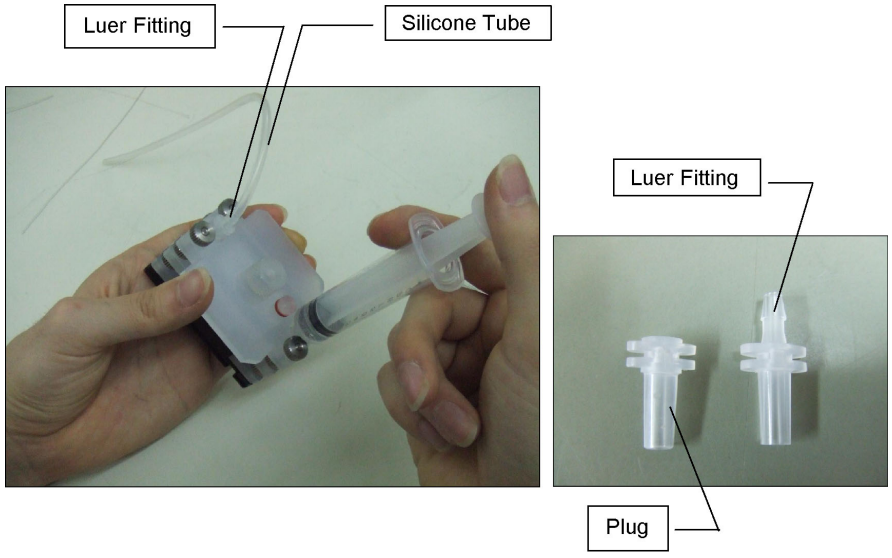
Figure 4.45 Mounting the Sample Fixing Block on the Sample Sealing Block



7 Insert the silicone tube (with the luer fitting) in one of the inlets. From the other inlet, fill the cell with the monitoring particle solution using a syringe.

8 Remove the luer fitting and insert a plug into the inlet. Remove the syringe and put another plug in that inlet.

Figure 4.46 Filling the Cell with Monitoring Particle Solution



⚠ CAUTION

Fill the monitoring particle solution slowly to prevent introducing air bubbles into the cell.

Washing the Flat Surface Cell

Be careful not to damage the Flat Surface Cell during handling.

Do not wash the cell in an ultrasonic cleaner. Wash the cell as described below.

To wash the Flat Surface Cell:

1 Wash the Flat Surface Cell with purified water.

⚠ CAUTION

Do not wash the cell in an alkaline solution. This may cause the coating to peel.

2 Wrap the cell with lens paper, and store it in purified water.

⚠ CAUTION

Do not wipe the cell surfaces. This may cause the coating to peel. If peeling occurs, you must re-coat the Flat Surface Cell.

3 Wipe off water drops around the glass cell with lens paper.

4 When washing parts other than the Flat Surface Cell, such as the O-rings, immerse the parts in a neutral detergent and insert into an ultrasonic washer. Rinse them well with purified water to remove the detergent completely.

Coating the Flat Surface Cell with Polyacrylamide

⚠ CAUTION

Please read the vendor's Material Safety Data Sheet (MSDS) for the Acrylamide before starting the procedure.

Regents:

- 3-Methylacryloxypropltrimethoxysilane (organosilane)
- Acrylamide-HG (acrylamide HG)
- N,N,N',N'–Tetramethyl-ethylenediamine (tetramethyl–ethylenediamine)
- Potassium peroxodisulfate
- Methanol
- 0.1 mol/L Sodium hydroxide
- 0.1 mol/L Hydrochloric acid
- Concentrated sulfuric acid

Equipment:

- Drying oven
- Beakers as necessary

To coat the Flat Surface Cell:

1. Immerse the Flat Surface Cell in concentrated sulfuric acid for 2-3 hours. Wash it well with purified water, and rinse with methanol.

2. Dry the cell in N_2 flow.

3. Place the cell by itself in a drying oven at approximately 160° C. Allow to dry overnight.

⚠ CAUTION

Perform steps 4-8 in a draft chamber.

4 Remove the cell from the drying oven, and let it stand at room temperature for 15 minutes.

5 Immerse the cell in 0.1 mol/L sodium hydroxide solution for 1.5 hours.

6 Wash the cell with purified water, rinse with methanol, and dry it in N_2 flow.

7 Prepare 200 mL of 60% 3–methylacryloxypropyltrimethoxysilane methanol solution. Filter the methanol in advance with 0.1 μm filter.

8 Wash the Flat Surface Cell with the above solution approximately 10 times. Then, immerse the cell in the solution at approximately 30°C overnight.

9 Wash the cell with methanol to remove the saline reagent which has not reacted, then wash with water. Then, wash the cell with methanol, and dry it in N_2 flow.

10 Prepare 3.5% acrylamide solution containing 1 mg/mL potassium peroxodisulfate and 1 μg/mL N,N,N',N'–Tetramethyl-ethylenediamine.

11 Wash the cell with the above acrylamide solution, and immerse it in the solution for 2 hours.

12 Wash the cell with water, then with 0.1 mol/mL hydrochloric acid to remove excess acrylamide.

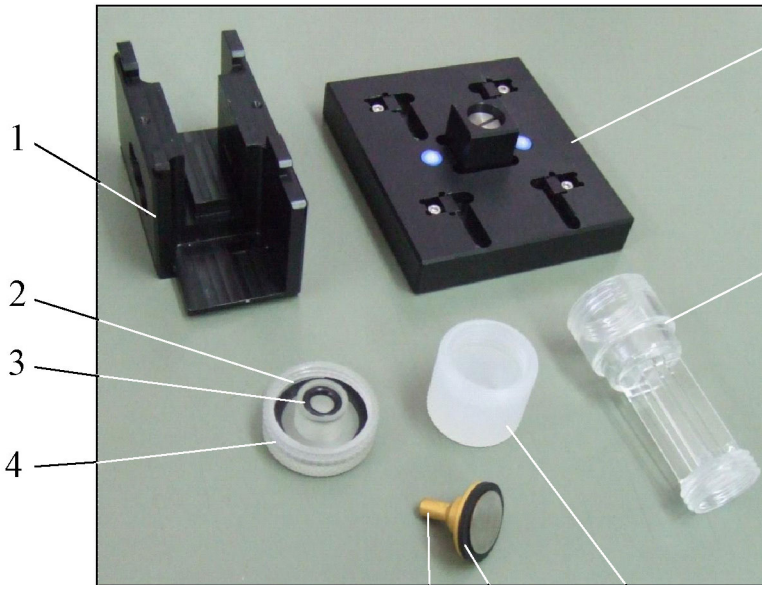
13 Wash the cell with water again, and wrap with lens paper. Store the cell in water to protect from drying.

Disposable Zeta Cell Maintenance

This section describes how to maintain the Disposable Cell for Zeta Potential measurement.

Figure 4.47 Disposable Cell for Zeta Potential

Figure 4.48 Disposable Cell for Zeta Potential Components



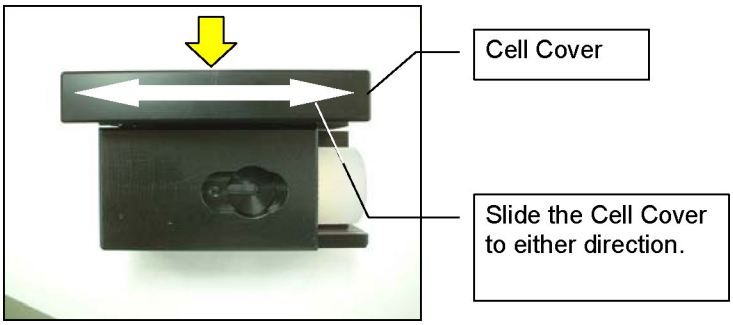
1. Cell Holder
2. Large Packing
3. O-Ring
4. Knob A
5. Electrode (2)
6. Small Packing (2)
7. Knob B
8. Disposable Cell
9. Cell Cover

Disassembling the Disposable Cell

To disassemble the disposable cell:

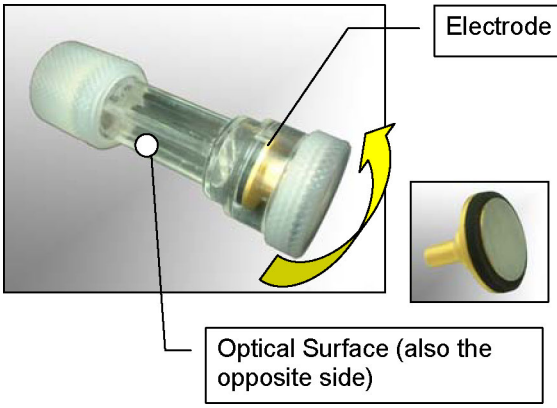
1 Remove the cell cover, sliding and pressing against the cell holder.

Figure 4.49 Removing the Disposable Cell Cover



2 Remove the Disposable Cell from the cell holder. Remove the electrode, turning knob A counter-clockwise.

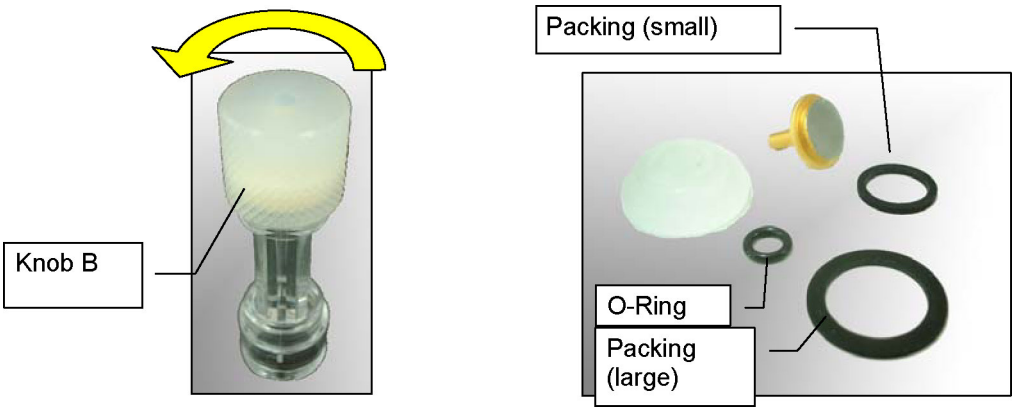
Figure 4.50 Removing the First Electrode from the Disposable Cell



3 Drain the sample solution from the cell.

4 Remove the other electrode, turning knob B counter-clockwise. Remove the small packing from the electrode, then remove the O-ring and large packing from knob A.

Figure 4.51 Removing the Second Electrode from the Disposable Cell

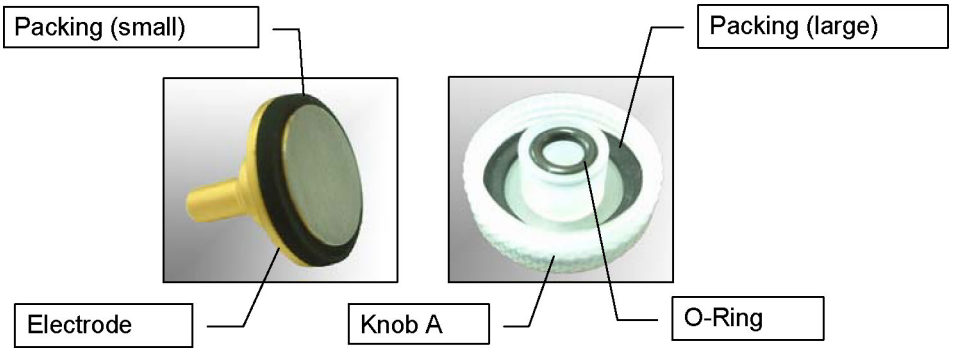


Assembling the Disposable Cell

To assemble the Disposable Cell:

1 Attach the small packing to the electrode, and insert the O-ring and large packing in knob A.

Figure 4.52 Installing the Packing and O-Rings in the Disposable Cell

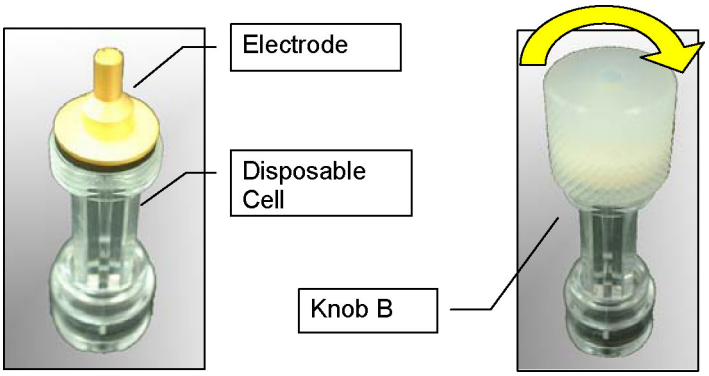


2 Mount the electrode on the disposable cell, and attach knob B, turning it clockwise. Finger-tighten gently.

⚠ CAUTION

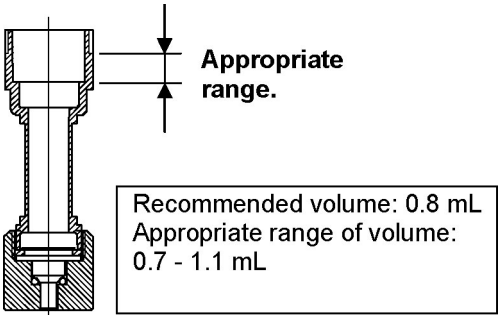
Turn knob B until it knocks the end lightly. Do not over-tighten.

Figure 4.53 Mounting the Electrode on the Disposable Cell



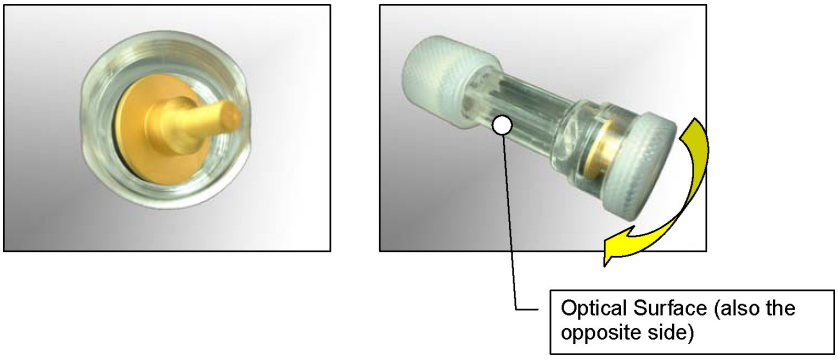
3 In the open end of the cell, inject the sample solution to the recommended volume.

Figure 4.54 Recommended Volume of Sample Solution for Disposable Cell



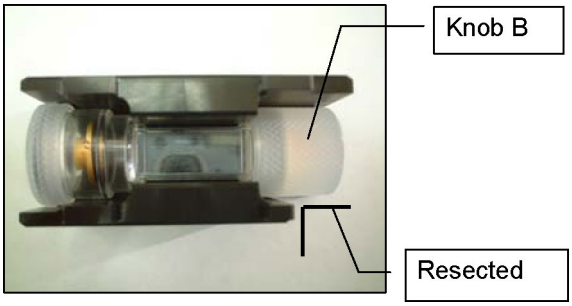
4 Insert the electrode into the cell, tilting the cell slightly to release air bubbles. Turn knob A to the right (clockwise) to secure the electrode.

Figure 4.55 Inserting the Electrode into the Disposable Cell



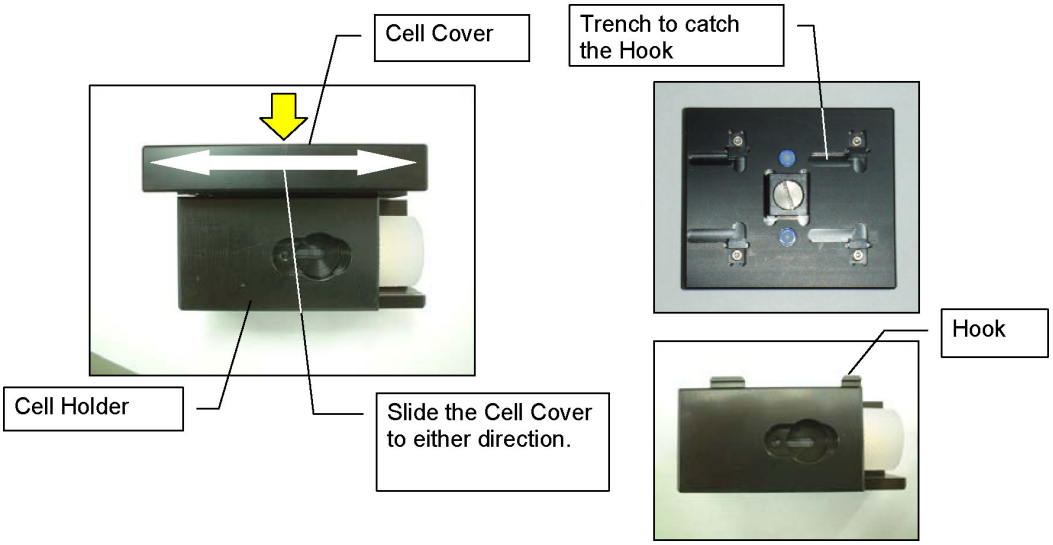
5 Attach the cell to the cell holder, making sure you attach it in the proper direction.

Figure 4.56 Attaching the Disposable Cell to the Cell Holder



6 To attach the cell cover, align the trenches with the hooks in the cell holder, and slide the cover into place until it stops.

Figure 4.57 Attaching the Disposable Cell Cover



Washing the Disposable Cell

When using a new sample, use a new Disposable Cell. The Disposable Cell should be used one time only. All other components of the Disposable Cell can be washed and reused.

⚠ CAUTION

Do not use packings from other cells.

To wash the Disposable Cell:

1 Wash the Disposable Cell with purified water.

2 To remove heavy dirt, immerse the components in a detergent, treat with ultrasound, and rinse thoroughly with purified water to remove all detergent.

⚠ CAUTION

Do not touch the optical surface of the Disposable Cell.

⚠ CAUTION

The cell holder is equipped with glass plates on both sides. Dry them thoroughly after washing. Dirt adhering to the glass plates may interfere with measurements.

Low Conductivity Cell Maintenance

This section describes how to maintain the Low Conductivity Cell for Zeta Potential measurement.

Figure 4.58 Low Conductivity Cell for Zeta Potential

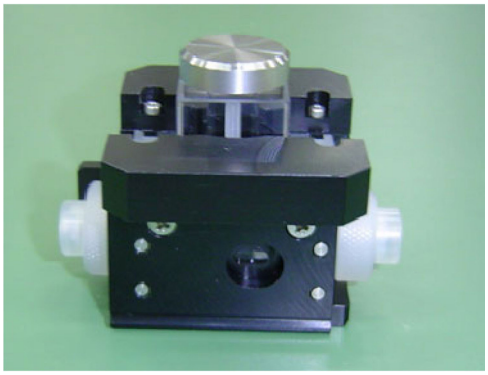
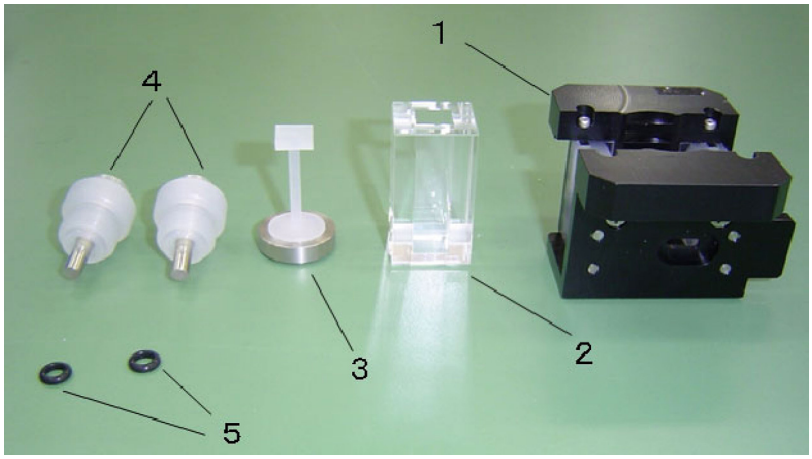


Figure 4.59 Low Conductivity Cell for Zeta Potential Components



1. Cell Holder
2. Glass Cell
3. Cell Cap
4. Electrode Block (2)
5. O-Rings (P4 perfluro)

Assembling the Low Conductivity Cell

To assemble the Low Conductivity cell:

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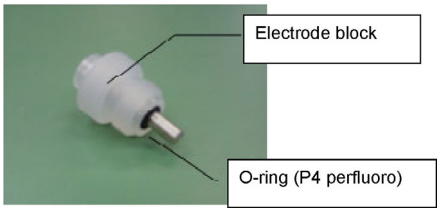
1 Attach an O-ring to each electrode block.

Figure 4.60 Installing the O-Rings in the Low Conductivity Cell



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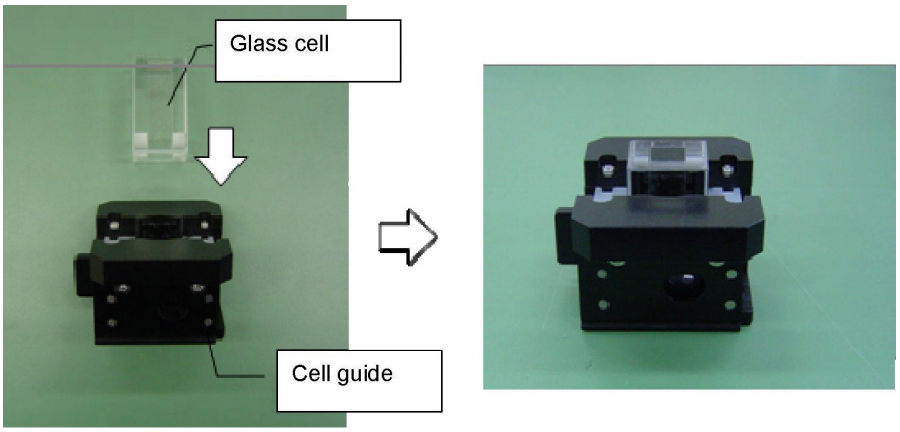
2 Insert the glass cell into the cell holder. Orient and align the surface of the glass cell so that the opening is flat against the surface of the cell block.

Figure 4.61 Inserting the Glass Cell into the Low Conductivity Cell

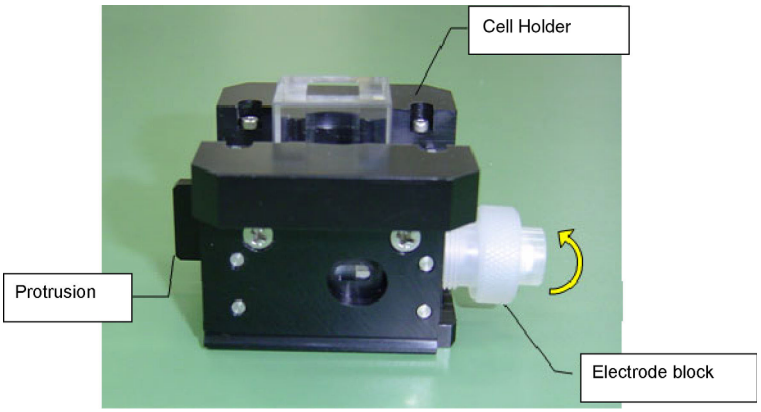


⚠ CAUTION

Do not touch the optical surface of the disposable cell.

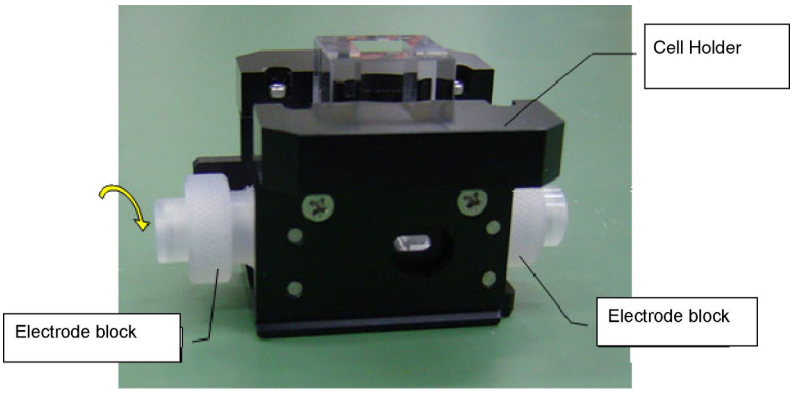
3 Insert the electrode block from the side opposite the protrusion in the cell holder, and screw in until fully tightened.

Figure 4.62 Inserting the First Electrode Block



4 Tighten the other electrode block on the other side. Both electrode blocks are identical. There is no left versus right orientation.

