

SPECTREX

CORPORATION

493 Seaport Court, Suite 105 • Redwood City, CA 94063

OPERATING MANUAL

SPECTREX LASER PARTICLE COUNTER MODEL PC-2300

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For full operation of the Spectrex particle counting system (Model PC-2300 + computer) in conjunction with the SuperCount™ Software Manual.

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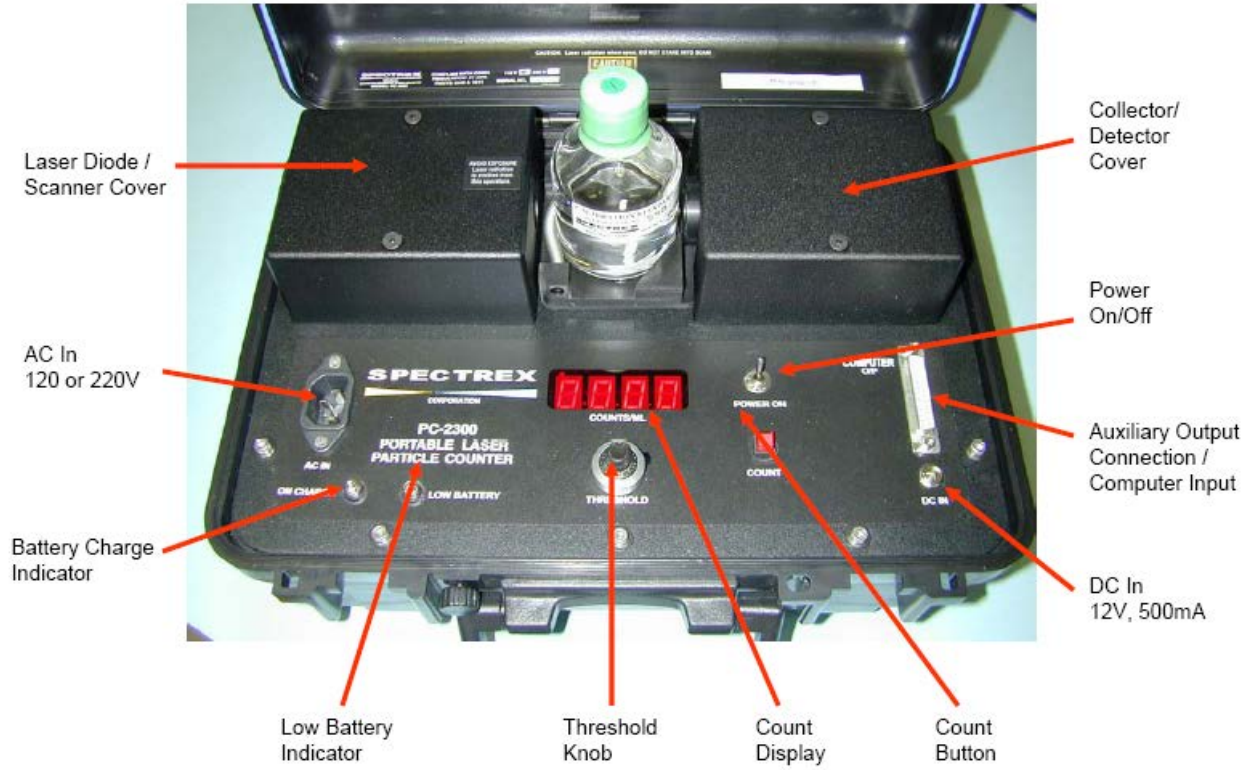
The Spectrex PC-2300, In-Situ Particle Counter, is a unique and easy-to-use instrument designed to permit thorough inspection of liquids without consuming the sample. It is ideal when critically clean liquids are needed. A laser beam permits observation of small particles by near-forward light scatter. In addition, a scanning and detection system is provided to automatically quantify the number of small particles in one cc (milliliter) of the contained liquid.

Sealed calibration standards are provided so that quality control levels may be established against which individual samples may be compared. An analog output is provided for computer interfacing to give automatic particle sizing and counting results.

A specially designed fixture, the "V" Block, automatically holds in correct alignment any standard 150 mL beaker or calibration standard. The spring automatically pushes the bottle up into the "V" position. It is easily removed (by unscrewing two 6/32 screws) for small vial or flow-thru cell attachment.

The Model PC-2300 Particle Counter provides all of the facilities needed for rapid inspection of bottled and flowing liquids. Small particulate matter is automatically quantified by a scanning laser beam and the results presented on a digital display. A minimum size selector is provided for this quantification measurement and is normally set to 1 μ m.

The direct hookup to a computer with interface card, cable and software (provided by Spectrex) give full, automatic sizing and counting of particles. The PC-2300 is ruggedly constructed with all the optical members mounted on one solid aluminum base plate. Thus, the instrument should never go out of alignment.



SPECIFICATIONS

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Minimum Detectable Particle Diameter	Continuously adjustable through two ranges from 0.5 μm to 100 μm .
Volume Scanned in 16 seconds	10 mL (10cm ³)
Read out	Counts/mL
Bottle Material	Transparent glass (Note: unscratched Polystyrene can be used for Hydrofluoric Acid)
Maximum Bottle Wall Thickness	5mm
Display	4 digits
Outputs	Connection provided for external computer
Warranty	One year parts and labor
Power	115/230 volts 50/60 Hz
Size	6" x 9.5" x 14.5"
Weight	15lbs.

This instrument uses as its basic light source a laser diode (650nm wavelength). The beam from this laser is spatially filtered and focused by a lens assembly to form a small and well-defined illuminated volume within the liquid being inspected. A scanning mechanism provides a circular displacement of this illuminated volume at a constant rate of speed.

As the illuminated volume moves across a particle suspended in the liquid, some light from the beam will be scattered. This is known as Fraunhofer diffraction. Most of this scattered light is in the near-forward direction and is collected by the optical system of the photodetector assembly. The flash of light striking the photodetector will cause an electrical pulse in the preamplifier connected to the photodetector. The amplitude and width of this pulse is a function of the size of the particles.

The optical collection system which is a part of the photodetector assembly is designed to provide a definite depth-of-focus. This zone which is in focus ranges from approximately 1.5 cm to approximately 3.5 cm from the black target and lens assembly. A pulse caused by an out-of-focus particle will be broader than a pulse caused by an in-focus particle. The electronic circuits are designed to count only the narrow pulses caused by in-focus particles.

If the illuminating beam does not sweep directly across a particle, the resulting light flash will have two characteristics. First, the amount of scattered light will be less than expected for that size of particle. Second, the duration of the light flash will be less than normal. The electronic circuits to identify such degraded flashes use this second characteristic, and gates are operated to reject them. The displayed count includes only those particles which have been properly illuminated and can therefore be sized correctly.