Figure 4.63 Inserting the Second Electrode Block

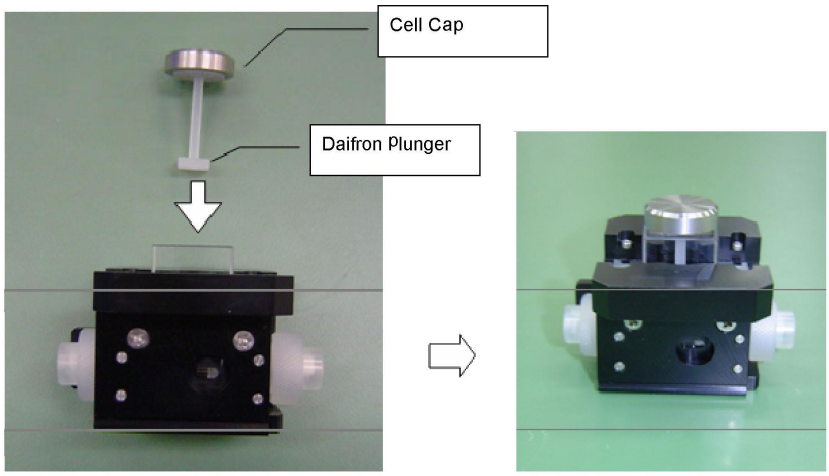


5 Put the sample into the glass cell using a pipette. The recommended usable volume range is 1.5 mL to 2.5 mL.

Figure 4.64 Pouring Sample into the Glass Cell

6 Remove any bubbles and/or foam, and insert the cell cap. Ensure that there are no bubbles or foam below the Daifron plunger.

Figure 4.65 Replacing the Cell Cap



NOTE To assemble the Low Conductivity Cell, follow the steps above in the reverse order.

Cleaning the Low Conductivity Cell

To prevent damaging the glass cell during handling, avoid using ultrasonic cleaning. Follow the instructions described below.

To clean the Low Conductivity Cell:

1 Rinse the glass cell thoroughly with the same solvent that was used for the dispersant of the sample (diluent), or with an alcohol (such as ethanol or methanol). Dry thoroughly after rinsing with the solvent.

2 If the inside of the glass cell is severely stained, moisten a cotton swab with ethanol or methanol, and wipe out the soil from the inside of the cell directly. After wiping, rinse thoroughly with purified water, and verify that there are no fibers from the cotton swab remaining in the glass cell.

⚠ CAUTION

If the glass cell is severely stained, rinse in purified water after soaking for several hours in concentrated sulfuric acid or concentrated hydrochloric acid. Handling concentrated sulfuric acid or concentrated hydrochloric acid is dangerous. Exercise caution.

3 Wipe up water droplets around the glass cell using lens paper.

4 Dry the glass cell in N_2 flow or using a dryer. When you use N_2 flow, it should be passed through a gas filter to reduce spots after drying. When an organic solvent is used for washing, rinse with acetone and dry the cell.

5 When cleaning parts other than the glass cell (the electrodes, O-rings, and so on), soak in a mild detergent and perform ultrasonic cleaning, then rinse thoroughly to ensure that there is no residual detergent.

Low Conductivity Cell Analysis

Data obtained using the Low Conductivity cell can be analyzed by Zeta Potential Analysis. The plot of mobility/zeta potential distribution can be opened by Low Conductivity Cell Analysis. Select Low Conductivity Cell Analysis on the Main menu bar.

To obtain a Low Conductivity Cell data file:

1 Click **Open** from the File menu and select the desired file.

2 The saved mobility/zeta potential distribution data will be displayed. Select a file to be analyzed. If you select Manual Save at measurement, files will not be displayed until you save the results of the measurement manually.

3 Click **Print** to print the selected data.

4 Click **Print Preview** to print the data file. A Print Preview window opens to show the print results prior to printing.

5 Click **Close** to close the file.

System Software Configuration Maintenance

System parameters, cell listing, system registration information, and data mirroring are included in the system configuration.

NOTE You must be an Administrator or Supervisor to change system parameters, change system registration information, and enable data mirroring.

System Parameters

⚠ CAUTION

The system parameters include parameters that are critical to the operation of the equipment. The information in Instrument Constants and Communication Ports should not be changed. The Options can be changed according to your particular needs and setup.

To view system parameters:

1. Select the Maintenance function icon panel. The System Configuration screen opens. The System Parameters panels open. On the left is the folder view, and on the right is a detailed view.

2. Select System Parameters in the System Configuration panel to open the detailed view.

Figure 4.66 Maintenance System Parameters

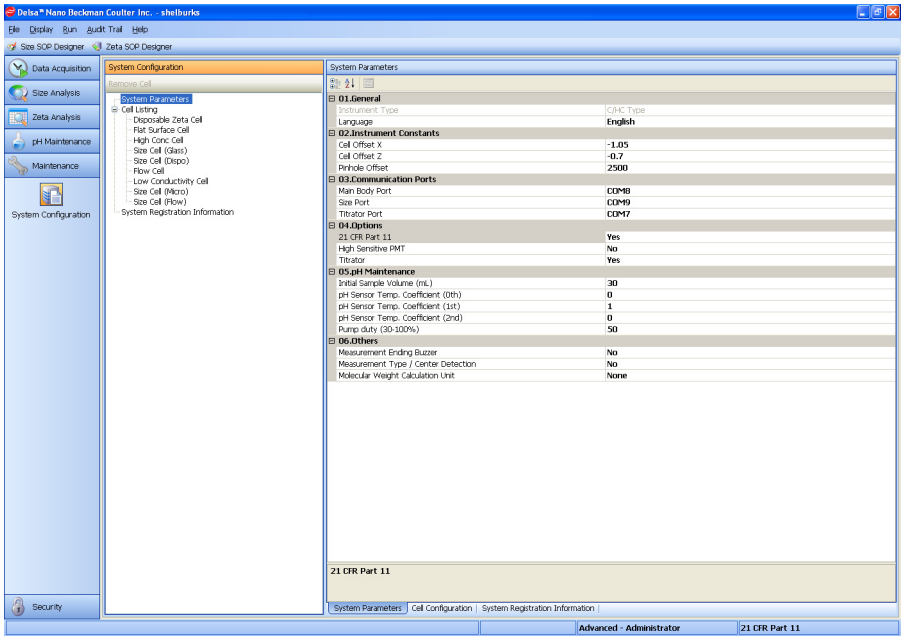


Table 4.2 Maintenance System Parameters

Parameter Set	Parameter	Description
General	Instrument Type	Equipment model.
	Language	The language displayed.
Instrument Constants	Cell Offset X	The offset value for the cell block (X axis). This value is set at the factory.
	Cell Offset Y	The offset value for the cell block (Y axis). This value is set at the factory.
	Pinhole Offset	The offset value for the origin point of the pinhole. This value is set at the factory.
Communication Ports	Main Body Port	The communications (RS-232) port for communications with the equipment DelsaNano interface board.
	Size Port	The communications (RS-232) port for communications with the DelsaNano DSP board.
	Titrator Port	The communications (RS-232) port for communications with the titrator.

Table 4.2 Maintenance System Parameters *(Continued)*

Parameter Set	Parameter	Description
Options	21 CFR Part 11	Shows if 21 CFR Part 11 is enabled.
	High Sensitive PMT	Select Yes if the equipment model is DelsaNano HC. Otherwise, select No.
	Titrator	Select Yes to use the titrator. Select No if you do not want to use the titrator.
	Initial Sample Volume	This refers to the initial sample volume in the sample vial of the Auto Titrator. This prevents the sample from overflowing pH titration.
	pH Sensor Temp. Coefficient (0th, 1st, 2nd)	These are the temperature coefficients of pH sensor. It has 3 Coefficients with default values of (0th - 0, 1st-1, 2nd -0)
	Pump Duty (30-100%)	The percentage of the pumps being used during titration.
	Measurement Ending Buzzer	Allows a buzzer to indicate the end
	Measurement Type/Center Detection	Select Yes to see the Cell Center Detection window Figure 1.26 prior to the measurement.
	Molecular Weight Calculation Unit	Allows you to select the molecular unit (kDa)

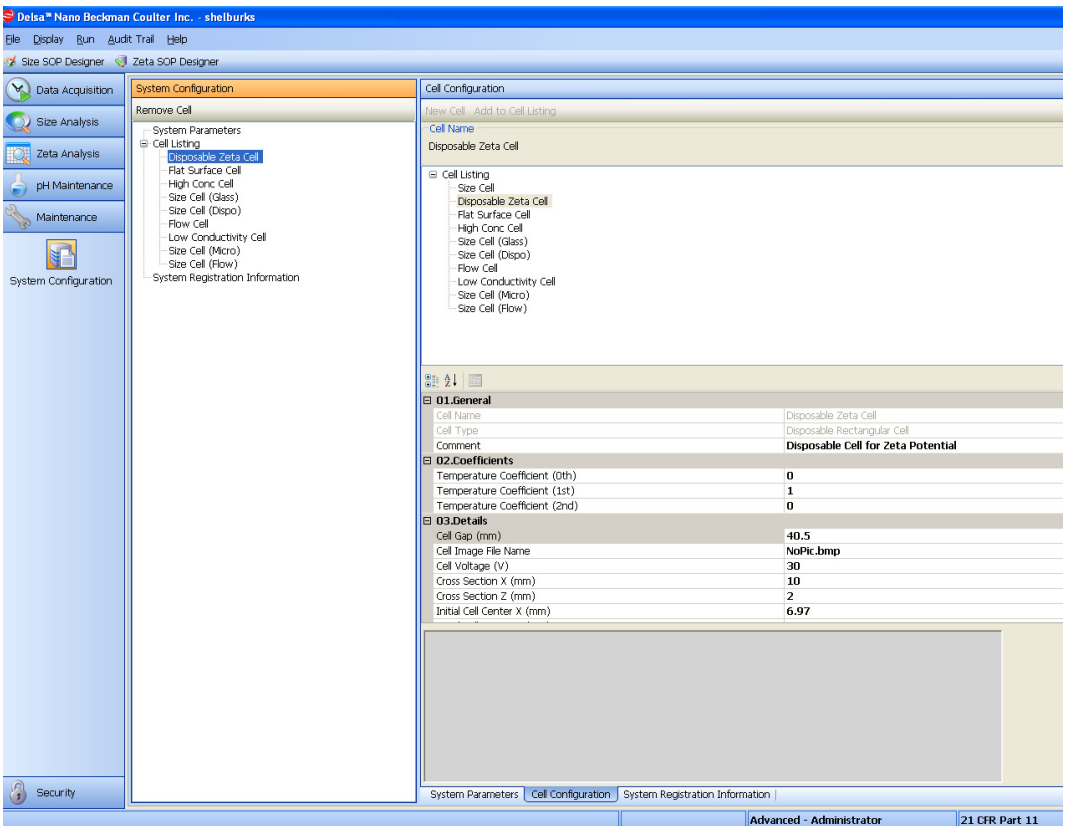
Cell Listing

To view cell listing parameters:

1 Select the Maintenance function icon panel. The System Configuration screen opens.

2 Select Cell Listing in the System Configuration panel to open the detailed view, then select a specific cell to view its details.

Figure 4.67 Maintenance Cell Listing Parameters



System Registration Information

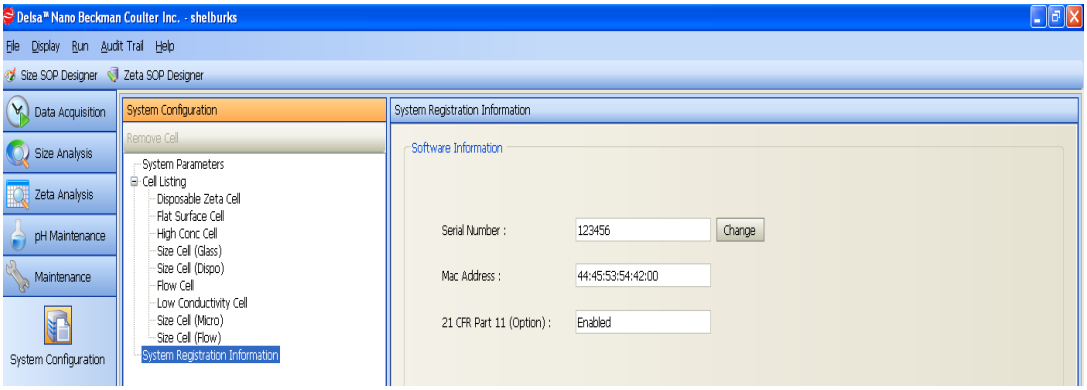
NOTE You must be a Supervisor or an Administrator to change system registration information.

To view system registration information:

1 Select the Maintenance function icon panel. The System Configuration screen opens.

2 Select System Registration Information in the System Configuration panel. A screen showing the registration parameters opens.

Figure 4.68 System Registration Parameters



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APPENDIX A

Auto Titrator

Alerts for Danger, Warning, Caution, Important, and Note

⚠ DANGER

DANGER indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury. This signal word is to be limited to the most extreme situations.

⚠ WARNING

WARNING indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury. May be used to indicate the possibility of erroneous data that could result in an incorrect diagnosis (does not apply to all products).

⚠ CAUTION

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. May be used to indicate the possibility of erroneous data that could result in an incorrect diagnosis (does not apply to all products).

IMPORTANT IMPORTANT is used for comments that add value to the step or procedure being performed. Following the advice in the Important adds benefit to the performance of a piece of equipment or to a process.

NOTE NOTE is used to call attention to notable information that should be followed during installation, use, or servicing of this equipment.

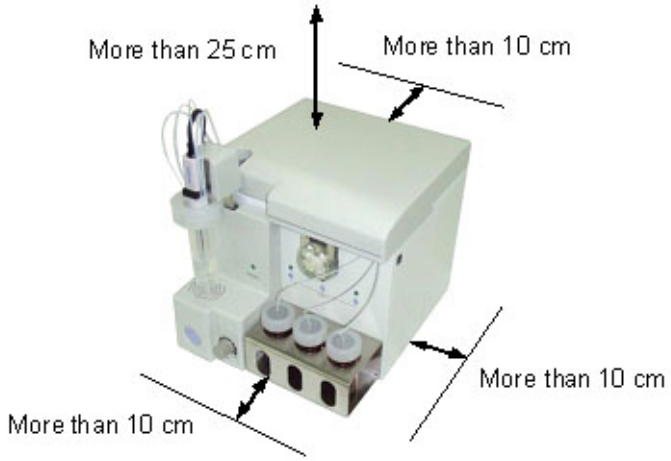
Precautions and Environment Specifications

Read this manual completely before using the DelsaNano Auto Titrator (AT) so that you understand fully how to operate the instrument.

Site requirements:

- Avoid inclination, vibration, and shock during operation and transport. Position the Auto Titrator on a stable, level surface.
- Set up the instrument in a place free from the effects of pressure, extreme temperatures and humidity, poor ventilation, sunlight, dust, or salty or sulfurous air.
  - Surrounding temperature: 15-35° C
  - Relative humidity: 30-85% (without condensation)
- Set up this instrument considering the frequency and voltage of power supply and allowable electric current. Confirm the status of electric discharge and polarity when a battery is to be used as the power supply.
  - Rated voltage: AC 100-230V, 50/60 Hz
  - Rated electricity consumption: 55 VA
- Do not set up the instrument in a strong magnetic or electric field.
- Do not set up the instrument in a place where chemicals are stored or where chemical gases may be generated.
- Do not set up the instrument in a place where it will be splashed with water.
- Do not set up the instrument near the blowout hood of an air conditioner.
- Connect the ground wire correctly.
- Maintain the specified clearances around the instrument shown in Figure A.1.

Figure A.1 Auto Titrator Setup Clearances



Prior to using the Auto Titrator, check the following:

- Confirm that there is no damage on the surface of the instrument and no extraneous substances inside the instrument.
- Confirm that the instrument runs normally, including all switch contacts.
- Confirm that the instrument is grounded properly.
- Confirm that all electric cords are connected properly and securely.

During use, take note of the following:

- Be aware of proper instrument function at all times.
- Do not touch any operational parts other than those specified in the instructions.
- Operate the instrument appropriately; stop the instrument when you observe any abnormal instrument functions.
- Do not use electric devices that use radio waves around the instrument.
- Operate the instrument according to instructions printed on the labels attached to the instrument.

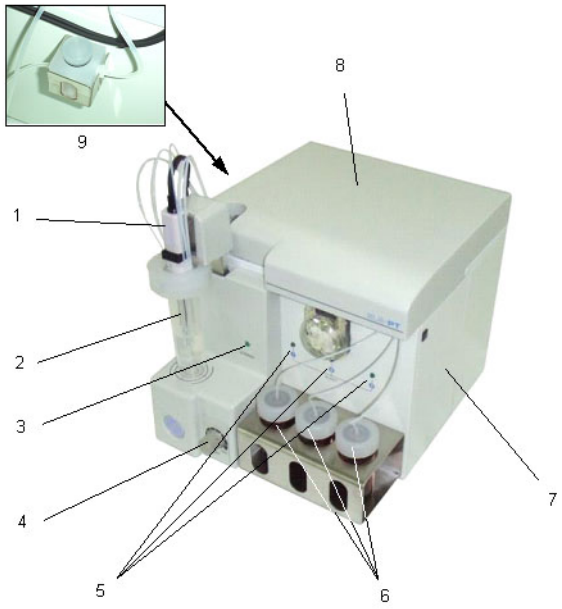
If any problems occur, stop operation immediately and contact your Beckman Coulter representative.

Do not disassemble the instrument.

Auto Titrator and pH Electrode Components

The components of the Auto Titrator are shown below.

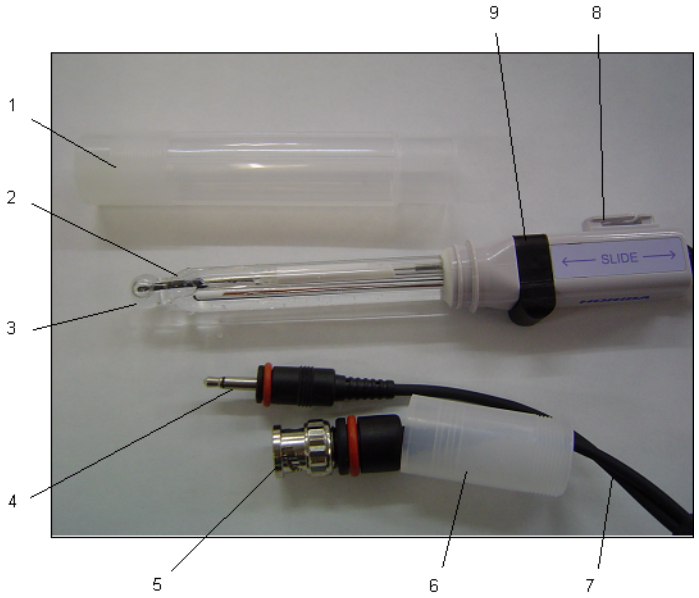
Figure A.2 Auto Titrator Components



1. pH Electrode
2. Vial (plastic)
3. LED for Stirrer
4. Stirrer Switch
5. LED for Titration
6. Vial (glass)
7. Syringe Cover
8. Top Panel
9. Air Bubble Trap

The components of the pH electrode are shown below.

Figure A.3 pH Electrode Components



1. Protective Cap
2. Contact Portion to Solution
3. Responding Glass Membrane
4. Connector for temperature sensor
5. Electrode Connector
6. Connector Protective Cover
7. Lead Wire
8. Cap
9. Rubber Stopper for Refill Opening (Inner Solution)

Stirrer LED Indicators

Use the Power switch to turn the power On/Off. Once the power is On, you can rotate the switch to the right (clockwise) to adjust the speed of the Stirrer from minimum (MIN) to maximum (MAX). Minimum power is the Off position.

Table A.1 Stirrer LED Status Indicators

LED Type	Status of Instrument
STIRRER	Green: Stirrer On Off: Stirrer Off

Titration LED Indicators

Table A.2 Titration LED Status Indicators

LED Type	Status of Instrument
(1), (2), (3)	Green: Instrument startup is complete. Blinking in Green: During instrument startup, or during dispensing sample solutions. Off: Power Off, or no vial is selected.

⚠ CAUTION

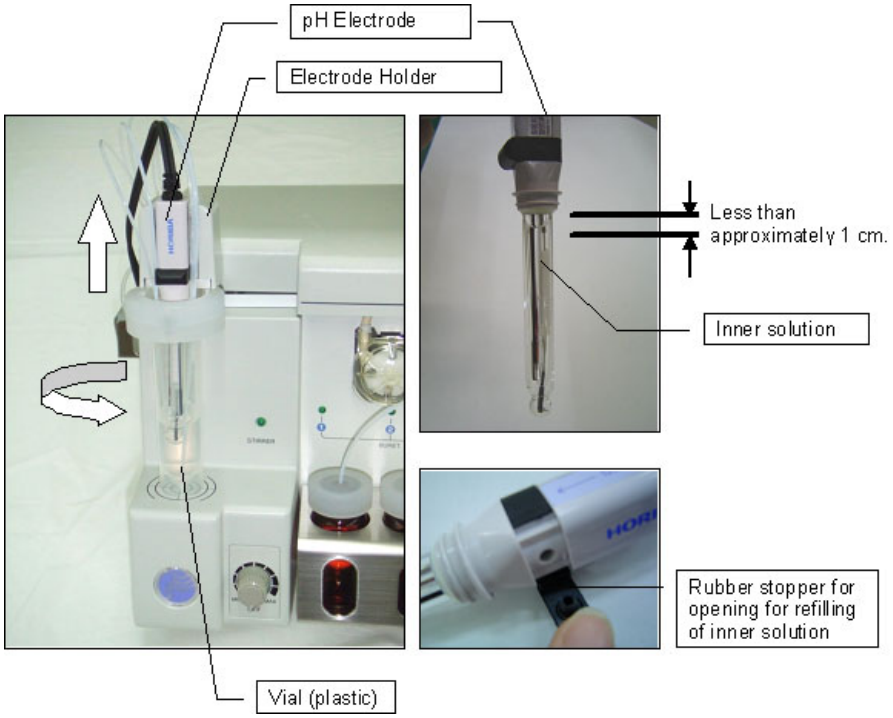
Each LED corresponds to the vial just under the LED. Be careful not to switch the tubing.

Preparing for Measurement

Inspecting the pH Electrode

Remove the pH electrode by sliding it upward and rotating the vial (plastic) to the right (looking down). Confirm the volume of inner solution in the pH electrode. If the volume has decreased, refill the inner solution (3.33 mol/L KCl solution) by removing the rubber stopper from the refill opening (for refilling the inner solution).

Figure A.4 Inspecting the pH Electrode



⚠ CAUTION

The inner solution for the electrode is KCl solution of high concentration (3.33 mol/L). If your hands/skin contact the inner solution, wash your hands/skin under running water immediately. If the inner solution enters your eye(s), wash with running water immediately, and seek medical attention.
The outer tube and tip of the electrode are made of glass. Be careful not to break the glass.

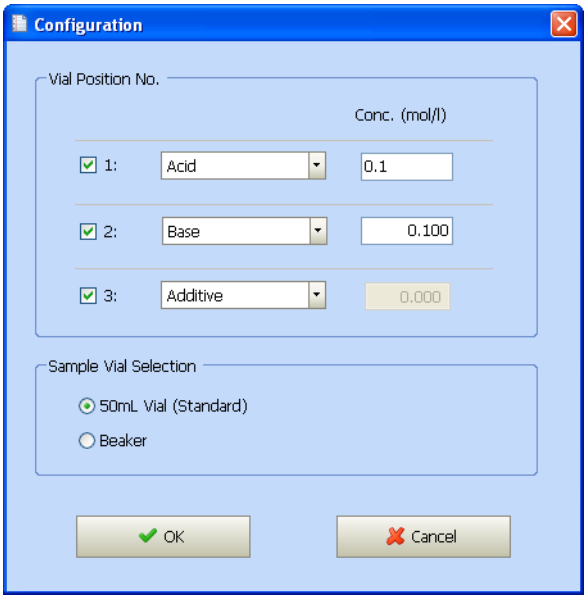
Setting the pH Configuration

You can set acid/base/other solutions such as additive for the titration.

To set the pH Configuration:

1 Select **Configuration** in the pH Maintenance window.

Figure A.5 pH Configuration Dialog



2 Select the vial position number to which acid, base, or other solution is to be set and select the type of solution. Then enter the molar concentration for each solution.
If a vial is not going to be used, deselect the vial position number.

3 Select the type of vial to be used: 50 mL or Beaker.

4 When finished, click **OK** to accept your changes and close the dialog.

Calibrating the pH Electrode

Calibrate the pH electrode before starting measurement. Calibration is not required for each measurement, but it should be done once a day, for example, before the first titration.

Priming

When the instrument is used for the first time, or when the titration solution is exchanged for another solution, fill the solution according to the procedures described below.

1 Pour the titration solution into the glass vial or plastic vial and put the cap on. Use the glass vial if the titration solution will be used continuously for more than one day. You can use plastic vials if using the titration solution for a shorter time.

Figure A.6 Glass Vial

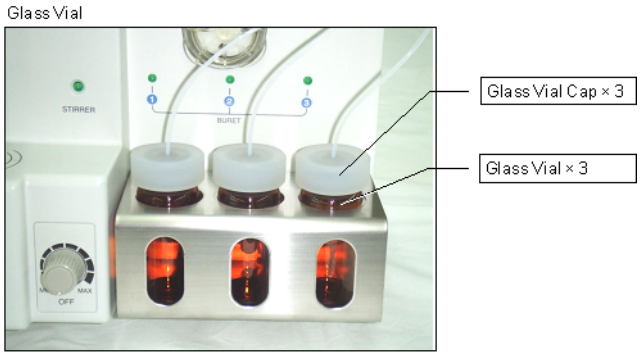
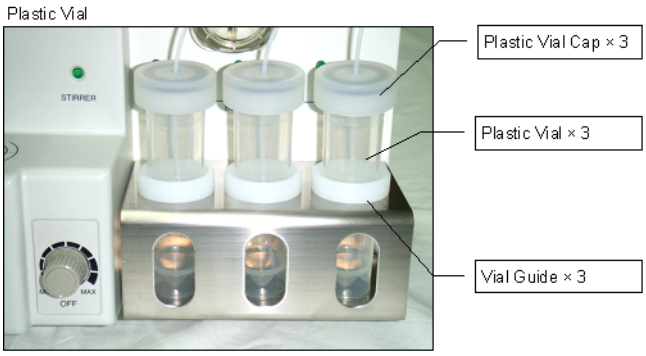


Figure A.7 Plastic Vial



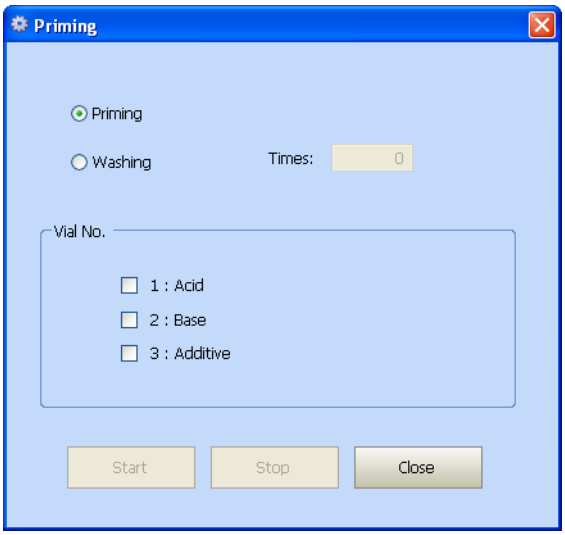
⚠ CAUTION

Be careful not to swap the Teflon tube when pouring the titration solution. If tubes are mistakenly inserted into the wrong vial, exact titration cannot be done.

2 Set a beaker for waste fluid underneath the sample vial setting portion.

3 Select Priming in the pH Maintenance window. The Priming Dialog opens.

Figure A.8 Priming Dialog



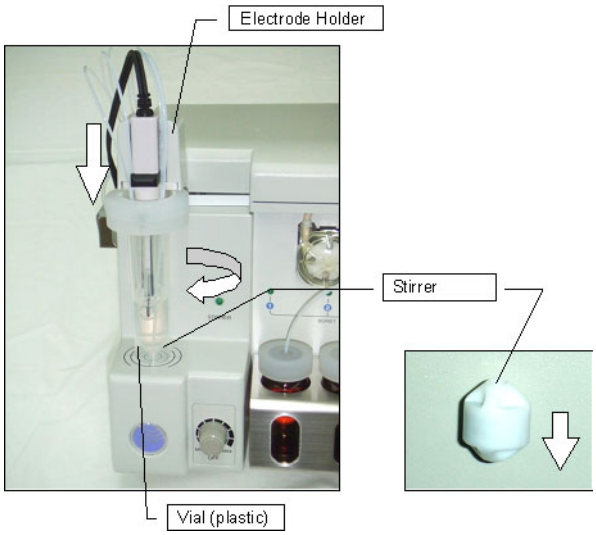
4 Select the vial number, and click **Start**. The solution will start filling. Click **Stop** after confirmation that no air remains in the tube. After completion of filling, click **Close** to return to the previous window.

Pouring the Sample Solution

To pour the sample solution:

Set the stirrer into the vial and pour in the sample solution. Set the vial, and slide the electrode holder down until it reaches the bottom.

Figure A.9 Pouring the Sample

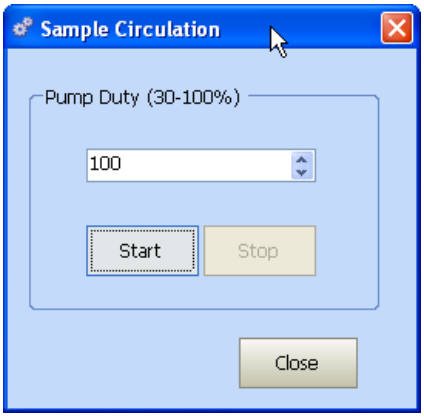


Circulating the Sample

To circulate the sample:

1 Select Circulation of Sample from the Sample Circulation of pH Maintenance window. The Sample Circulation dialog opens.

Figure A.10 Sample Circulation Dialog



2 Set Duty (pump speed) for circulation. You can select a Duty range of 30-100%. The larger the value, the faster the circulation speed. It is recommended that you select 50 when filling the sample solution.

3 Click **Start** to start circulation.

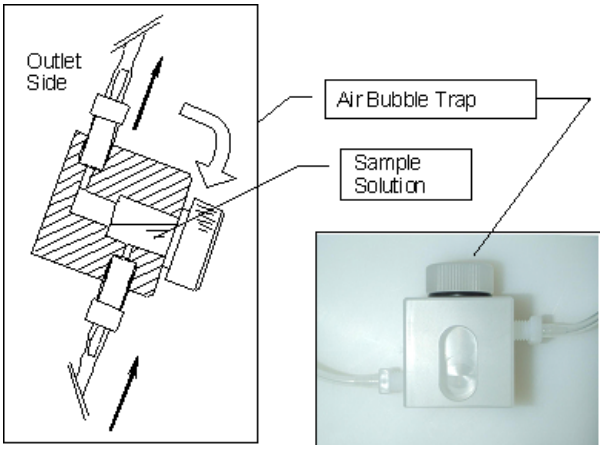
4 Click **Close** to close the dialog.

Degassing the Tube

To degass the tube:

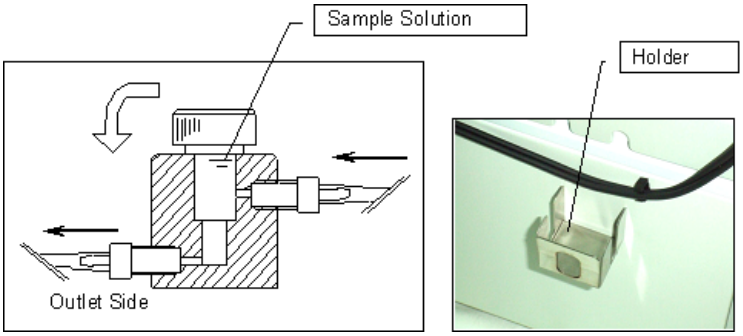
1 Tilt the air bubble trap, holding the outlet side upright while the sample solution is circulating.

Figure A.11 Degassing the Tube, Step 1



2 When you see no bubbles in the outlet, return the air bubble trap to a horizontal, level position. Then put the trap on the holder.

Figure A.12 Degassing the Tube, Step 2



⚠ CAUTION

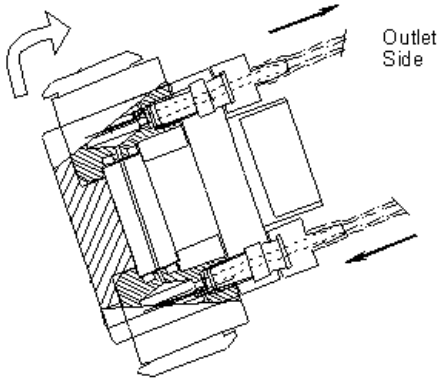
Be sure that degassing is complete. Air bubbles in the tube might enter the cell of the Titrator and cause inaccurate measurements.

Degassing the Glass Cell

To degass the glass cell:

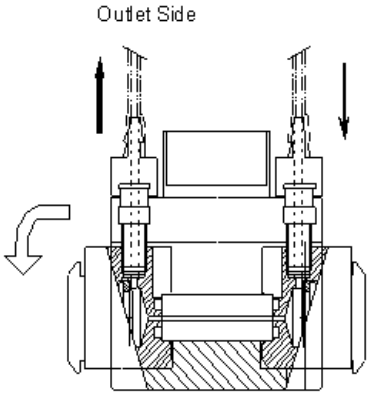
1 Tilt the cell holding the outlet side upright while the sample solution is circulating.

Figure A.13 Degassing the Glass Cell, Step 1



2 When you see no bubbles in the outlet, return the cell to a horizontal, level position.

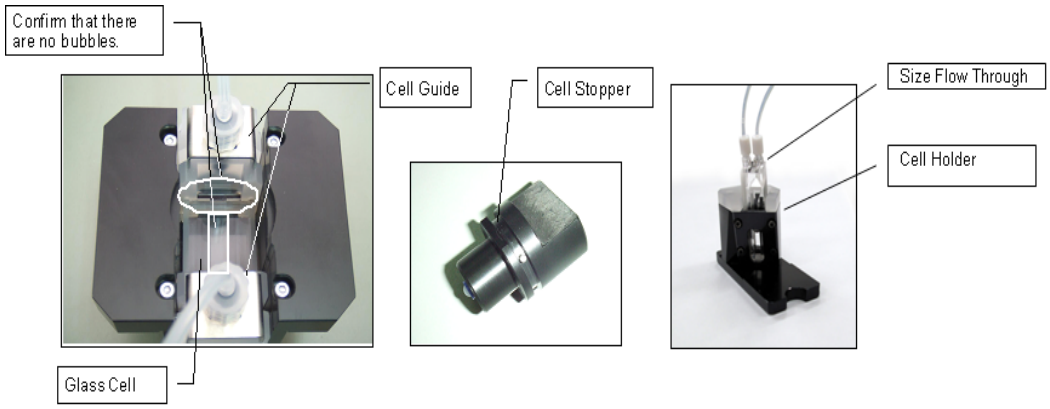
Figure A.14 Degassing the Glass Cell, Step 2



3 Remove the cell stopper and confirm that there are no bubbles inside the glass cell and between the cell holder and glass cell.

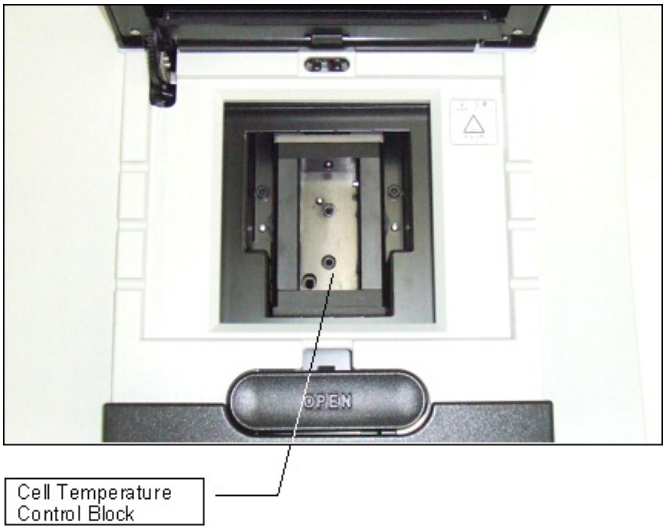
If bubbles cannot be removed, shake the cell gently or tap the side or bottom of the cell with your palm to remove bubbles.

Figure A.15 Checking for Bubbles inside the Glass Cell



4 Insert the cell into the cell temperature control block.

Figure A.16 Cell Temperature Control Block



Measurement

When measurement preparations are complete, start the measurement. First you must confirm the parameters used for the measurement.

Setting Measurement Conditions

Confirm the following prior to starting a measurement: Selection of Cell, Scattering Light Intensity, Diluent Properties, and Measurement Routine.

Figure A.17 Intensity Monitor

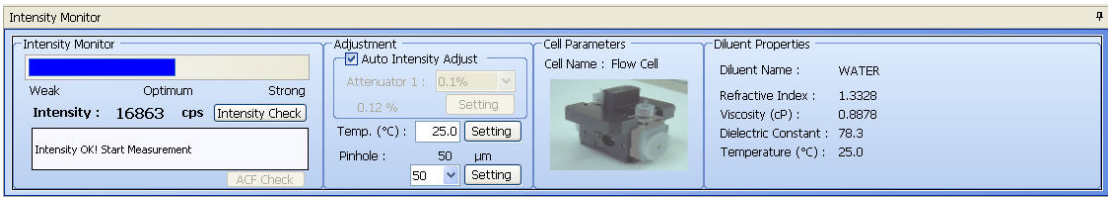


Figure A.18 Intensity Measurement Parameters

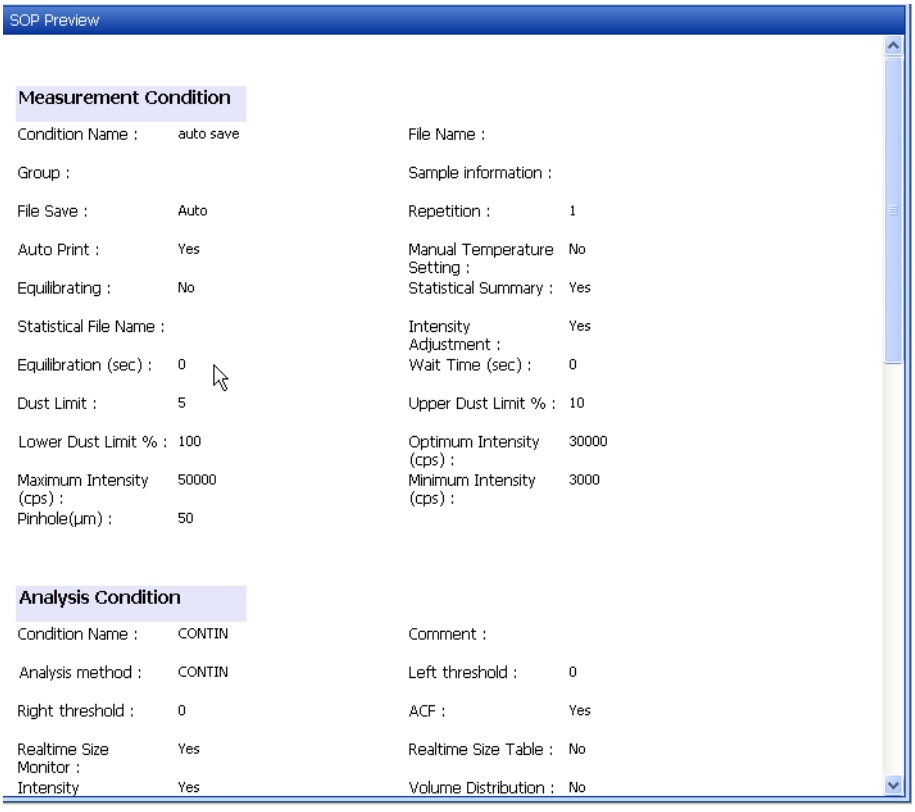


Table A.3 Intensity Measurement Parameters

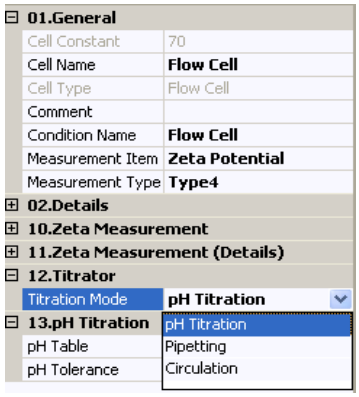
Selection	Description
Selection of Cell	Ensure that the Flow cell/Size cell (Flow) are displaying.
Scattering Light Intensity	The appropriate light intensity is 3000 – 30000 cps. Confirm that the light intensity is within this range. If the light intensity is over or under this range, check that the "Adjustment of ND Filter" is set to "Automatic Adjustment." If the light intensity is out of the appropriate range, even though it is set to "Automatic Adjustment," then adjust the concentration of the sample solution.
Diluent Properties	Select the solvent that is used for sample solution.
Measurement Routine	When you use pH titration, confirm that the appropriate routine has been selected. There is a special routine for pH titration. For details of the routine, see *Preparing for pH Titration*.

Preparing for pH Titration

The following modes are available for pH Titration:

- **pH Titration** — A mode to adjust pH as prescribed for measurement of Zeta Potential/Size.
- **Pipetting** — A mode to add additive(s) as prescribed for measurement of Zeta Potential/Size.
- **Circulation** — A mode to measure Zeta Potential/Size, circulating sample solution for the prescribed period.

Figure A.19 Titrator Modes



This section describes how to prepare the Titration mode.

Preparing the Titration Mode

To prepare the Titration mode:

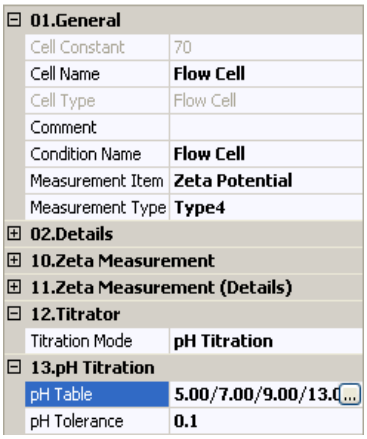
1 Select the cell parameters you want to use.

NOTE If the cell is used in an SOP that is registered or being using in a measurement, you must delete the cell parameter from that SOP in order to edit the cell parameters.

2 Under the Titrator section of the cell parameters, select **pH Titration**.

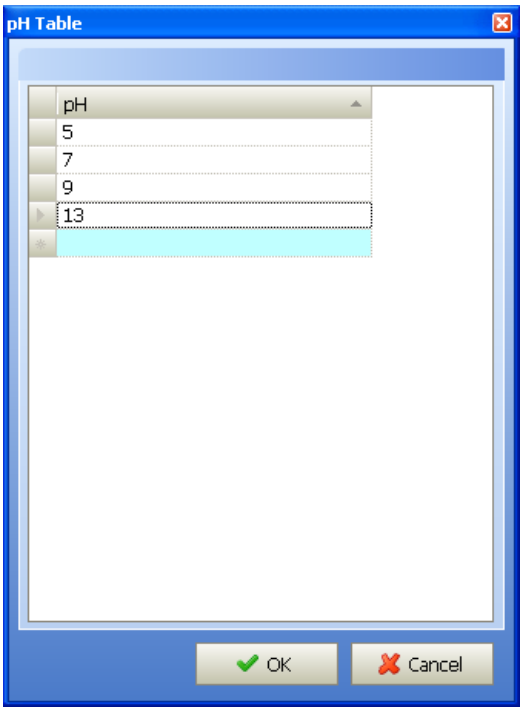
3 Under the pH Titration section of the cell parameters, click **Browse** next to pH Table.

Figure A.20 pH Table Selection



The pH Table dialog opens.

Figure A.21 pH Table Dialog



4 Input the pH values to be attained in the pH table.

5 Click **OK** to accept the values and close the dialog.

6 Adjusting the pH value is considered complete when the pH value falls within the prescribed pH allowance value. The smaller the allowance value, the more exactly the adjustment can be done. The default value should be “0.1” if no other value is specifically required. This is because when the value is smaller than the default, it takes time to adjust the system manually.

7 Set the appropriate measurement type. The measurement types available for use with the Auto Titrator are types 3 and 4.

8 Once you’ve completed your modifications, insert the modified cell parameter into a Size/Zeta SOP to be used in a measurement.

Starting the Measurement

When you’ve completed setting the conditions (such as preparation/selection of routine), click **Start** to begin the measurement.

pH Analysis

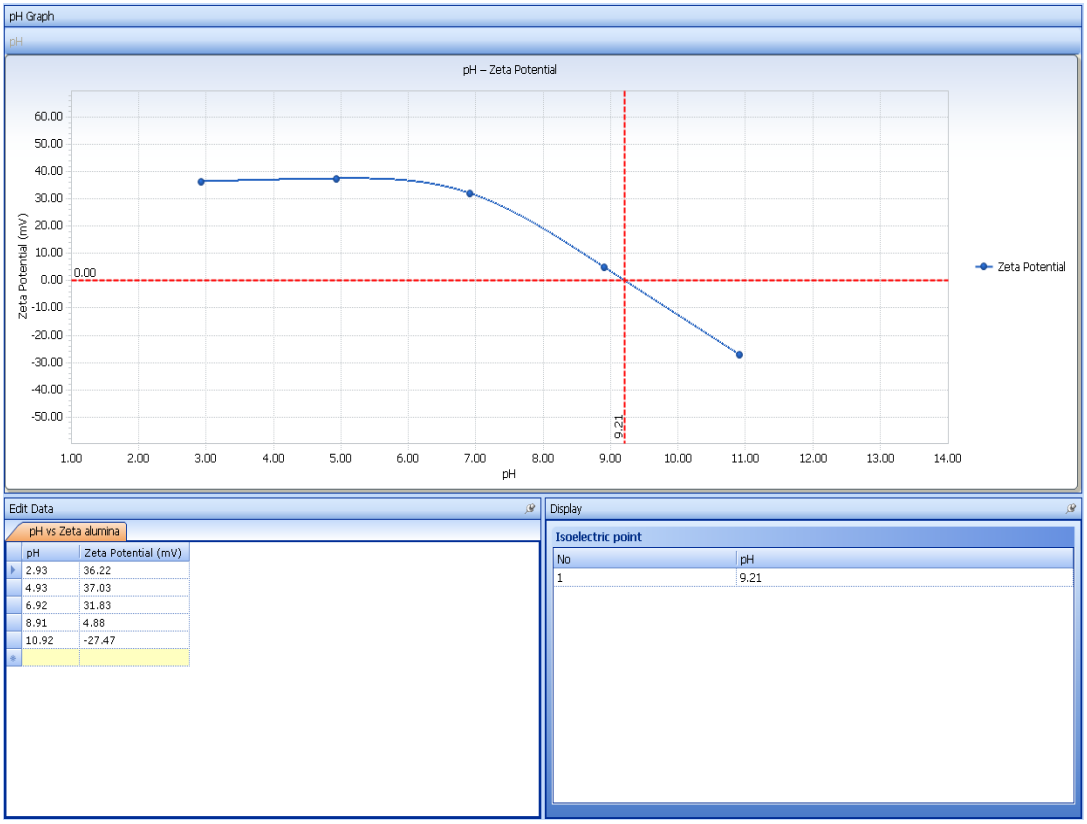
Data obtained using the Auto Titrator can be analyzed by Zeta Potential Analysis. The plot of isoelectric points can be opened by pH Analysis. Select **pH Analysis** on the Main menu bar.

To open a titration file:

1 Select **Open** from the File menu and select the desired file.

2 The saved pH titration data (isoelectric points) will be displayed. Select a file to be analyzed. If you select **Manual Save** at measurement, files will not be displayed until you save the results of the measurement manually.

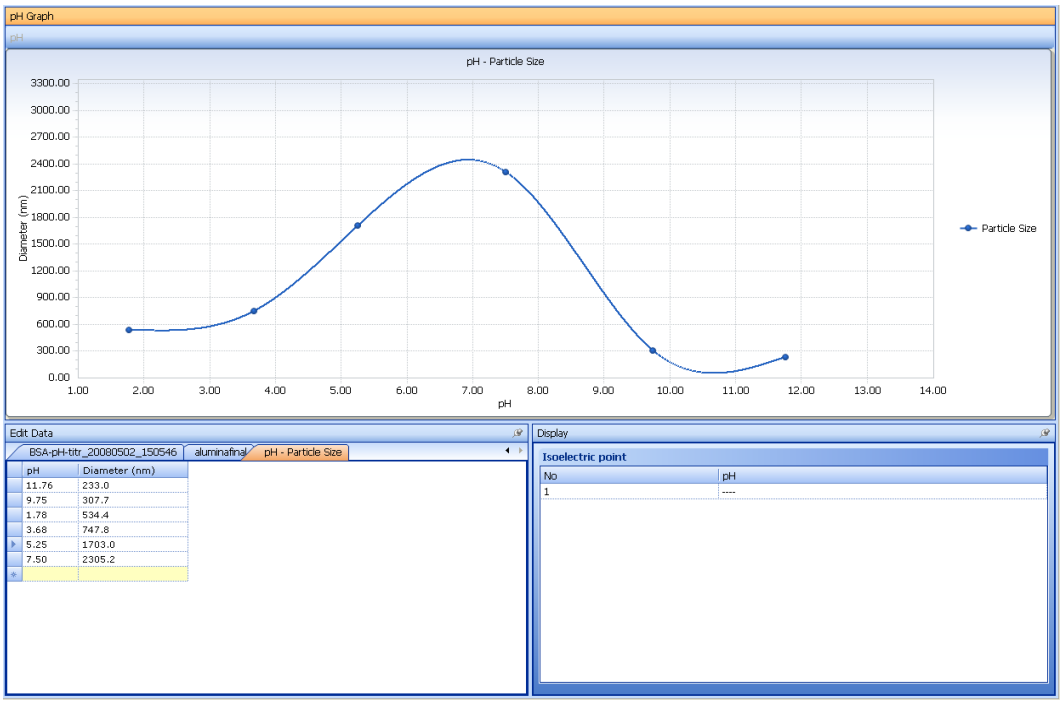
Figure A.22 pH Graph (Zeta Potential)



The data you selected will be displayed and the isoelectric points will be calculated. In addition, this window calculates and displays the plot of isoelectric points when you input Zeta Potential values, using pH values displayed in the window.

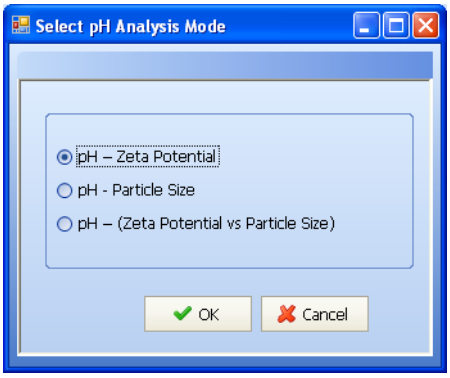
3 Similarly, you can perform analysis pH vs. Size titration and pH vs. Size and Zeta Potential.

Figure A.23 pH Graph (pH vs. Particle Size)



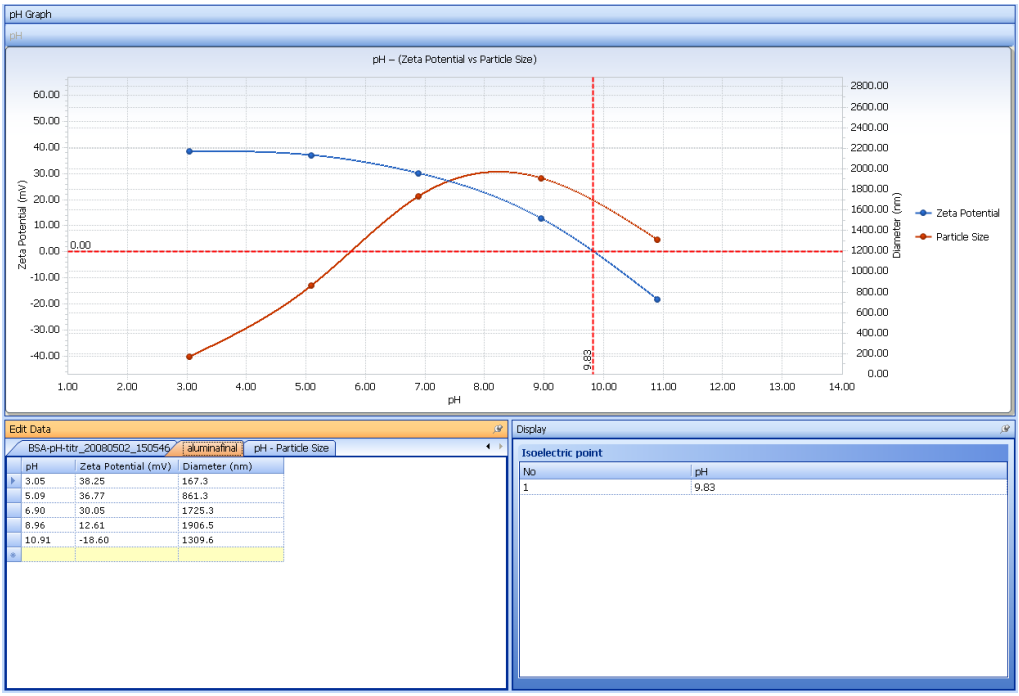
4 To create a pH graph showing Size vs. Zeta Potential, select **New** in the pH Analysis tab, then select pH - Zeta Potential vs. Particle Size graph. Enter the desired values.

Figure A.24 pH Analysis Mode Selection Dialog



5 Select the desired mode, and click **OK**. A window for data input displays.

Figure A.25 pH Graph (Zeta Potential vs. Particle Size)



Calibration of pH Electrode

Calibrating the pH Electrode

To calibrate the pH electrode:

1 pH Standard Solution

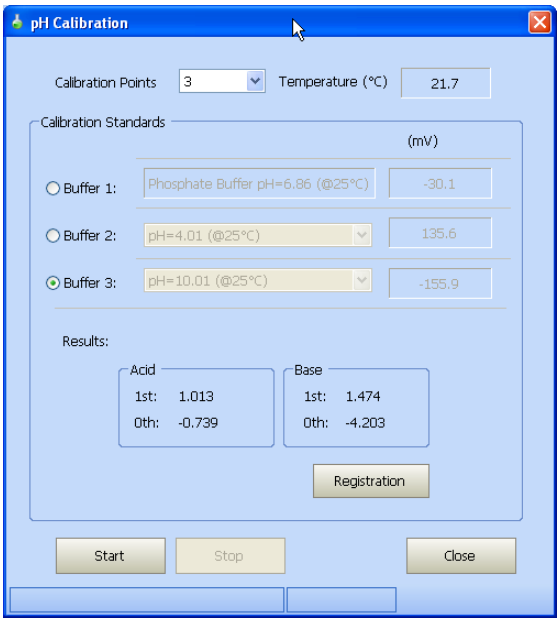
You need commercial pH standard solutions to calibrate the pH electrode. Two or three kinds of pH standard solutions are used for pH calibration (2-point or 3-point calibration). In either case, standard solution of pH 6.86 or pH 7.00 (phosphate) is necessary for both 2-point and 3-point calibration.

2 pH Calibration

Select the pH Calibration icon in the pH Maintenance function panel. The pH Calibration dialog opens.

Select the Number of Points (2 or 3 points) for pH calibration using the drop-down menu.

Figure A.26 pH Calibration Dialog



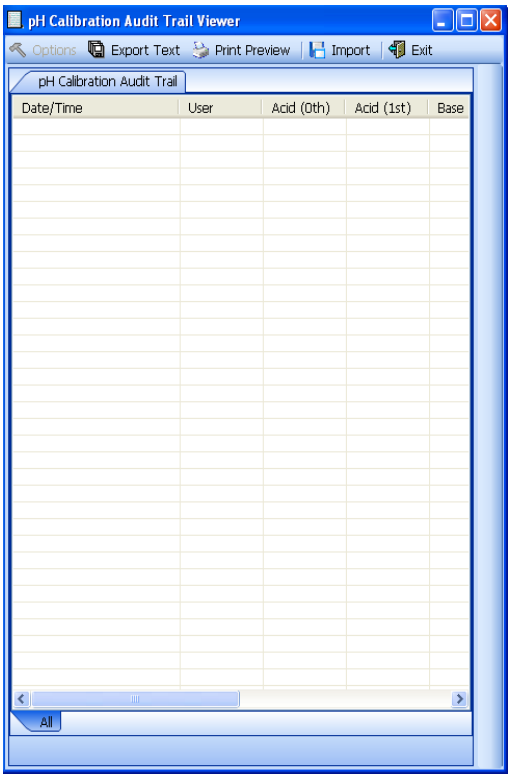
First, set the standard solution of pH6.86 (phosphate) to the sample vial and select Buffer 1. Then, click **Start**.

A progress bar at the bottom of the screen displays during standard solution measurement. Measurement ends automatically when the potential becomes stable.

Next, pH measurements should be done with pH4 standard (phthalate) and pH9 standard (borate). After the end of measurements with the three standard solutions, the results display on the lower part of the pH Calibration dialog.

Click **Registration** to save the results of calibration in the pH Calibration Audit Trail. Otherwise, the results of calibration will not be saved.

Figure A.27 pH Calibration Log Viewer



NOTE When a value out of the specified range for the following items is found in the pH Calibration Audit Trail, this means that an abnormality may have occurred in the electrode. Wash the electrode and exchange the inner solution, then perform the pH calibration again.

- Asymmetry Potential: Specified range of potential: ±30 mV at pH 7.
- Sensitivity: Difference between actual and calculated values for electromotive force between pH 7 and pH 4: Specified range: more than 90%.

Temperature Constants of pH Electrode

When a new electrode is used, the temperature constants of the original pH electrode become invalid, though they were set at shipment. In this case, reset the titrator temperature constants in the System Maintenance Input for Instrument window as follows: the "0th" temperature constant = "0," "1st" = "1," and "2nd" = "0."

Figure A.28 Temperature Constants of pH Electrode

05.pH Maintenance	
Initial Sample Volume (mL)	30
pH Sensor Temp. Coefficient (1st)	0
pH Sensor Temp. Coefficient (2nd)	1
pH Sensor Temp. Coefficient (3rd)	0
Pump duty (30-100%)	50

Maintenance and Inspection

Cleaning the Instrument

If the outside of the instrument or inside of the cover is dirty, wipe the dirt off with a soft cloth dampened with water or neutral detergent.

⚠ CAUTION

The outside of the instrument is coated with a synthetic resin. Wipe off any solution spills immediately.

Replacing the Fuses

The instrument is equipped with two fuses in the power socket on the rear panel.

To change the fuses:

1. Turn the power off and unplug the power cord.
2. Using a small, flathead screwdriver, pry open the fuse holder cover from the AC power input module.
3. Carefully remove the fuse holder from the AC power input module.
4. Using your hands, gently remove the blown fuses and replace with two properly rated fuses (per the fuse rating table below).

Figure A.29 Replacing the Auto Titrator Fuses

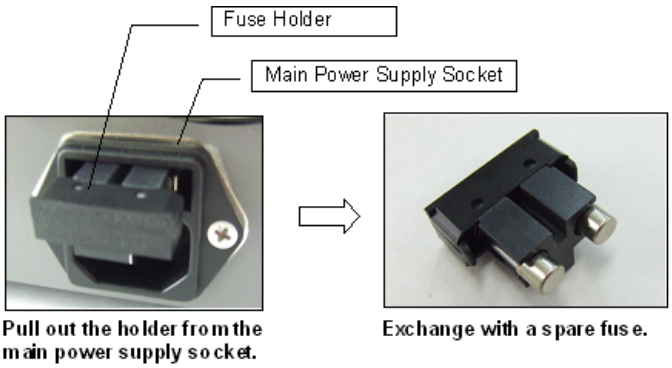


Table A.4 Fuse Rating Table

Type	Current	Voltage
100-120V	T4A	125 V
220-240	T2A	250 V

Consumable Supplies

Consumable supplies are listed in Table A.5. Please contact your Beckman Coulter representative to reorder consumables according to the exchange limit and life span information in Table A.5.

Table A.5 Consumable Supplies

Item	Approximate Exchange Limit or Life Span
Tube (1), (2)	Every exchange of titration solution
Tube (3), (5)–(9)	Every exchange of sample solution
Tube (4)	1 Month
Syringe Packing	6 Months

pH Electrode Maintenance

Precautions for Daily Use

- Inner solution for the reference electrode must be 3.33 mol/L KCL solution.
- Immerse the electrode in purified water for 12 hours or more if the glass membrane of the electrode has dried out.
- Do not touch the electrode connector or allow fluids to contact it.
- When the inner solution does not come down to the inside of the responding glass membrane, shake down the electrode two or three times, holding the cap part of the electrode.

Precautions for First-Time Use or After Long-Term Storage

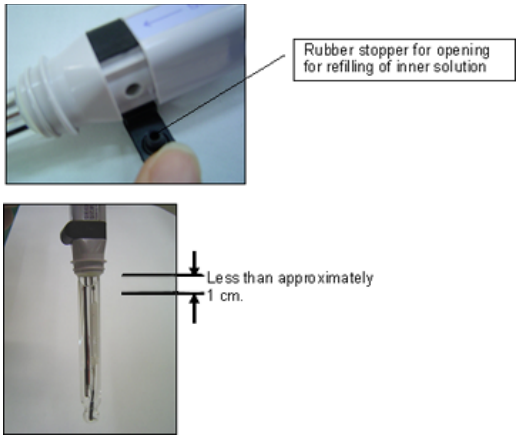
For information on storing the pH electrode, see *pH Electrode Storage Conditions*.

1 Remove the protective cap.

2 Remove the rubber stopper for opening for draining off the inner solution with a Pasteur pipette.

3 Refill the inner solution.

Figure A.30 Refilling the pH Electrode



4 Wash the tip of the electrode with purified water, and wipe with a soft cloth.

NOTE If the inside of the protective cap dries out, wash the protective cap and then refill it with distilled water until the sponge is immersed.

NOTE There may be white crystals of KCL attached to the protective cap and around the refill opening. This is not harmful to performance. Wash the crystals off with purified water and use the electrode.

pH Electrode Storage Conditions

To store the pH electrode for longer than two weeks:

1. Wash the sample solution with purified water.
2. Put the rubber stopper on tightly.
3. Remove the plastic vial.
4. Replace the protective cap.

   NOTE If the inside of the protective cap dries out, wash the protective cap and then refill it with distilled water until the sponge is immersed.

pH Electrode Daily Maintenance

When the electrode has been used for a long time, the sample solution may contaminate the inside of the reference electrode or its inner solution may become diluted. In this case, follow the steps in *Precautions for First-Time Use or After Long-Term Storage*.

Improving Response Time

If the response time or reproducibility is decreasing, follow the steps below to improve response time.

To improve response time:

1. Remove, then reinstall the rubber stopper.
2. Confirm that the inner solution exudes from the opening.
3. Repeat steps 1 and 2 several times. If performance does not improve, do one of the following, as appropriate:
   - To remove dirt, wipe off with a soft cloth soaked with a neutral detergent.
   - To remove oil residue, wipe off with a soft cloth soaked with an appropriate organic solvent, such as acetone or alcohol.
   - To remove inorganic substances, rinse with approximately 1-Normal HCL. Be sure not to immerse the electrode in a concentrated acid for a long time.

APPENDIX B

Diluent Physical Constants

Introduction

There are several types of physical constants used in particle characterization using light scattering technologies, including laser diffraction, photon correlation spectroscopy, and electrophoretic light scattering. The physical constants include values of refractive index, viscosity and dielectric constant for different substances. The values listed here are selected from various sources, including handbooks and internet sources. For several types of materials, more complete lists can be found from the following:

- *Handbook of Chemistry and Physics*, CRC Press, Boca Raton;

  This handbook is revised and published every year. The following tables are particularly useful in particle characterization:

  Physical Constants of Inorganic Compounds

  Physical Constants of Minerals

  Carbohydrates: Waxes

  Index of Refraction of Organic Compounds

  Optical Properties of Metals

- *Polymer Handbook*, Eds. Brandrup, J., Immergut, E. H., Grulke, E. A., 4th Ed., Wiley-Interscience, New York, 1999.
- *Handbook of Optical Constants of Solids*, Ed. Palik, E. D., Academic Press, New York, 1997.
- *Pigment Handbook*, Ed. Lewis, P. A., John Wiley & Sons, New York, 1988.

Water

Refractive Index

Refractive index is the ratio of the wavelength or phase velocity of an electromagnetic wave in a vacuum to that in the substance. It is a function of wavelength, temperature, and pressure. If the material is non-absorbing and non-magnetic at any wavelength, then the square of refractive index is equial to the dielectric constant at that wavelength. For absorbing materials, the complex refractive index m = *i*k is related to the absorptive index k, where the real term describes the refraction and the imaginary term describes the absorption. The following empirical equation, from

International Critical Tables Of Numerical Data, Physics, Chemistry and Technology, National Research Council (U.S.), McGraw-Hill, New York, 1926-30, describes the refractive index of water as a function of wavelength (λ) in microns and temperature. In the temperature range from 0° C to 50° C and wavelength range from 0.4 µm to 0.7 µm, the values computed from the formula are accurate up to five significant figures as compared with the numerical values in the *CRC Handbook of Chemistry and Physics*, CRC Press, Boca Raton, 1999.

Equation 16

$$n(\lambda,t)=\left(1.75648-0.013414\lambda^2+\frac{0.0065438}{\lambda^2-0.11512^2}\right)^{0.5}+0.00204976-10^{-5}\left(0.124(t-20)+0.1993(t^2-20^2)-0.000005(t^4-20^4)\right)$$

Viscosity

Viscosity is a measure of a fluid's resistance to flow. It describes the internal friction of a moving fluid. Viscosity is expressed in dyne-seconds per cm^2 or poises (g/cm's). The common unit for viscosity in centipoise (cp), which is equal to 0.01 poise. Kinematic viscosity is the ratio of viscosity to density in stokes (cm^2/s). The following empirical equations are for the viscosity of water in centipoises at different temperatures. Equation 17 is for the temperature range from 0° C to 20° C, from Hardy, R. C., Cottington, R. L., *J. Res. NBS*, 1949, 42, 573; and Equation 18 is for the temperature range from 20° C to 100° C, from Swindells, J. F., NBS. This viscosity at 20° C is 1.002 cp.

Equation 17

$$\log_{10}\eta_t=\frac{1301}{998.333+8.1855(t-20)+0.00585(t-20)^2}-1.30233$$

Equation 18

$$\log_{10}\frac{\eta_t}{\eta_{20}}=\frac{1.3272(20-t)-0.001053(t-20)^2}{t+105}$$

Dielectric Constant

Dielectric constant is a measure of the amount of electrical charge a given substance can withstand at a given electric field as compared to air. The following empirical equation is from Maryott and Smith, *NBS Cir.* 514, 1951. In the temperature range from 0° C to 60° C, the values computed from the formula are accurate up to four significant figures as compared with the numerical values in the *CRC Handbook of Chemistry and Physics*, CRC Press, Boca Raton, 1999.

Equation 19

$$D=78.30\left(1-4.579\cdot10^{-3}(t-25)+1.19\cdot10^{-5}(t-25)^2-2.8\cdot10^{-8}(t-25)^3\right)$$

Other Liquids

Table B.1 Other Liquids

Liquid	T(°C)	η(CP)	R.I.	Dielectric Constant
1,1,2,2-Tetrabromoethane	25	9.00	1.6380	7.0
1,1,2,2-Tettrachloroethane	15	1.844	1.4944	7
1,2-Dichloroethane	25/50	.464/.362	1.4443	9.3
1,2-Propanediol	25	40.4	1.4324	32
1-Octanol	25/50	7.288/3.232	1.4293	10
1-Propyl Alcohol	20/30	2.231/1.72	1.3854	20
2,2,4-Trimethylpentane	20	.5	1.3916	1.94
2-Ethoxyethanol	20	1.72	1.402	16.9
2-Propyl Alcohol	15/30	2.86/1.77	1.377	18
Acetaldehyde	10/20	.255/.22	1.3316	22
Acetic Acid	18/25	1.30/1.16	1.3718	6.15
Acetic Anhydride	18/50	.90/.62	1.3904	20
Acetone	20/25	.326/.316	1.3589	20.7
Acetonitrile	20/25	.360/.345	1.3460	37.5
Acetophenone	20/25	1.8/1.62	1.5342	17.4
Allyl Alcohol	20/30	1.363/1.07	1.4135	22
Amyl Acetate(iso)	20	.867	1.4012	7.252
Aniline	20/50	4.40/1.85	1.5863	6.89
Anisole	20	1.32	1.5179	4.3
Benzaldehyde	20/25	1.6/1.35	1.5463	17.8
Benzene	20/50	.652/.436	1.5011	2.28
Benzyl Alcohol	20/50	5.8/2.57	1.5396	13.1
Benzylamine	20	1.59	1.5401	4.6
Bromobenzene	15/30	1.196/.985	1.5602	5.5
Bromoform	15/25	2.15/1.89	1.5980	4.4
Carbon Disulfide	20/40	.363/.330	1.6280	2.64
Carbon Tetrachloride	20/50	.969/.654	1.4630	2.24
Castor Oil	25	600	1.47	4.0
Chlorobenzene	20/50	.799/.58	1.5248	2.71
Chloroform	20/25	.580/.542	1.4464	4.81
Cyclohexane	17/20	1.02/.696	1.4264	2.02
Cyclohexanol	25/50	47.5/12.3	1.4655	15

Table B.1 Other Liquids *(Continued)*

Liquid	T(°C)	η(CP)	R.I.	Dielectric Constant
Cyclohexanone	15/30	2.435/1.803	1.451	18.3
Cyclohexene	20/50	.696/.456	1.4451	2.02
Cyclopentane	20	.44	1.406	1.97
Delphi Liquid	20		1.2718	
Dibutyl Phthalate	25/50	16.6/6.47	1.4900	~ 8
Dichloromethane	15/30	.449/.393	1.4244	9.09
Diethylamine	25	.346	1.3864	3.7
Dimethyl Sulfate	15/30	2.0/1.57	1.3874	55
Dimethyl Sulfoxide	25	2.0	1.47	4.7
Dimethylaniline	20/50	1.41/.9	1.5582	4.4
Dimethylformamide	25	.802	1.42	36.7
Dioxane	15/25	1.44/1.177	1.4175	2.2
Ether (Di-Ethyl)	20/25	.233/.222	1.3497	4.3
Ethyl Acetate	20/25	.455/.441	1.3722	6.0
Ethyl Alcohol	20/30	1.2/1.003	1.3611	25
Ethyl Benzene	17/25	.691/.640	1.49	2.5
Ethyl Bromide	20/25	.402/.374	1.4239	4.9
Ethylene Bromide	20	1.721	1.5379	
Ethylene Glycol (100%)	20/30	19.9/12.2	1.4627	38.7
Ethylene Glycol (70%)	20/30	7.11/5.04	1.4627	
Ethylene Glycol (50%)	20/30	4.2/3.11	1.4627	
Ethylene Glycol (20%)	20/30	1.835/1.494	1.4627	
Ethylene Glycol (10%)	20/30	0.8120/0.699	1.4627	
Formamide	20/25	3.76/3.30	1.4453	84
Formic Acid	20/50	1.80/1.03	1.3714	58
Freon (11 and 13)	25	.415	1.36	3.1
Furfural	20/25	1.63/1.49	1.5261	42
Glycerin (100wt%)	20/25	1499/945	1.4729	42.5
Glycerin (99wt%)	20		1.4723	
Glycerin (98wt%)	20		1.4707	
Glycerin (97wt%)	20		1.4691	
Glycerin (96wt%)	20		1.4675	
Glycerin (95wt%)	20		1.4660	
Glycerin (94wt%)	20		1.4644	
Glycerin (93wt%)	20		1.4629	

Table B.1 Other Liquids *(Continued)*

Liquid	T(°C)	η(CP)	R.I.	Dielectric Constant
Glycerin (92wt%)	20		1.4614	
Glycerin (91wt%)	20		1.4599	
Glycerin (90wt%)	20		1.4584	
Glycerin (89wt%)	20		1.4569	
Glycerin (88wt%)	20		1.4554	
Glycerin (87wt%)	20		1.4539	
Glycerin (86wt%)	20		1.4524	
Glycerin (85wt%)	20		1.4509	
Glycerin (84wt%)	20		1.4493	
Glycerin (83wt%)	20		1.4477	
Glycerin (82wt%)	20		1.4461	
Glycerin (81wt%)	20		1.4445	
Glycerin (80wt%)	20		1.4429	
Glycerin (79wt%)	20		1.4414	
Glycerin (78wt%)	20		1.4398	
Glycerin (77wt%)	20		1.4383	
Glycerin (76wt%)	20		1.4368	
Glycerin (75wt%)	20		1.4353	
Glycerin (74wt%)	20		1.4339	
Glycerin (73wt%)	20		1.4324	
Glycerin (72wt%)	20		1.4309	
Glycerin (71wt%)	20		1.4294	
Glycerin (70wt%)	20		1.4279	
Glycerin (69wt%)	20		1.4264	
Glycerin (68wt%)	20		1.4249	
Glycerin (67wt%)	20		1.4234	
Glycerin (66wt%)	20		1.4219	
Glycerin (65wt%)	20		1.4204	
Glycerin (64wt%)	20		1.4190	
Glycerin (63wt%)	20		1.4175	
Glycerin (62wt%)	20		1.4160	
Glycerin (61wt%)	20		1.4145	
Glycerin (60wt%)	20		1.4130	
Glycerin (59wt%)	20		1.4115	
Glycerin (58wt%)	20		1.4100	

Table B.1 Other Liquids *(Continued)*

Liquid	T(°C)	η(CP)	R.I.	Dielectric Constant
Glycerin (57wt%)	20		1.4085	
Glycerin (56wt%)	20		1.4070	
Glycerin (55wt%)	20		1.4055	
Glycerin (54wt%)	20		1.4041	
Glycerin (53wt%)	20		1.4026	
Glycerin (52wt%)	20		1.4011	
Glycerin (51wt%)	20		1.3996	
Glycerin (50wt%)	20		1.3981	
Glycerin (49wt%)	20		1.3966	
Glycerin (48wt%)	20		1.3951	
Glycerin (47wt%)	20		1.3937	
Glycerin (46wt%)	20		1.3923	
Glycerin (45wt%)	20		1.3909	
Glycerin (44wt%)	20		1.3895	
Glycerin (43wt%)	20		1.3882	
Glycerin (42wt%)	20		1.3868	
Glycerin (41wt%)	20		1.3855	
Glycerin (40wt%)	20/25	3.750/3.181	1.3841	
Glycerin (39wt%)	20		1.3828	
Glycerin (38wt%)	20		1.3814	
Glycerin (37wt%)	20		1.3809	
Glycerin (36wt%)	20		1.3787	
Glycerin (35wt%)	20		1.3774	
Glycerin (34wt%)	20		1.3761	
Glycerin (33wt%)	20		1.3747	
Glycerin (32wt%)	20		1.3734	
Glycerin (31wt%)	20		1.3720	
Glycerin (30wt%)	20/25		1.3707	
Glycerin (29wt%)	20		1.3694	
Glycerin (28wt%)	20		1.3680	
Glycerin (27wt%)	20		1.3667	
Glycerin (26wt%)	20		1.3654	
Glycerin (25wt%)	20		1.3640	
Glycerin (24wt%)	20		1.3627	
Glycerin (23wt%)	20		1.3614	

Table B.1 Other Liquids *(Continued)*

Liquid	T(°C)	η(CP)	R.I.	Dielectric Constant
Glycerin (22wt%)	20		1.3601	
Glycerin (21wt%)	20		1.3588	
Glycerin (20wt%)	20/25	1.769/1.542	1.3575	
Glycerin (19wt%)	20		1.3562	
Glycerin (18wt%)	20		1.3549	
Glycerin (17wt%)	20		1.3536	
Glycerin (16wt%)	20		1.3523	
Glycerin (15wt%)	20		1.3511	
Glycerin (14wt%)	20		1.3498	
Glycerin (13wt%)	20		1.3485	
Glycerin (12wt%)	20		1.3473	
Glycerin (11wt%)	20		1.3460	
Glycerin (10wt%)	20/25	1.311/1.153	1.3448	
Glycerin (9wt%)	20		1.3436	
Glycerin (8wt%)	20		1.3424	
Glycerin (7wt%)	20		1.3412	
Glycerin (6wt%)	20		1.3400	
Glycerin (5wt%)	20		1.3388	
Glycerin (4wt%)	20		1.3376	
Glycerin (3wt%)	20		1.3365	
Glycerin (2wt%)	20		1.3353	
Glycerin (1wt%)	20		1.3342	
Heptane	20/25	.409/.386	1.3876	1.92
Hexane	20/25	.326/.294	1.3754	1.89
Iodoethane	25/50	.556/.444	1.5168	7.4
Isobutyl Alcohol	15/20	4.703/3.9	1.3968	15.8
Isopar G	20/40	1.491/1.12	1.4186	2.0
Isopar M	37.8	34-36.5	1.4362	
Isopentane	20	.223	1.3550	
Isopropyl Alcohol	15/30	2.861/1.77	1.377	18
Isopropyl Ether	25/50	.396/.304	1.3680	3.85
Iso-Propylacetate	20	.525	1.377	
m-Bromoaniline	20	6.81	1.6260	13
Methanol	20/25	.597/.547	1.3312	33.6
Methyl Acetate	20/40	.381/.320	1.3614	7

Table B.1 Other Liquids *(Continued)*

Liquid	T(°C)	η(CP)	R.I.	Dielectric Constant
Methyl Cyclohexane	25/50	.679/.501	1.4253	2
Methyl Ethyl Ketone	20/50	.42/.31	1.38	19
Methyl Iodide	20	.500	1.5293	7.0
Methyl Isobutyl Ketone	20/50	.579/.542	1.396	18
Methylacetate	20/50	.381/.286	1.3594	6.7
Methylene Chloride	15/30	.449/.393	1.4237	9.08
m-Toluidine	20	.81	1.5711	6.0
m-Xylene	15/20	.650/.620	1.4972	2.37
n-Amyl Alcohol	20/50	2.948/1.42	1.4099	13.9
n-Butyl Acetate	20	.73	1.3951	5.0
n-Butyl Alcohol	20/50	2.948/1.42	1.3993	17.8
n-Decane	20/50	.92/.615	1.4120	2.0
Nitrobenzene	20/50	2.0/1.24	1.5529	35
Nitromethane	20/25	.66/.620	1.3818	39.4
n-Nonane	20/50	.711/.492	1.4054	1.972
n-Octane	20/50	.542/.389	1.3975	2.0
n-Pentane	0/20	.277/.240	1.3570	1.84
n-Propylacetate	20/50	.537/.39	1.384	6.3
o-Dichlorobenzene	25	1.32	1.5515	99
o-Nitrotoluene	20/40	2.37/1.63	1.5474	27.4
o-Toluidine	20	.39	1.5728	6.34
o-Xylene	16/20	.876/.810	1.5055	2.568
Propyl Bromide	20	.524	1.4341	7.2
Propylene Glycol (100%)	20/40	56/18	1.433	
Propylene Glycol (30%)	20/30	3.0/2.1	1.367	
Propylene Glycol (20%)	20/30	2.18/1.59	1.355	
Propylene Glycol (10%)	20/30	1.5/1.2	1.344	
p-Toluidine	20	80	1.5532	6.0
p-Xylene	16/20	.696/.648	1.4958	2.27
Pyridine	20	.95	1.5102	12.5
Sec-Butyl Alcohol	25/50	3.096/1.332	1.3954	15.8
Styrene (Vinyl Benzene)	20/50	.749/.502	1.55	2.4
Sulphuric Acid	20	.254	1.8430	84
Tert-Butyl Alcohol	25/50	4.312/1.421	1.3847	11.5
Tetrachloroethylene	15	.93	1.5044	2.5

Table B.1 Other Liquids *(Continued)*

Liquid	T(°C)	η(CP)	R.I.	Dielectric Constant
Tetradecane	20/50	2.31/1.32	1.429	
Tetrahydrofuran	20/30	.575/.525	1.40	7.6
Toluene	20/30	.590/.526	1.4969	2.4
Trichloroethane	20	.2	1.4377	7.5
Trichloroethylene	20	.57	1.4784	3.4
Triethylamine	25/50	.347/.273	1.4003	2.4
Water	20/25	1.002.8904	1.3330	80.2

Liquid viscosity values in the third column are at the corresponding temperatures in the second column. Refractive indices are at the sodium yellow line (λ = 589.3 nm) at 20° C. Dielectric constants are at 20° C. The values are from the *CRC Handbook of Chemistry and Physics*, CRC Press, Boca Raton, 1999.

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APPENDIX C

Alpha and Beta Values

Introduction

The alpha (a) and beta (b) constants are determined separately for each component. The following table provides a list of a and b values for a select number of polymers.

Table C.1 Alpha and Beta Values

Sample	Solvent	Temp. (°C)	Alpha	Beta
cis-4 Polybutadiene	Hexatriacontan	80	1.45´10-4	0.561
Methyl cellulose	Water	20	0.79´10-4	0.56
Poly(1,4-benzamide)	Dimethylacetamide	25	4.38´10-4	0.82
Poly(1-hexene sulfone	Acetone	20	3.0´10-4	0.71
Poly(1-p-tolyl-2,5-dioxopyrrolidin-3,4-diyl)	Dimethylformamide	21	3.47´10-3	0.58
Poly(3-methyl-1-butene silsesquioxane)	Butyl acetate	24	1.1´10-3	0.69
Poly(acrilonitrile)	Dimethylformamide	25	3.2´10-4	0.63
Poly(acrylamide)	Water	20	8.46´10-4	0.69
Poly(acrilonitrile)	Dimethylformamide	35	7.8´10-4	0.63
Poly(butadiene co-acrylonitrile)	Hexane	22	1.38´10-4	0.5
Poly(butadiene) linear	Dioxane	25	6.34´10-5	0.496
Poly(butyl methacrylate)	2-propyl alcohol	21.5	6.3´10-5	0.5
Poly(cetyl methacrylate)	Heptane	21	1.74´10-3	0.64
Poly(cholesteryl acrylate)	Benzene	21	3.2´10-4	0.54
Poly(ethyl acrylate-co-acrylic-acid)	Water/NaCl (0.05 mol/dm3)		5.76´10-4	0.65
Poly(ethyl acrylate-co-acrylic-acid)	Water/NaCl (0.05 mol/dm3)		1.54´10-4	0.52
Poly(ethyl acrylate-co-acrylic-acid)	Water/NaCl (1.0 mol/dm3)		1.24´10-4	0.5
Poly(G-benzyl-L-glutamate)	Dimethylformamide	21	2.8´10-3	0.8
Poly(isobutene)	Heptane	25	5.01´10-4	0.555
Poly(isobutene)	Octane	20.9	5.01´10-4	0.555

Table C.1 Alpha and Beta Values *(Continued)*

Sample	Solvent	Temp. (°C)	Alpha	Beta
Poly(isoprene)	Chloroform	20	3.5´10-4	0.42
Poly(isoprene) (18-armed star)	Carbon tetrachloride	50	1.33´10-8	0.57
Poly(isoprene) (4-armed star)	Carbon tetrachloride	50	1.17´10-8	0.61
Poly(isoprene) (linear)	Carbon tetrachloride	5	1.54´10-8	0.54
Poly(isoprene) (linear)	Carbon tetrachloride	50	1.3´10-8	0.61
Poly(isoprene) 1,4-cis	Hexane	20	3.98´10-2	0.55
Poly(methyl methacrylate)	n-butyl chloride	35.6	9.6´10-4	0.59
Poly(methyl methacrylate)	Ethyl acetate	20	1.61´10-4	0.48
Poly(m-phenylene isophthalamide)	Dimethylacetamide	26	1.13´10-4	0.56
Poly(m-phenylene isophthalamide)	Dimethylacetamide/3% LiCl	25	0.55´10-4	0.56
Poly(n-butyliminocarbonyl)	Tetrahydrofuran	20	1.69´10-4	0.85
Poly(N-isopropyl methacrylate)	Water	20	2.02´10-4	0.57
Poly(oxydiphenylsilylene)	Benzene	21	6.16´10-4	0.63
Poly(p-carbethoxyphenylmethacrylamide)	Ethyl acetate	21	2.8´10-4	0.69
Poly(p-ethoxycarbonyl phenylmethacrylate)	Ethyl acetate		2.8´10-4	0.69
Poly(styrene)	Methyl ethyl ketone	25	3.1´10-4	0.53
Poly(styrene)	Toluene	20	unknown	0.53
Poly(styrene) NBS 419	Cyclohexane	35	1.21´10-4	0.5
Poly(styrene) NBS 705	Methyl ethyl ketone	25	1.96´10-4	0.49

Determining alpha and beta

Photon correlation spectroscopy (PCS) is an indirect means of determining the molecular weight of polymeric samples, such as proteins, polymers, and colloidal particles. The principle of using PCS to determine the molecular weight of a sample in solution or in suspension is based on the empirical relationship between the molecular weight (M_w) and the translation diffusion coefficient (D_T) of the sample:

Equation 20

$$M_w=(\alpha/D_T)^{1/\beta}$$

The two constants in this empirical equation represent the structural effect (the parameter a) and the solvent effect (the parameter b), respectively, on the translational motion of the molecules (or

particles). For polydisperse samples, the obtained molecular weight is an ill-defined mean value that may not be the same as the mean values obtained by other means.

The values of a and b can be obtained from literature or experimentally. There are listings of a and b values in the DelsaNano and in the Polymer Handbook and CRC Physical Chemistry Handbook. The values are only valid for the specific molecules (or particles) in the specific solvent (or dispersing medium) and temperature, although the temperature variation may be minimal in the normal temperature range. If the literature values are not available, a and b can be obtained experimentally with the DelsaNano using the following procedure, provided that two (or more) samples of known molecular weight are available.

Procedure

NOTE This procedure is executed outside the DelsaNano software.

1. Obtain two or more (preferably monodisperse) samples of the same type with known molecular weight values. These samples are going to be used as the "standards." The molecular weight range of these standards should be wider than or similar to the range of the unknown samples.

2. Make solutions (or suspensions) of these standards using the same solvent and at the same temperature that are to be used for the unknown samples.

3. Select Yes to Molecular Weight Calculation in the Analysis Parameters panel of the Size SOP Designer. Enter alpha = 1 and beta = 1.

4. Make good measurements of the standard samples and use the Cumulants analysis to obtain the molecular weight. The obtained values are called the apparent molecular weight $M_{w.app}$.

5. Plot $Log(M_w)$ versus $Log(M_{w.app})$ for all standards and do a linear least-square fitting.

6. The a and b values can then be obtained from the following equations:

   Equation 21

   $$\alpha = 10^{(-\text{intercept/slope})}$$

   Equation 22

   $$\beta = -1/\text{slop}$$

   Explanation: The above procedure is based on Equation 20. In Equation 20, if a = 1 and b = 1, then $M_{w.app} = 1/D_T$. When plotting in the logarithmic scale, $Log(M_w) = 1/bLog(a)-1/bLog(M_{w.app})$. Therefore, Slope = -1/b; and Intercept = 1/bLog(a).

References for Alpha and Beta Values

1. Kramer O. and Frederick, JE., 1971. Macromolecules 4:613.
2. Poddubnyi IY, Podalinski AV, and Grechanovskii VA; 1966. Vysokomolekul Soedin 8: 1556
3. Brandrup J., Immergut EH., Grylke EA., 1999. Polymer Handbook, fourth edition. Wiley & Sons, Inc.

APPENDIX D

Graphs and Table Displays

Introduction

This appendix includes descriptions and examples of the following:

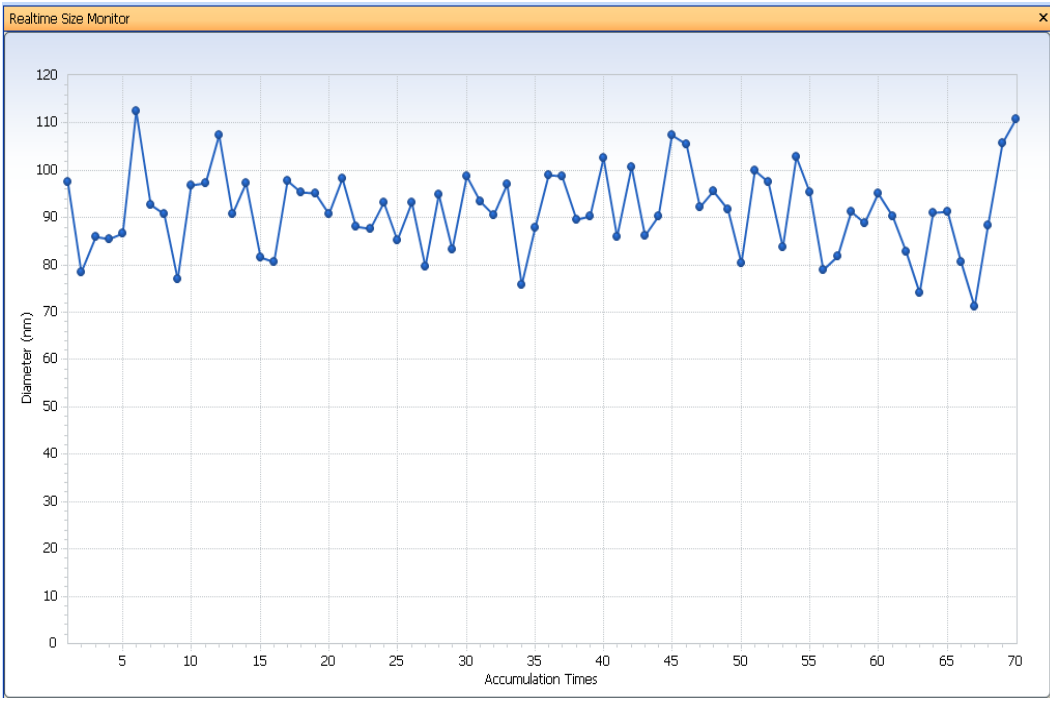
- Realtime Size Monitor
- Realtime Size Table
- Differential Intensity Distribution Graph
- Cumulative Intensity Distribution Graph
- Differential Volume Distribution Graph
- Cumulative Volume Distribution Graph
- Differential Number Distribution Graph
- Cumulative Number Distribution Graph
- Ln(G1(t)) Plot Graph
- Differential Size Distribution Table
- Cumulative Size Distribution Table
- ACF Listing
- Condition Summary
- Distribution Graph with Zeta Potential of the Sample
- Distribution Graph with Mobility of the Sample
- ACF (Base)
- Test Measurement
- 3D Graph
- Peak Value Table
- Graph display adjustments options

Realtime Size Displays

Realtime Size Monitor

This shows the stability of the sample during measurement.

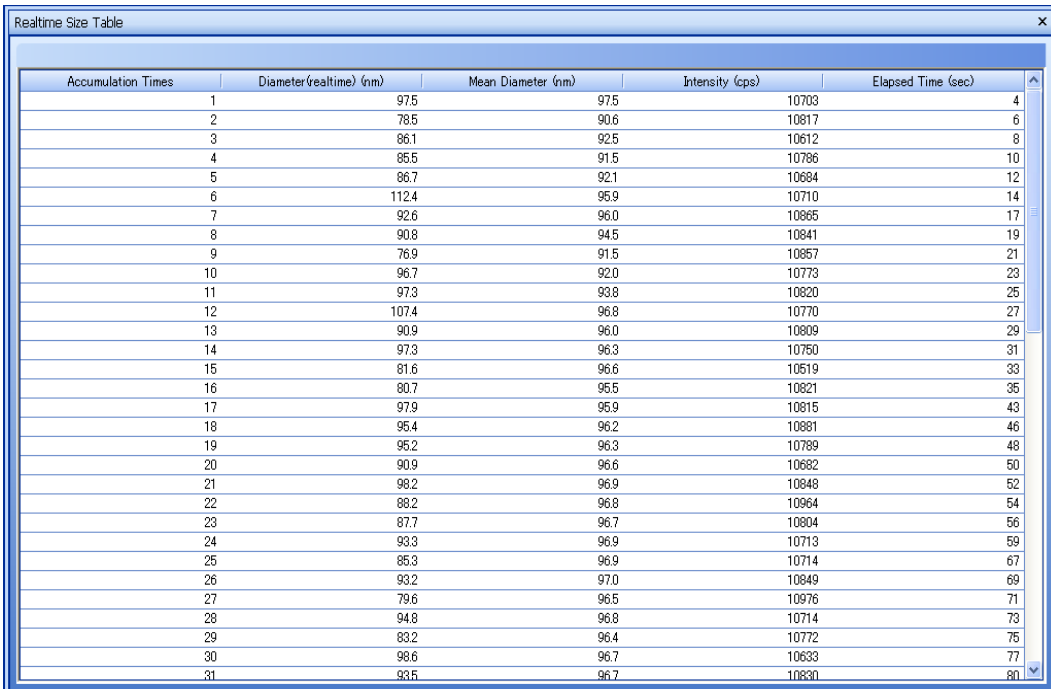
Figure D.1 Realtime Size Monitor



Realtime Size Table

This shows the realtime size data (raw) in tabular form.

Figure D.2 Realtime Size Table


Realtime Size Table

Accumulation Times	Diameter(realtime) (nm)	Mean Diameter (nm)	Intensity (cps)	Elapsed Time (sec)
1	97.5	97.5	10703	4
2	78.5	90.6	10817	6
3	86.1	92.5	10612	8
4	85.5	91.5	10786	10
5	86.7	92.1	10684	12
6	112.4	95.9	10710	14
7	92.6	96.0	10865	17
8	90.8	94.5	10841	19
9	76.9	91.5	10857	21
10	96.7	92.0	10773	23
11	97.3	93.8	10820	25
12	107.4	96.8	10770	27
13	90.9	96.0	10809	29
14	97.3	96.3	10750	31
15	81.6	96.6	10519	33
16	80.7	95.5	10821	35
17	97.9	95.9	10815	43
18	95.4	96.2	10881	46
19	95.2	96.3	10789	48
20	90.9	96.6	10682	50
21	98.2	96.9	10848	52
22	88.2	96.8	10964	54
23	87.7	96.7	10804	56
24	93.3	96.9	10713	59
25	85.3	96.9	10714	67
26	93.2	97.0	10849	69
27	79.6	96.5	10976	71
28	94.8	96.8	10714	73
29	83.2	96.4	10772	75
30	98.6	96.7	10633	77
31	93.5	96.7	10830	80

Intensity Distribution Graphs

Photon correlation spectroscopy (PCS) directly measures intensity (weighted) size distributions, displayed as intensity histograms in the DelsaNano. In these histograms, the magnitude of each peak is proportional to the percent (% amount of the total scattered intensity due to particles. For example, in the graph below, the intensity distribution peak average is shown as 100.9 ±-25.0 nm. The DelsaNano measures intensity distributions and optionally converts them to volume or number distributions.

Figure D.3 Differential Intensity Distribution

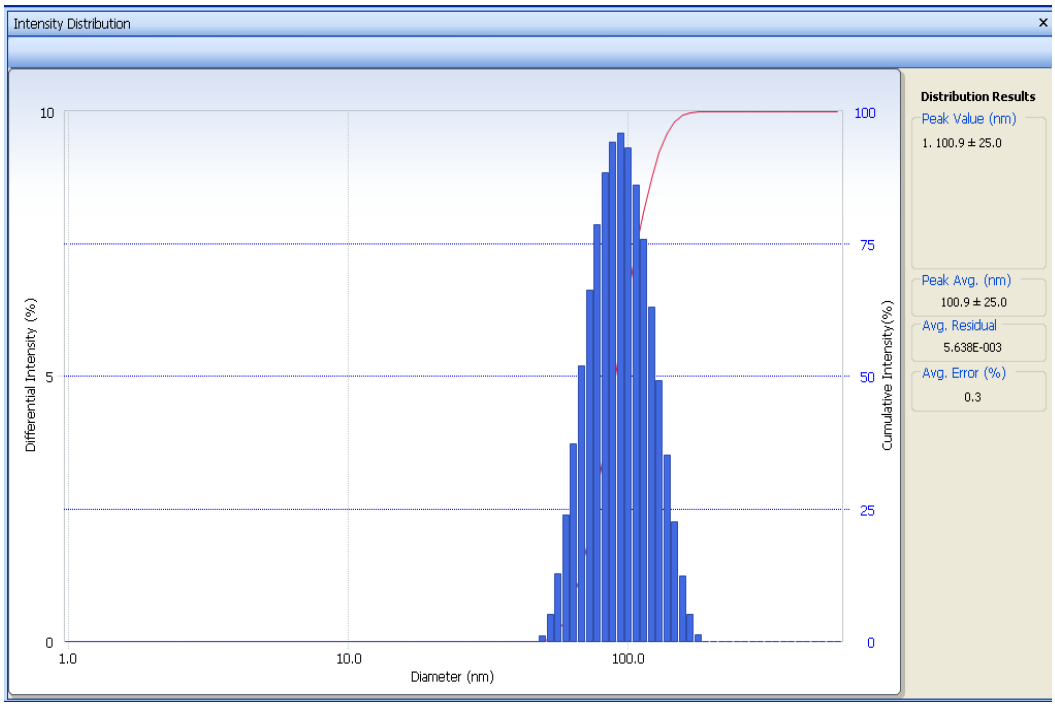
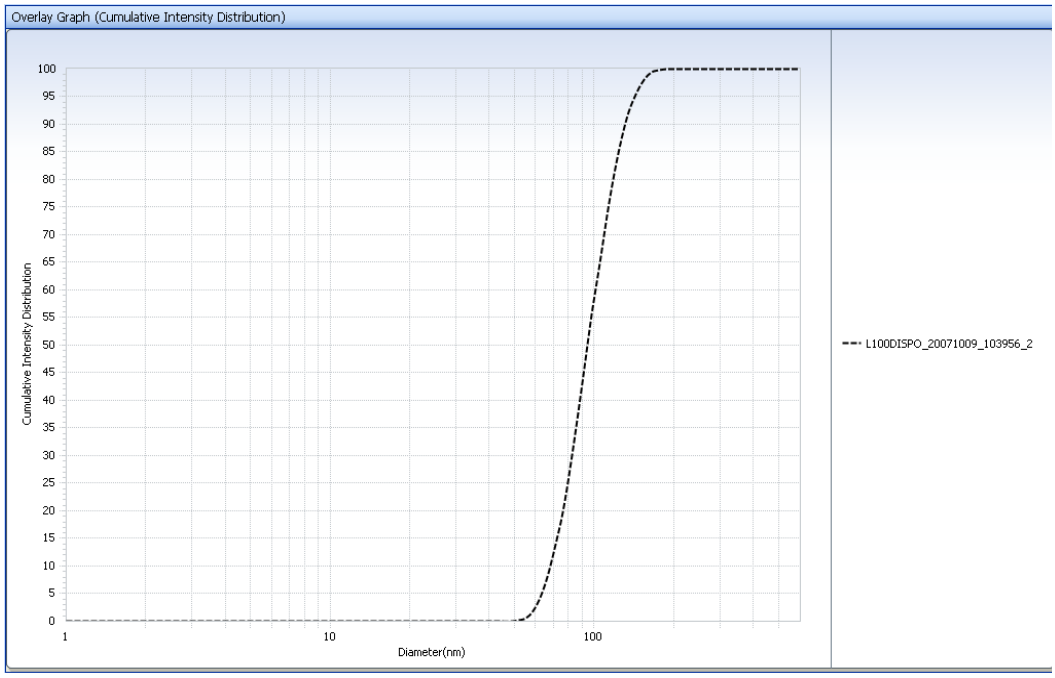


Figure D.4 Cumulative Intensity Distribution



Volume Distribution Graphs

Volume distributions (same as weight distributions) give the relative volume of particles of each size in a sample. In addition, intensity results are dependent on scattering angle; volume results are not. The following graph represents the volume distribution for the same sample shown in Figure D.3. For example, in the graph below, the volume distribution peak average is shown as 84.7 ±20.3 nm.

The corresponding percent (% amount) in each bin and cumulative percent (% amount) can be obtained from the size distribution table and cumulative size distribution table, respectively.

Figure D.5 Differential Volume Distribution

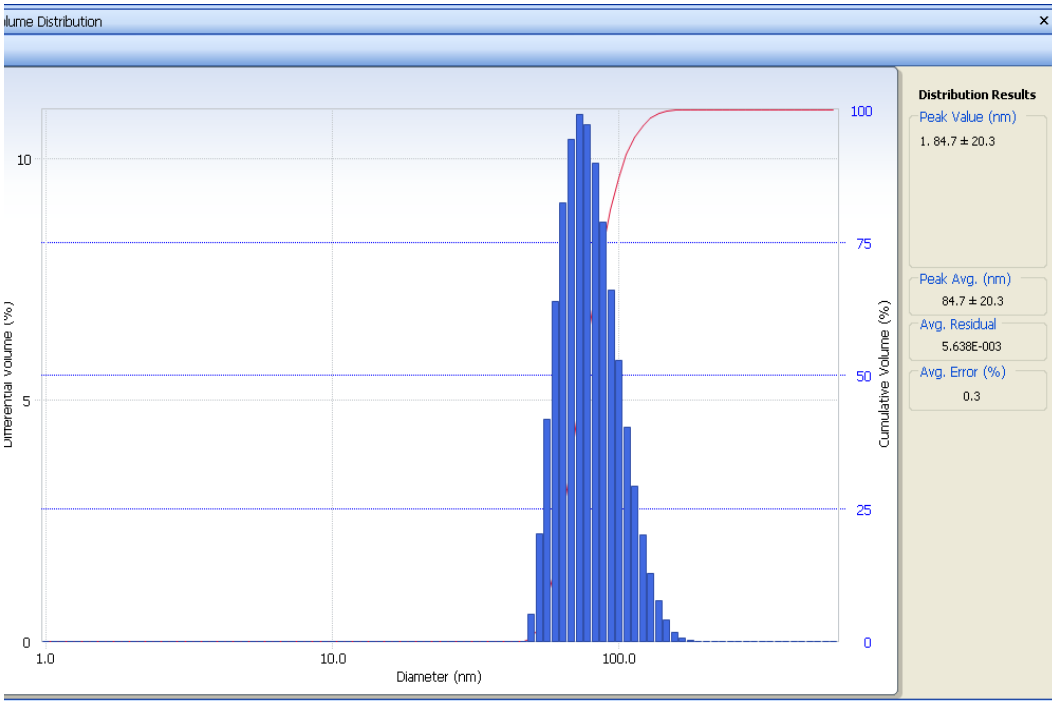
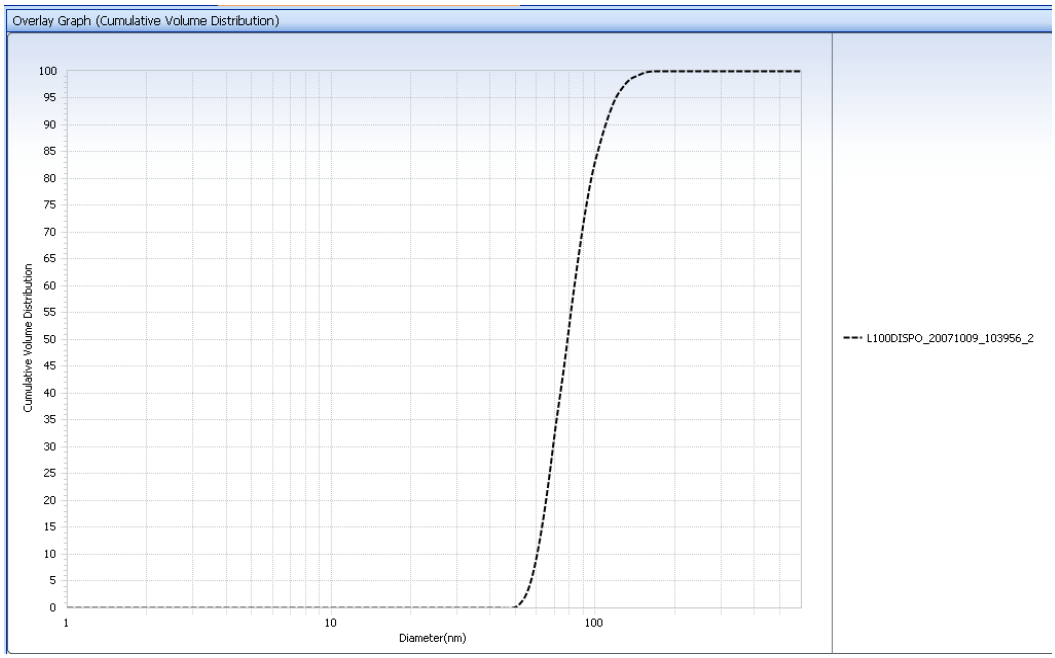


Figure D.6 Cumulative Volume Distribution



Number Distribution Graphs

Number percentage (%) distributions are determined by dividing the volume percentage results by the cubed diameter of the particles in the distribution. The following graph represents the number distribution for the sample shown in Figure D.3. For example, in the graph below, the number distribution peak average is shown as 73.6 ±15.2 nm.

The corresponding percent (% amount) in each bin and cumulative percent (% amount) can be obtained from the size distribution table and cumulative size distribution table, respectively.

Figure D.7 Differential Number Distribution

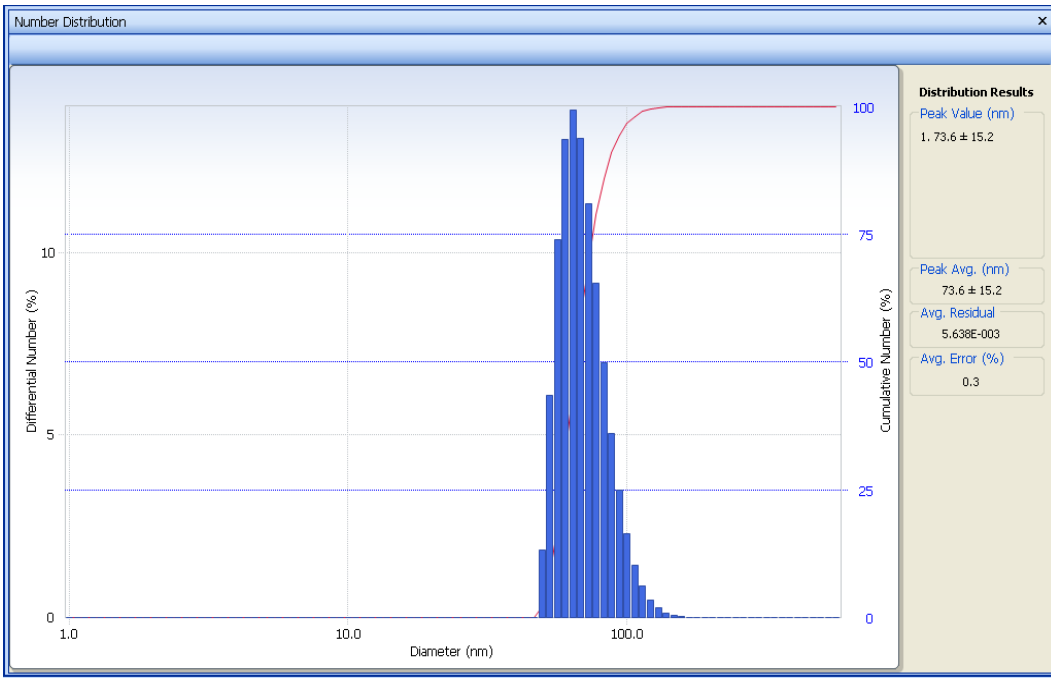
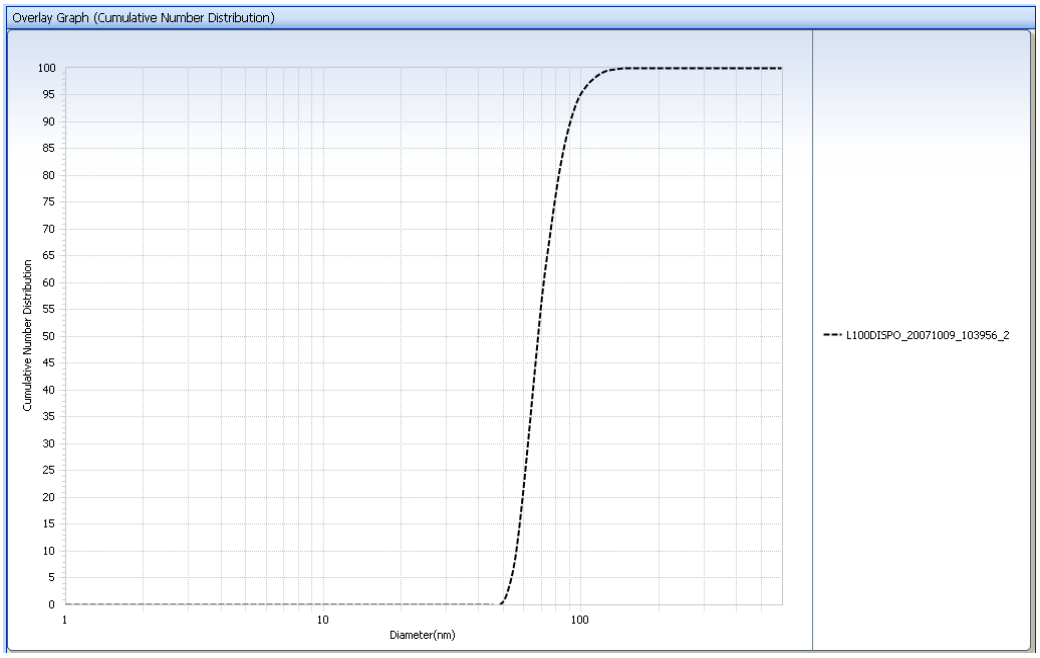


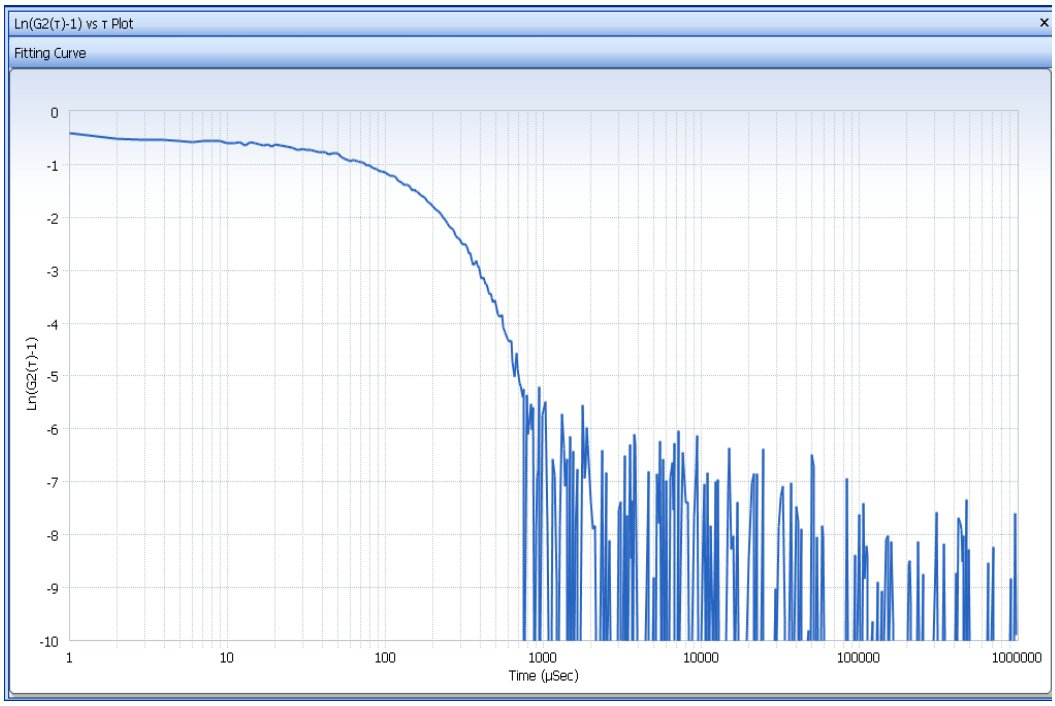
Figure D.8 Cumulative Number Distribution



Ln(G2($_t$)-1 Plot

This shows the natural logarithmic graph of (G2($_{t)-1}$) vs. decay time. This indicates if there is any noise in the signal. The curve should be smooth where the ACF exists and become noisy where the ACF touches the baseline.

Figure D.9 Ln(G2(t)-1) Plot

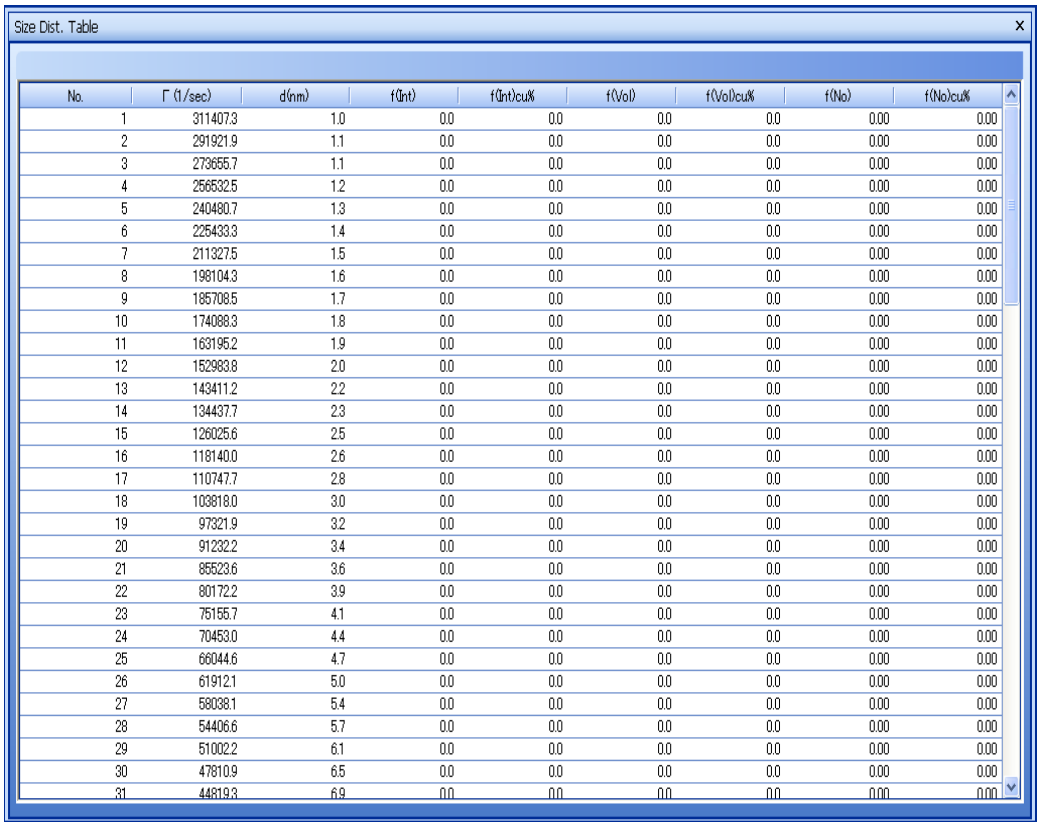


Size Distribution Tables

The differential and cumulative graphs are available in tabular form. In this form, the data is separated into a select number of bins spaced along your selected size range. The listings give the size corresponding to each bin and the relative amount of scattered intensity in each bin. The total intensity adds up to 100%. The absolute percentages appearing in the tabular data do not directly correspond to the absolute percentages appearing in the graphs because the graphs are also scaled to total 100%, and they contain more bins than the tabular form.

Differential Size Distribution Table

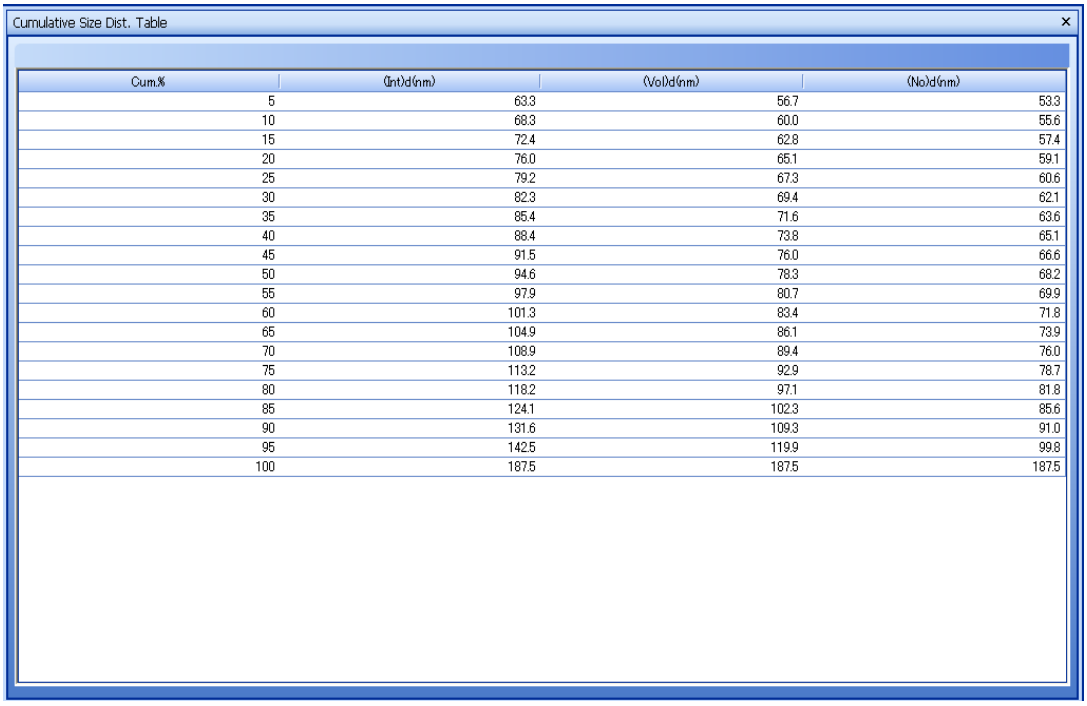
Figure D.10 Differential Size Distribution Table



No.	Γ (1/sec)	d(nm)	f(Int)	f(Int)cu%	f(Vol)	f(Vol)cu%	f(No)	f(No)cu%
1	311407.3	1.0	0.0	0.0	0.0	0.0	0.00	0.00
2	291921.9	1.1	0.0	0.0	0.0	0.0	0.00	0.00
3	273655.7	1.1	0.0	0.0	0.0	0.0	0.00	0.00
4	256532.5	1.2	0.0	0.0	0.0	0.0	0.00	0.00
5	240480.7	1.3	0.0	0.0	0.0	0.0	0.00	0.00
6	225433.3	1.4	0.0	0.0	0.0	0.0	0.00	0.00
7	211327.5	1.5	0.0	0.0	0.0	0.0	0.00	0.00
8	198104.3	1.6	0.0	0.0	0.0	0.0	0.00	0.00
9	185708.5	1.7	0.0	0.0	0.0	0.0	0.00	0.00
10	174088.3	1.8	0.0	0.0	0.0	0.0	0.00	0.00
11	163195.2	1.9	0.0	0.0	0.0	0.0	0.00	0.00
12	152983.8	2.0	0.0	0.0	0.0	0.0	0.00	0.00
13	143411.2	2.2	0.0	0.0	0.0	0.0	0.00	0.00
14	134437.7	2.3	0.0	0.0	0.0	0.0	0.00	0.00
15	126025.6	2.5	0.0	0.0	0.0	0.0	0.00	0.00
16	118140.0	2.6	0.0	0.0	0.0	0.0	0.00	0.00
17	110747.7	2.8	0.0	0.0	0.0	0.0	0.00	0.00
18	103818.0	3.0	0.0	0.0	0.0	0.0	0.00	0.00
19	97321.9	3.2	0.0	0.0	0.0	0.0	0.00	0.00
20	91232.2	3.4	0.0	0.0	0.0	0.0	0.00	0.00
21	85523.6	3.6	0.0	0.0	0.0	0.0	0.00	0.00
22	80172.2	3.9	0.0	0.0	0.0	0.0	0.00	0.00
23	75155.7	4.1	0.0	0.0	0.0	0.0	0.00	0.00
24	70453.0	4.4	0.0	0.0	0.0	0.0	0.00	0.00
25	66044.6	4.7	0.0	0.0	0.0	0.0	0.00	0.00
26	61912.1	5.0	0.0	0.0	0.0	0.0	0.00	0.00
27	58038.1	5.4	0.0	0.0	0.0	0.0	0.00	0.00
28	54406.6	5.7	0.0	0.0	0.0	0.0	0.00	0.00
29	51002.2	6.1	0.0	0.0	0.0	0.0	0.00	0.00
30	47810.9	6.5	0.0	0.0	0.0	0.0	0.00	0.00
31	44819.3	6.9	0.0	0.0	0.0	0.0	0.00	0.00

Cumulative Size Distribution Table

Figure D.11 Cumulative Size Distribution Table

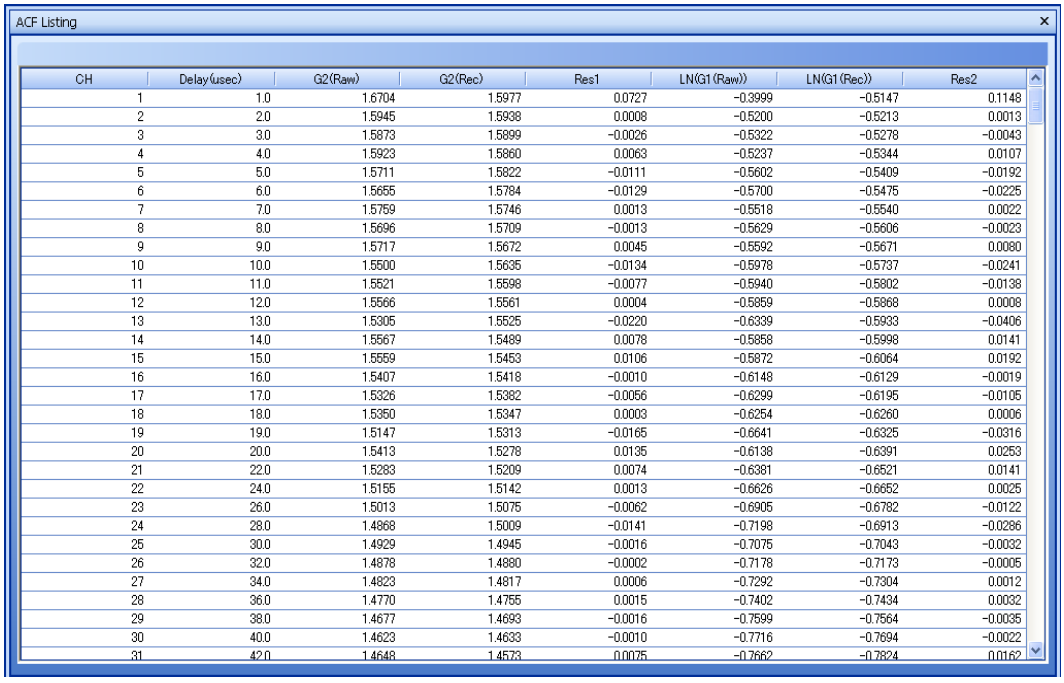


Cum.%	(Int)d(nm)	(Vol)d(nm)	(No)d(nm)
5	63.3	56.7	53.3
10	68.3	60.0	55.6
15	72.4	62.8	57.4
20	76.0	65.1	59.1
25	79.2	67.3	60.6
30	82.3	69.4	62.1
35	85.4	71.6	63.6
40	88.4	73.8	65.1
45	91.5	76.0	66.6
50	94.6	78.3	68.2
55	97.9	80.7	69.9
60	101.3	83.4	71.8
65	104.9	86.1	73.9
70	108.9	89.4	76.0
75	113.2	92.9	78.7
80	118.2	97.1	81.8
85	124.1	102.3	85.6
90	131.6	109.3	91.0
95	142.5	119.9	99.8
100	187.5	187.5	187.5

ACF Listing

This presents the Autocorrelation Function (ACF) raw data in tabular form.

Figure D.12 ACF Listing

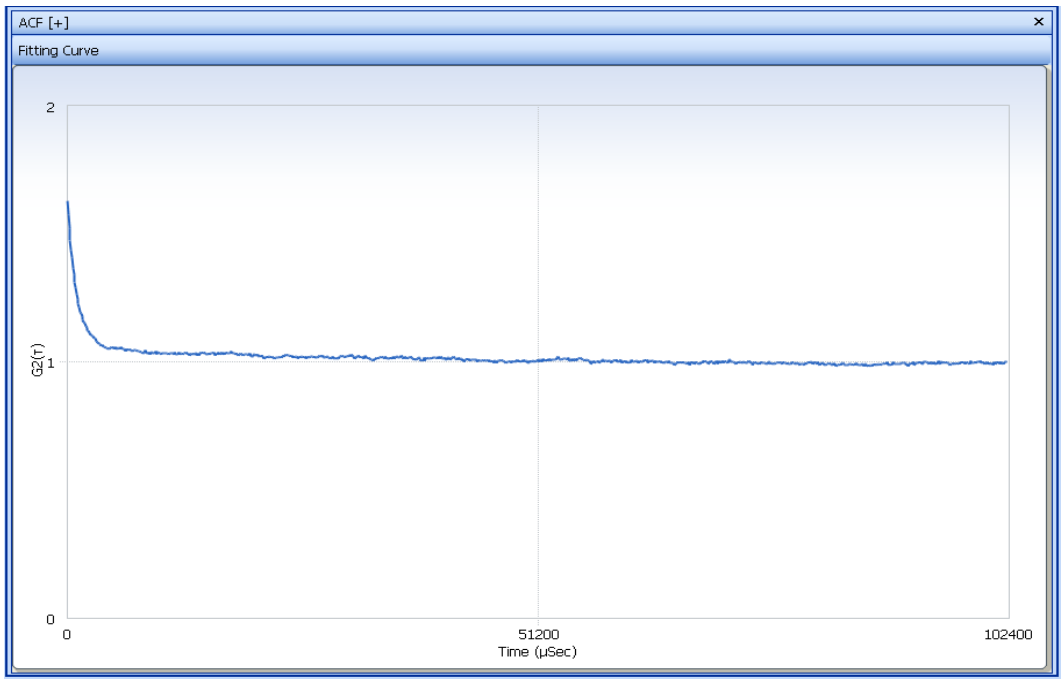


ACF Listing

CH	Delay(usec)	G2(Raw)	G2(Rec)	Res1	LN(G1(Raw))	LN(G1(Rec))	Res2
1	1.0	1.6704	1.5977	0.0727	-0.3999	-0.5147	0.1148
2	2.0	1.5945	1.5938	0.0008	-0.5200	-0.5213	0.0013
3	3.0	1.5873	1.5899	-0.0026	-0.5322	-0.5278	-0.0043
4	4.0	1.5923	1.5860	0.0063	-0.5237	-0.5344	0.0107
5	5.0	1.5711	1.5822	-0.0111	-0.5602	-0.5409	-0.0192
6	6.0	1.5655	1.5784	-0.0129	-0.5700	-0.5475	-0.0225
7	7.0	1.5759	1.5746	0.0013	-0.5518	-0.5540	0.0022
8	8.0	1.5696	1.5709	-0.0013	-0.5629	-0.5606	-0.0023
9	9.0	1.5717	1.5672	0.0045	-0.5592	-0.5671	0.0080
10	10.0	1.5500	1.5635	-0.0134	-0.5978	-0.5737	-0.0241
11	11.0	1.5521	1.5598	-0.0077	-0.5940	-0.5802	-0.0138
12	12.0	1.5566	1.5561	0.0004	-0.5859	-0.5868	0.0008
13	13.0	1.5305	1.5525	-0.0220	-0.6339	-0.5933	-0.0406
14	14.0	1.5567	1.5489	0.0078	-0.5858	-0.5998	0.0141
15	15.0	1.5559	1.5453	0.0106	-0.5872	-0.6064	0.0192
16	16.0	1.5407	1.5418	-0.0010	-0.6148	-0.6129	-0.0019
17	17.0	1.5326	1.5382	-0.0056	-0.6299	-0.6195	-0.0105
18	18.0	1.5350	1.5347	0.0003	-0.6254	-0.6260	0.0006
19	19.0	1.5147	1.5313	-0.0165	-0.6641	-0.6325	-0.0316
20	20.0	1.5413	1.5278	0.0135	-0.6138	-0.6391	0.0253
21	22.0	1.5283	1.5209	0.0074	-0.6381	-0.6521	0.0141
22	24.0	1.5155	1.5142	0.0013	-0.6626	-0.6652	0.0025
23	26.0	1.5013	1.5075	-0.0062	-0.6905	-0.6782	-0.0122
24	28.0	1.4868	1.5009	-0.0141	-0.7198	-0.6913	-0.0286
25	30.0	1.4929	1.4945	-0.0016	-0.7075	-0.7043	-0.0032
26	32.0	1.4878	1.4880	-0.0002	-0.7178	-0.7173	-0.0005
27	34.0	1.4823	1.4817	0.0006	-0.7292	-0.7304	0.0012
28	36.0	1.4770	1.4755	0.0015	-0.7402	-0.7434	0.0032
29	38.0	1.4677	1.4693	-0.0016	-0.7599	-0.7564	-0.0035
30	40.0	1.4623	1.4633	-0.0010	-0.7716	-0.7694	-0.0022
31	42.0	1.4648	1.4573	0.0075	-0.7662	-0.7824	0.0162

Normal and abnormal correlation functions are shown in the examples that follow.

Figure D.13 Normal Linear ACF



In the ACF with short sampling time example below, the sampling time has to be increased.

Figure D.14 ACF with Short Sampling Time

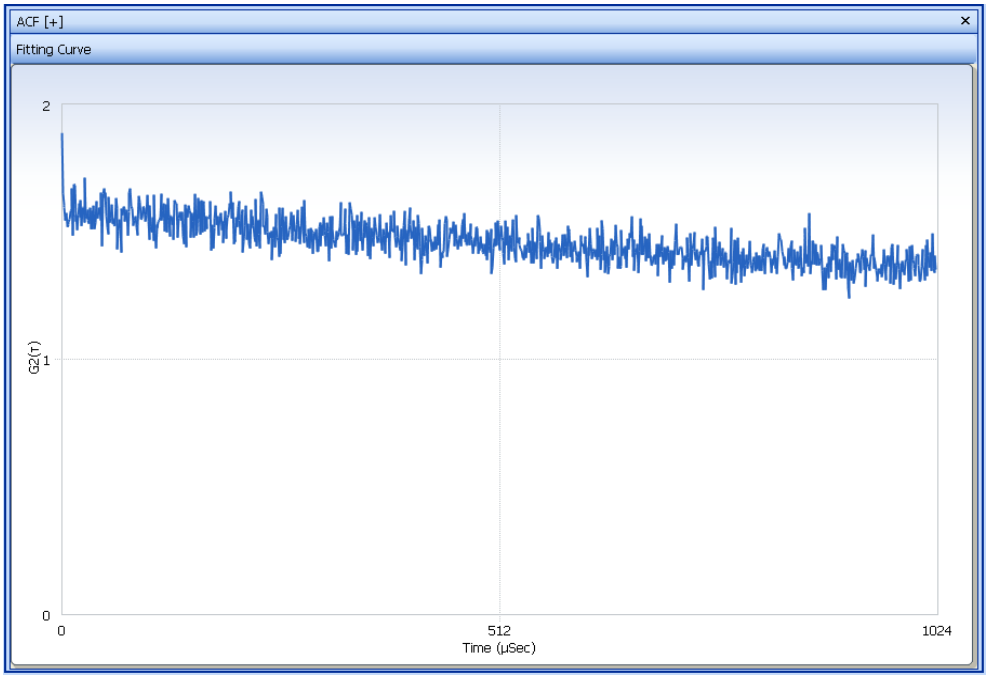


Figure D.15 Normal Log ACF

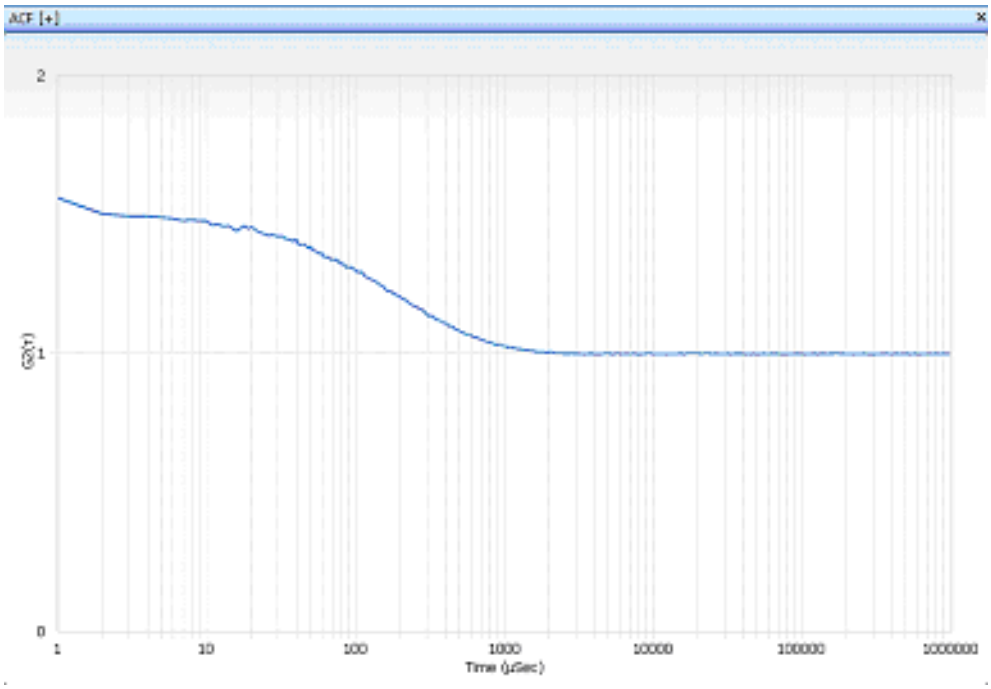


Figure D.16 ACF Noisy - Low Sample Concentration

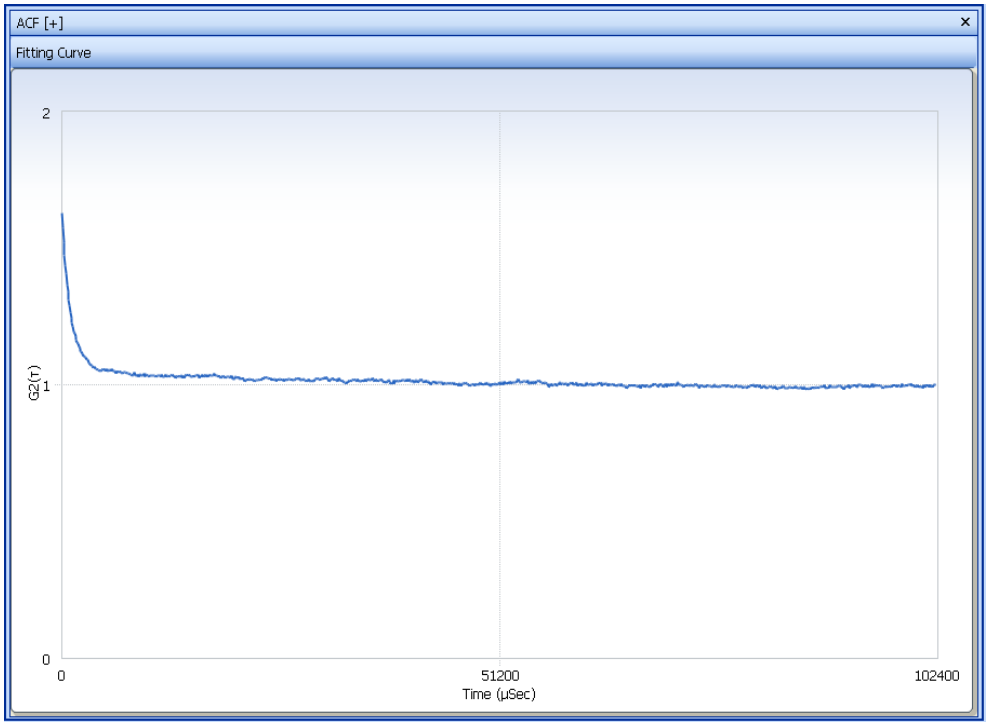
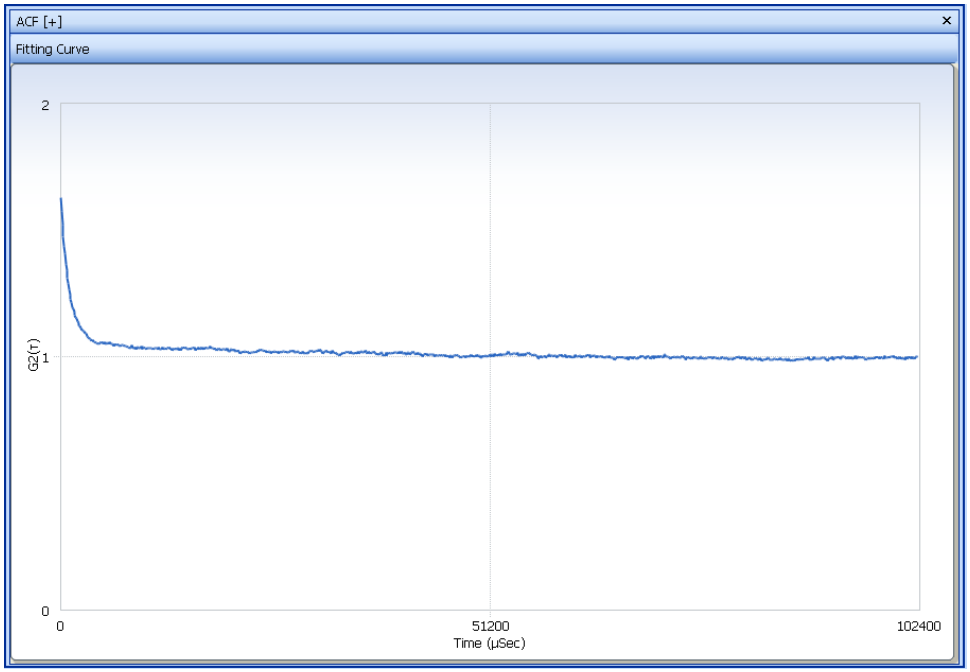


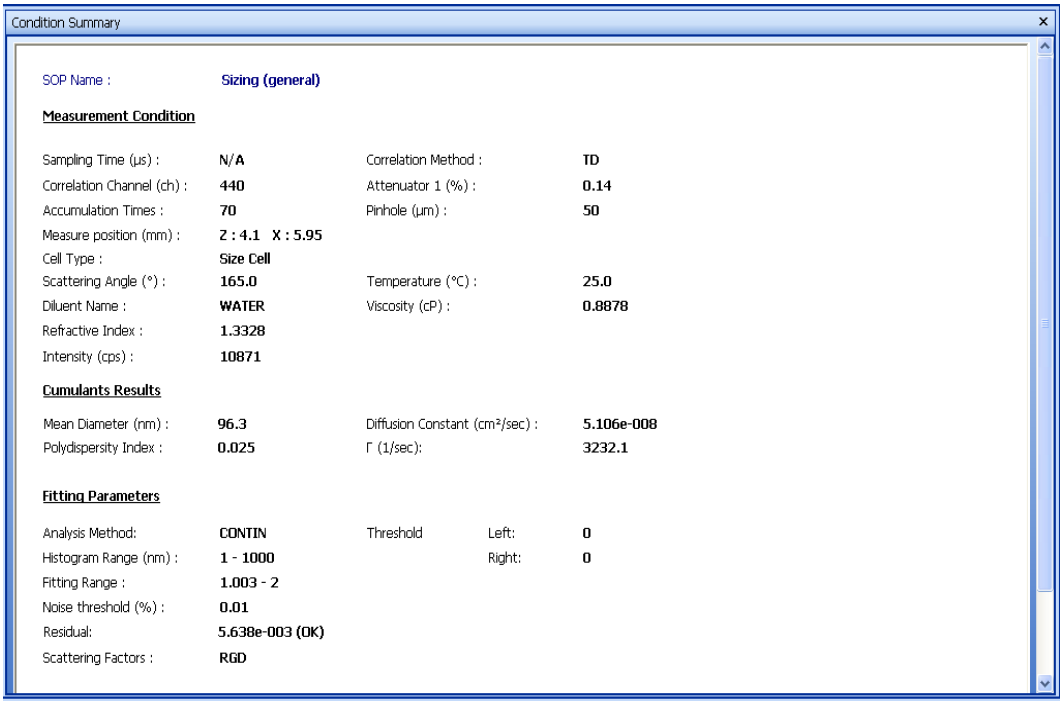
Figure D.17 ACF for Small Particles (Less than 10 mm)



Condition Summary

This presents the summary of the measurement, analysis, cell, and diluent conditions.

Figure D.18 Condition Summary



Distribution Graphs

Distribution graph is plotted between Intensity, Mobility/Zeta Potential and Frequency. You can view the graph at all positions at which the zeta potential/mobility is measured. The positions are set in the Cell Parameters of the Zeta SOP Designer. See CHAPTER 1, *Operation*.

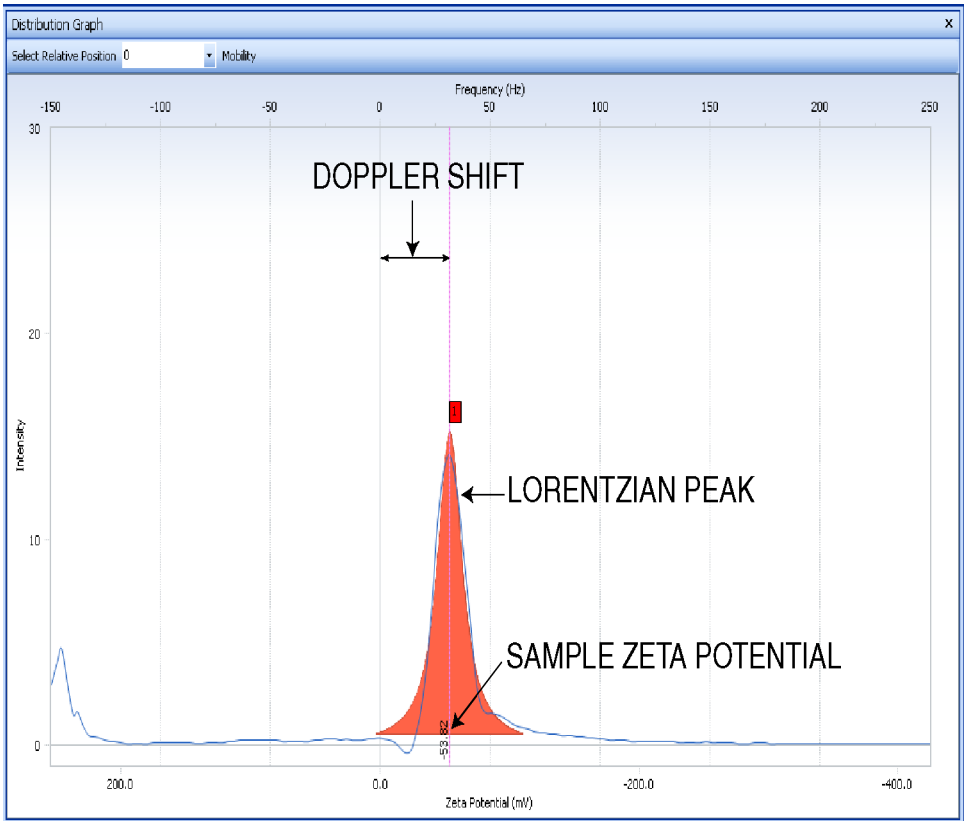
To change the position, select the desired position from the Select Relative Position drop-down menu at the top of the graph.

To switch from Zeta Potential to Mobility, click **Mobility** at the top of the graph. The name of the button then changes to **Zeta Potential**, allowing you to switch back to Zeta Potential.

The pink vertical line represents the Doppler Frequency Shift of the signal from Base Frequency.

The Lorentzian Peak represents the Brownian motion of the particles. The number “1” indicates the Lorentzian Peak number.

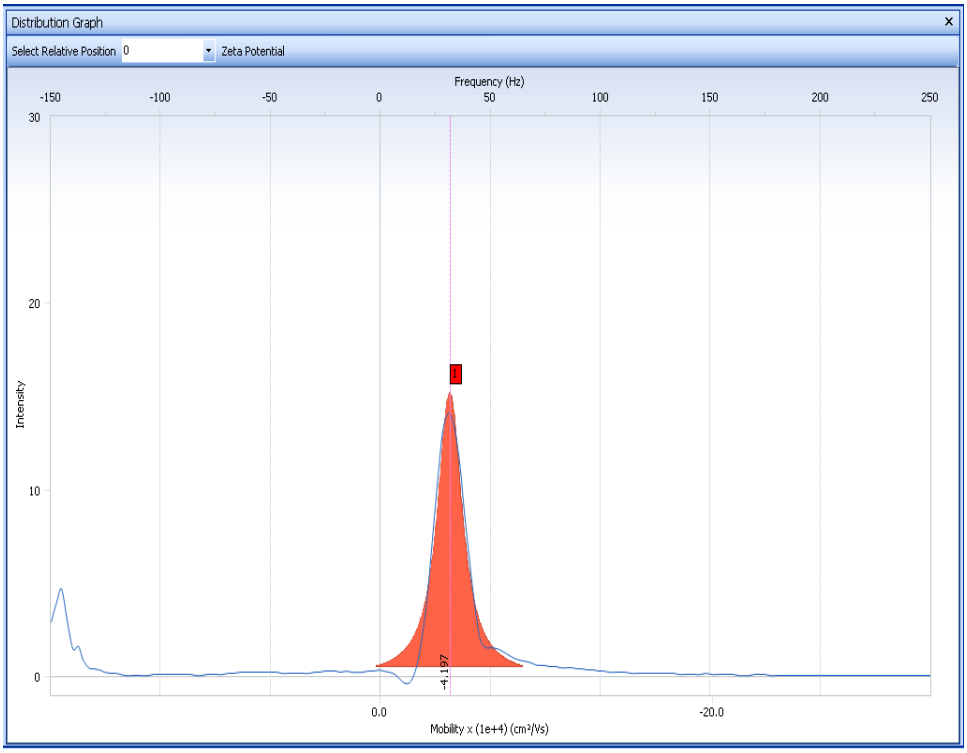
Figure D.19 Distribution Graph with Zeta Potential of the Sample



NOTE In Figure D.19, the blue line indicates raw data, and the red curve indicates fitted data.

The values displayed in the graph below represent the mobility of the sample.

Figure D.20 Distribution Graph with Mobility of the Sample



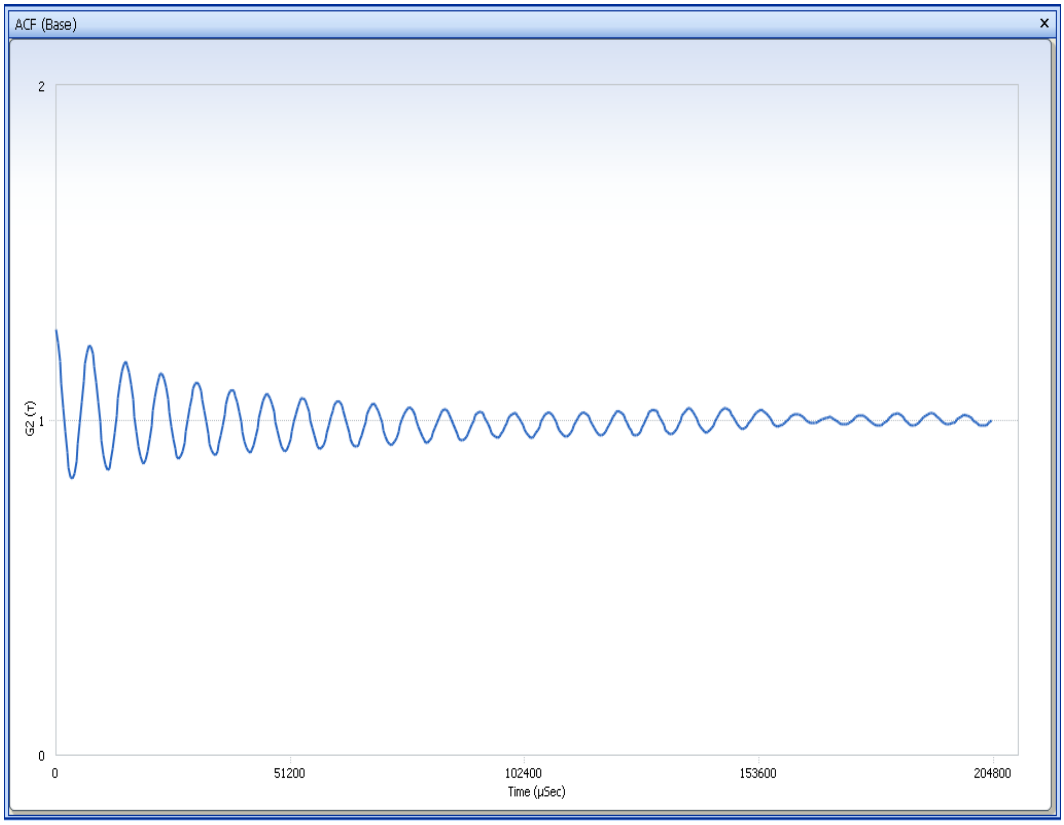
ACF (Base)

The Electrophoretic Mobility of the sample is determined by using heterodyne type of measurement. The measurement requires a reference signal, the Base, whose frequency is compared with the scattered laser light frequency from the sample and the Doppler Shift (modulator signal) is calculated.

The Base frequency is dependent on the type of cell used and the concentration of the sample. Typically, for Flow cell, Disposable Zeta cell, Low Conductivity and Flat Surface cell, the Base frequency varies between 110-140 Hz, and for High Concentration cell, the Base frequency varies between 220-270 Hz.

If the cell center is not appropriate, you may not get a good ACF for Base measurement. In that case, the cell center of the cell can be detected before carrying out the analysis.

Figure D.21 ACF Base



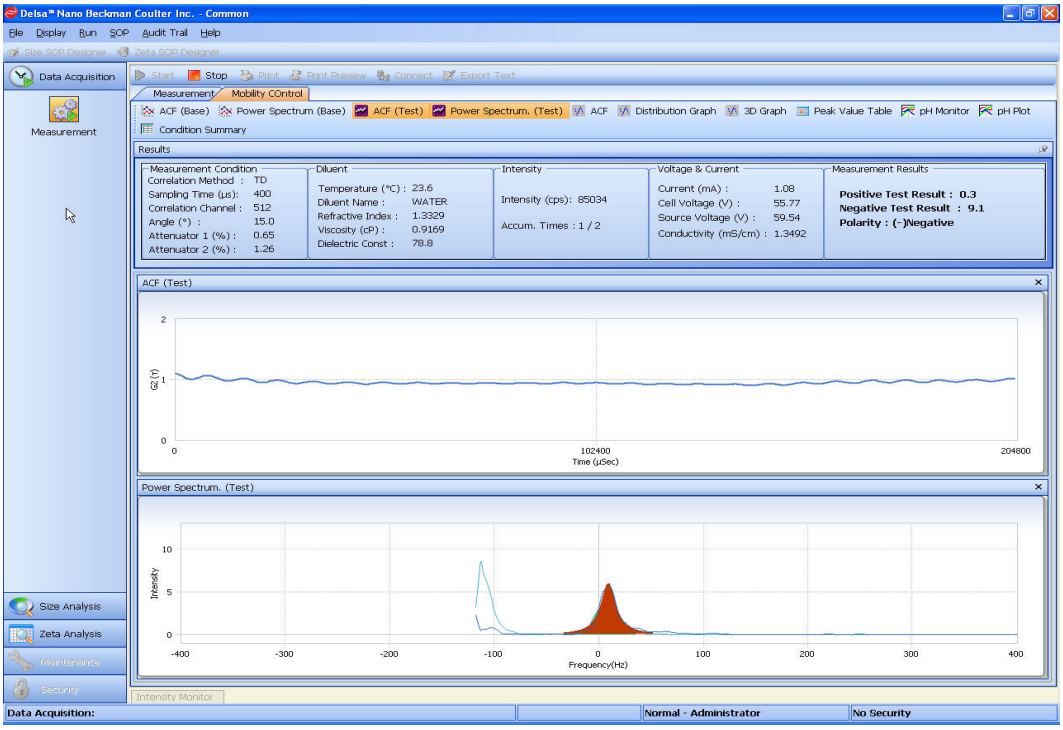
Test Measurement

This is a preliminary test the instrument performs to determine the sign of the polarity to be applied to the sample. The test measurement consists of ACF and Power Spectrum. After analysis, the test results are displayed. For example:

- Positive Test Results: -12.5
- Negative Test Results: 15.8
- Polarity: (-) Negative

This decision is based on the Doppler Shift in the frequency. To interpret the results, assume the sign of polarity that is to be determined as “x.” When “x” is multiplied with a positive value to get a negative result (-12.5), “x” has to be negative. Similarly, when “x” is multiplied with a negative value to get a positive result (15.8), “x” has to be negative. As a result, the sign of polarity is negative (-).

Figure D.22 ACF and Power Spectrum of Test Measurement



3D Graph

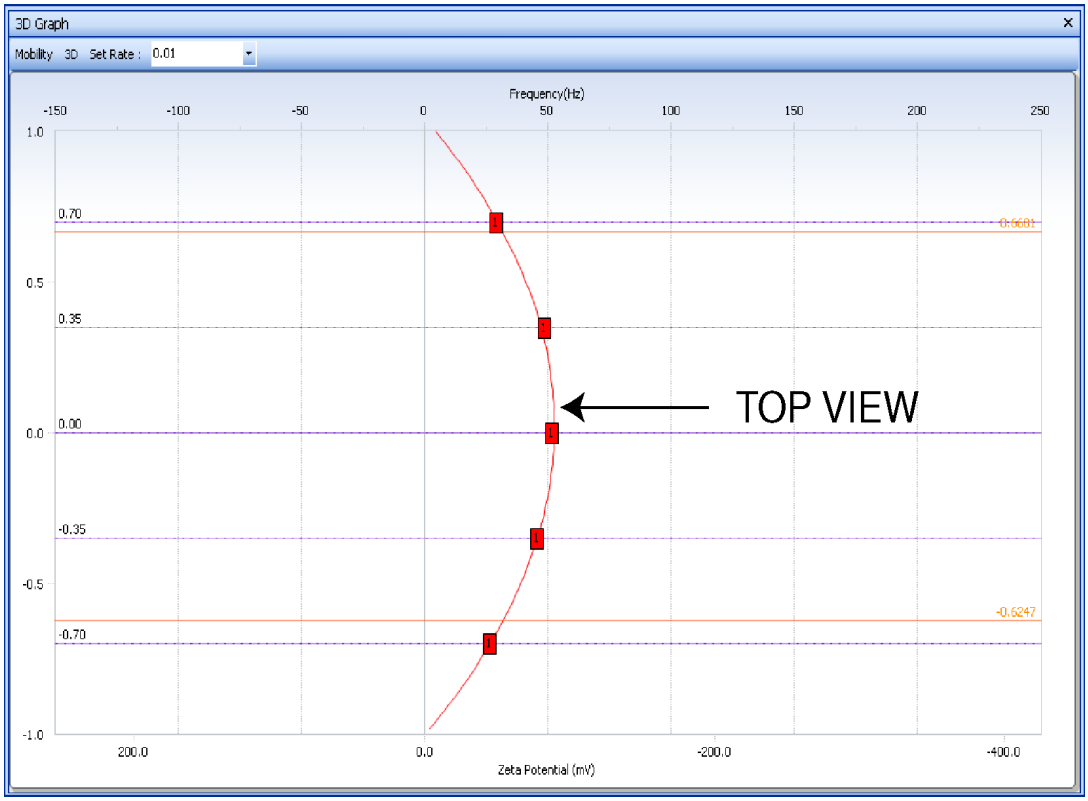
This provides the electroosmotic velocity profile of the sample.

This is a graph plotted between Intensity, Mobility/Zeta Potential, and Frequency.

The graph can be viewed at all positions at which the zeta potential/mobility is measured, including a Set Rate where you can change the height/peak/stationary layers. The positions can be set in the Cell parameters of the Zeta SOP Designer or selected under the Set Rate drop down menu.

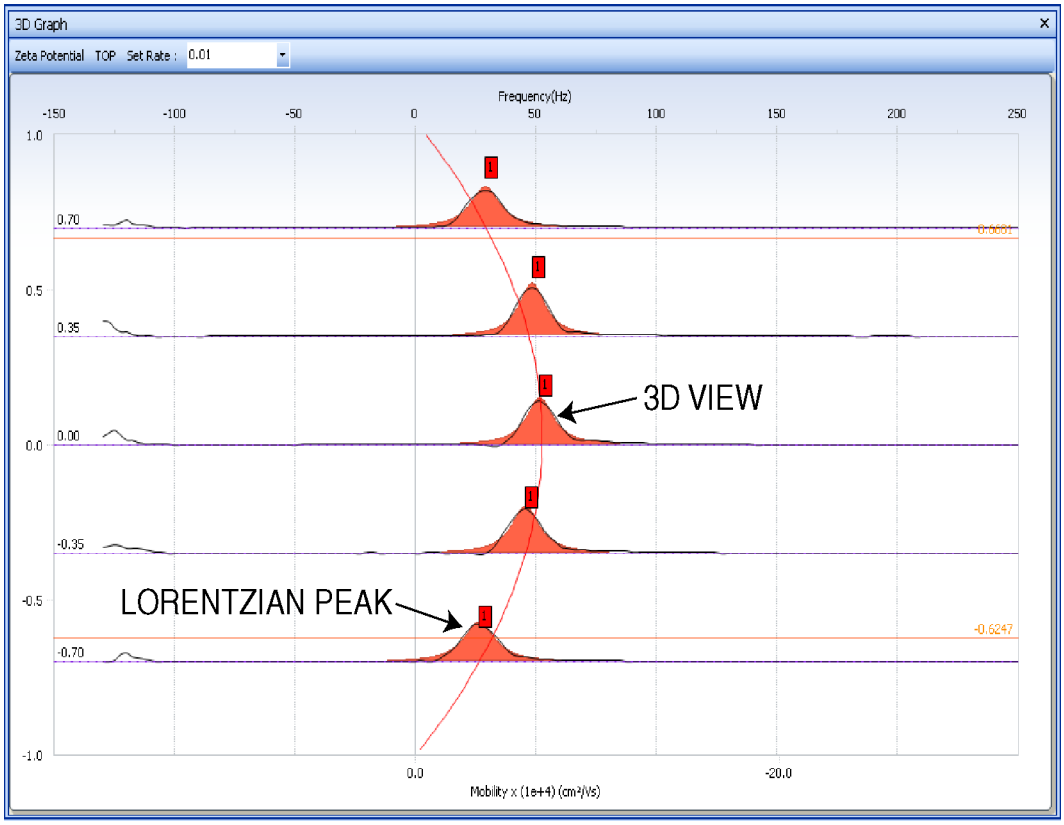
To switch from Zeta Potential to Mobility (or Mobility to Zeta Potential), click **Mobility / Zeta Potential** at the top of the graph. To change the view, click **TOP / 3D** .

Figure D.23 3D Graph with Zeta Potential and Top View



The Lorentzian Peak represents the Brownian motion of the particles, as shown in Figure D.24.

Figure D.24 3D Graph with Mobility and 3D View



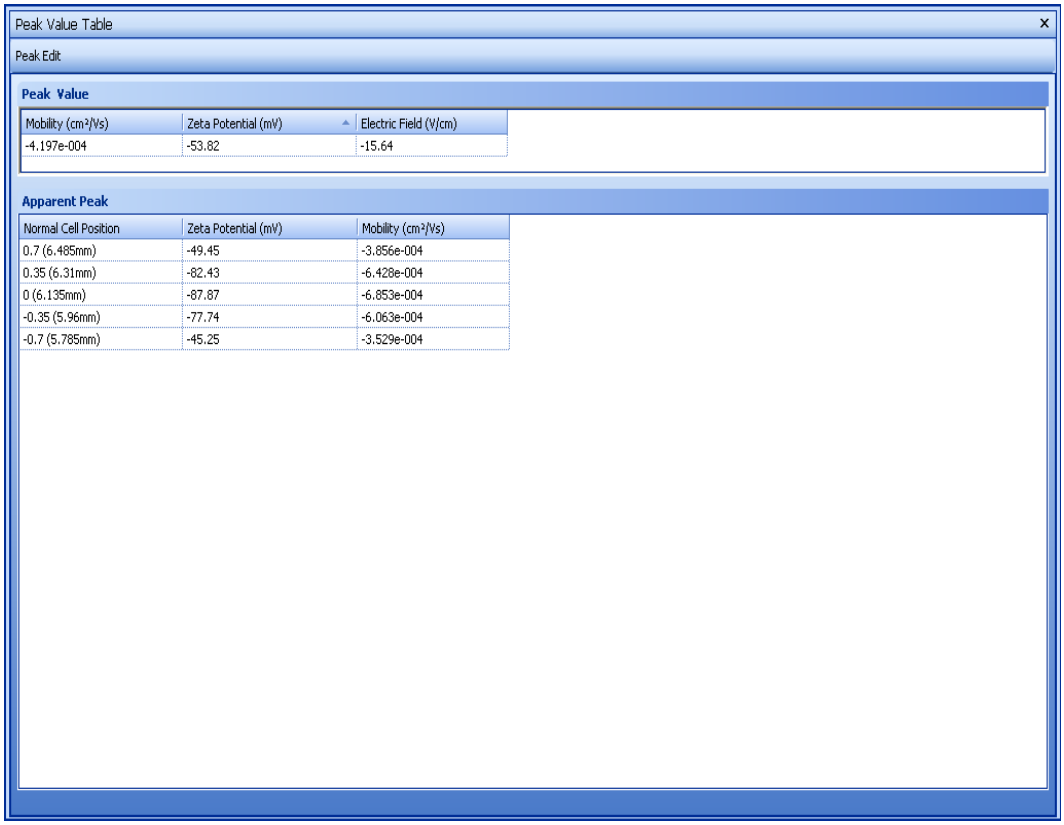
Peak Value Table

This provides the mobility, zeta potential of the sample, and the electric field applied.

This also provides apparent mobility and zeta potential peak values at the locations specified in the cell condition of the Zeta SOP Designer (for example, 5 locations for flow cell).

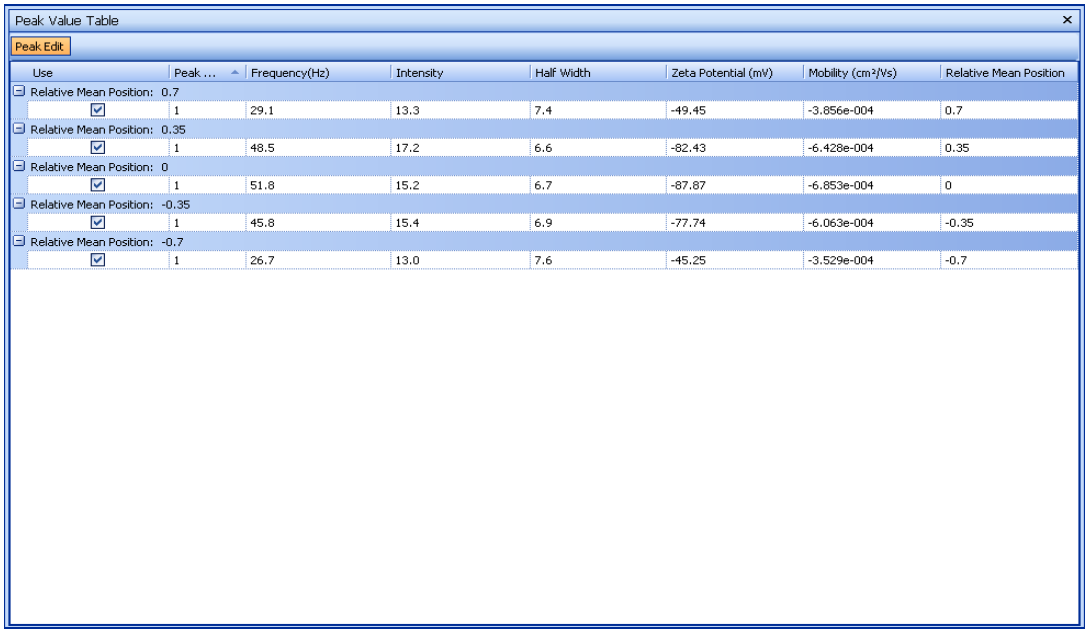
The Peak Edit feature allows you to select the peaks for analyzing data zeta potential.

Figure D.25 Peak Value Table



Peak Edit Feature

The Peak Edit feature allows you to select the peaks for analyzing data zeta potential. To adjust the values, click **Peak Edit** in the Peak Value Table and select or deselect the desired peaks.

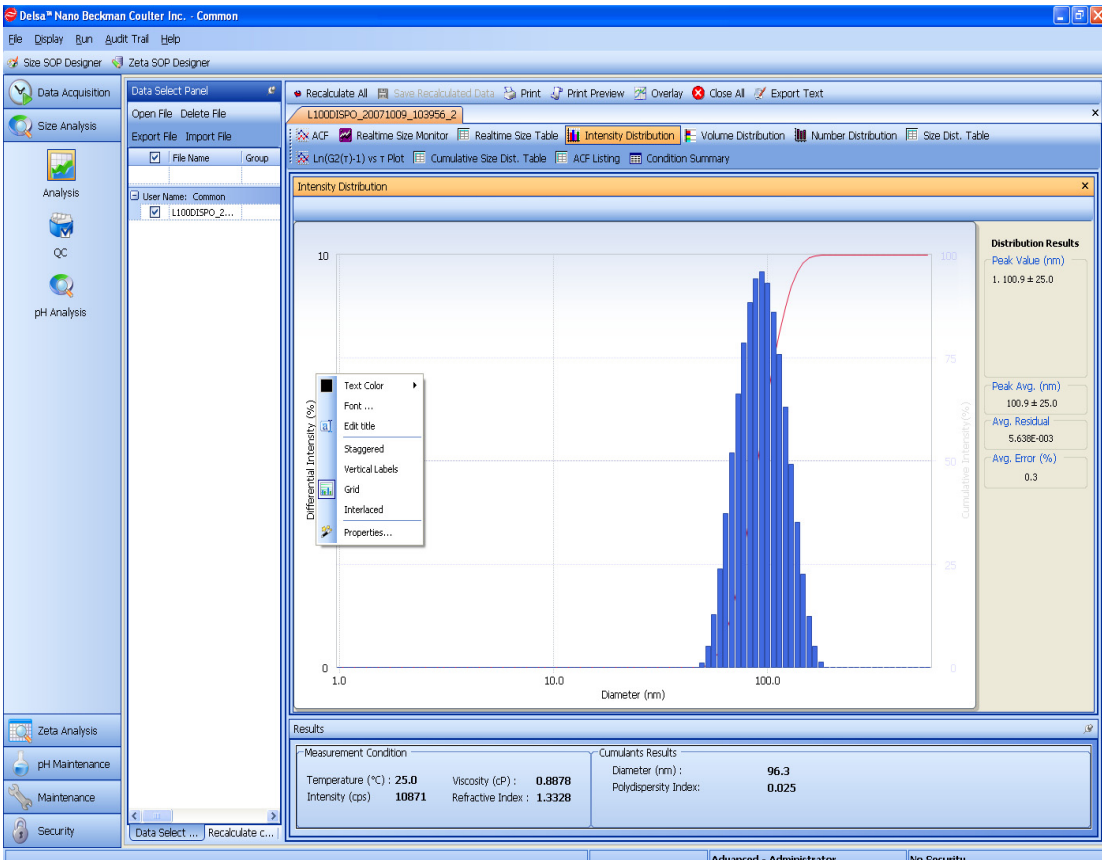


Graph Display Adjustment Options

The options for adjusting graph displays are in pop-up menus.

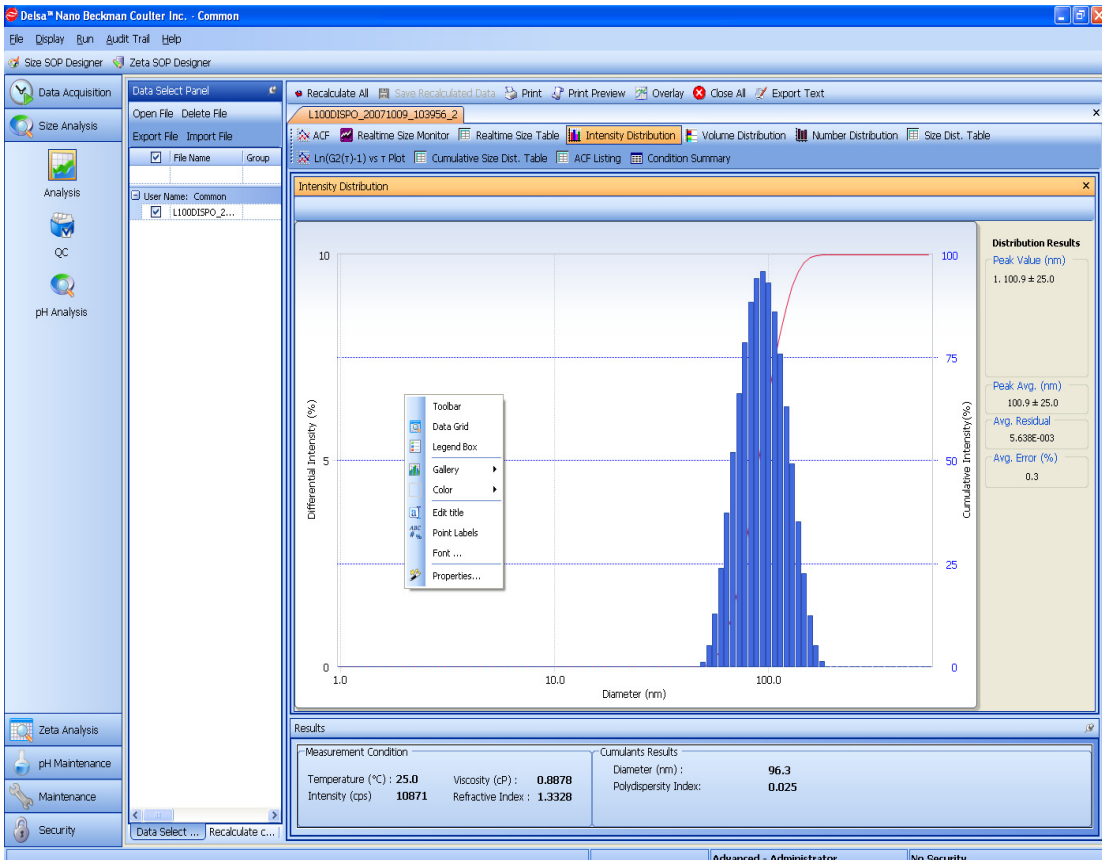
To adjust features on the X and Y axes, place the cursor on the desired axis, and right-click to open the menu (Figure D.26).

Figure D.26 Y Axis Pop-Up Menu



To add or edit features in the graph, place the cursor in the graph, and right-click to open the Toolbar pop-up menu (Figure D.27).

Figure D.27 Graph Toolbar Pop-Up Menu



APPENDIX E

Upgrading the DelsaNano Software

Introduction

This appendix describes how to install the DelsaNano software to upgrade to a newer version or to reinstall the software. You must be an Administrator.

Upgrading or Reinstalling the DelsaNano Software

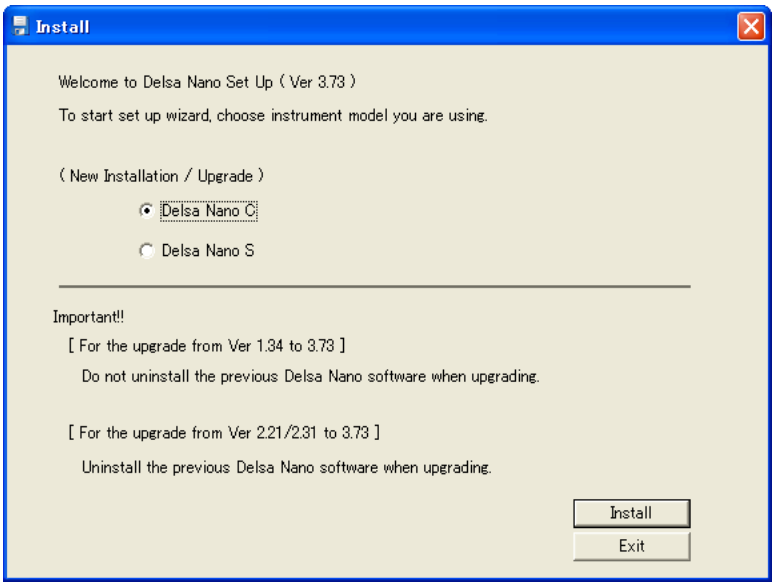
The instructions in this sections are provided in case you need to reinstall or upgrade the software.

⚠ CAUTION

The data files appear in the software under Data Select Panel as long as the software is installed in the same original folder, however, the system parameters are deleted. It is also very important to backup the data and update the system parameters after upgrading or installing the DelsaNano software.

1 Insert the DelsaNano software CD into the CD/DVD drive of your computer. The Install window opens.

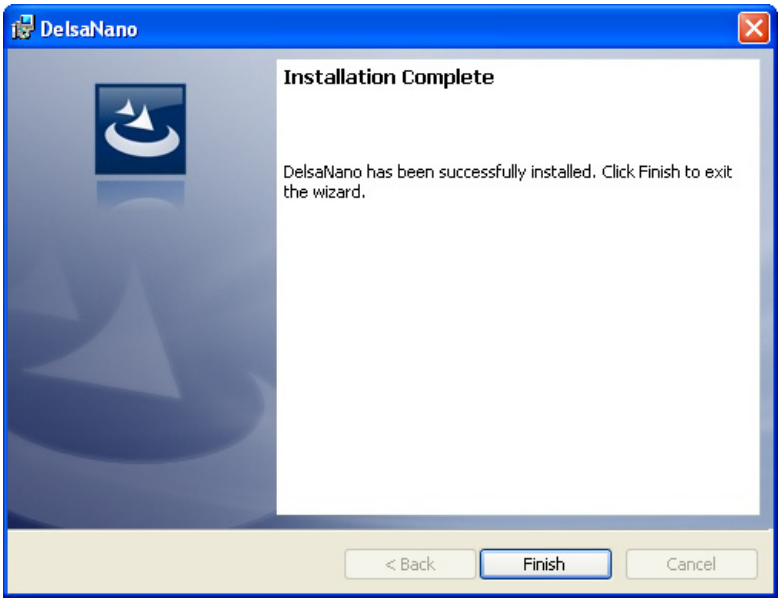
Figure E.1 Install Window



2 If you are reinstalling/upgrading the software, select the appropriate DelsaNano model under the New Installation/Upgrade option, and click **Install**. The software is installed in the selected folder on the computer. The installation progress appears.

When the DelsaNano software has been installed successfully, a confirmation message appears (Figure E.2).

Figure E.2 Installation Complete Window



3 Click **Finish** to close the window.

4 From Windows, double-click the DelsaNano icon on the computer desktop, or select the program from your program list. If you are upgrading to a newer version, a message appears briefly, and then the upgrade commences.

During upgrade, the progress appears. When the upgrade is finished, the software is ready to use, and the DelsaNano Main screen appears.

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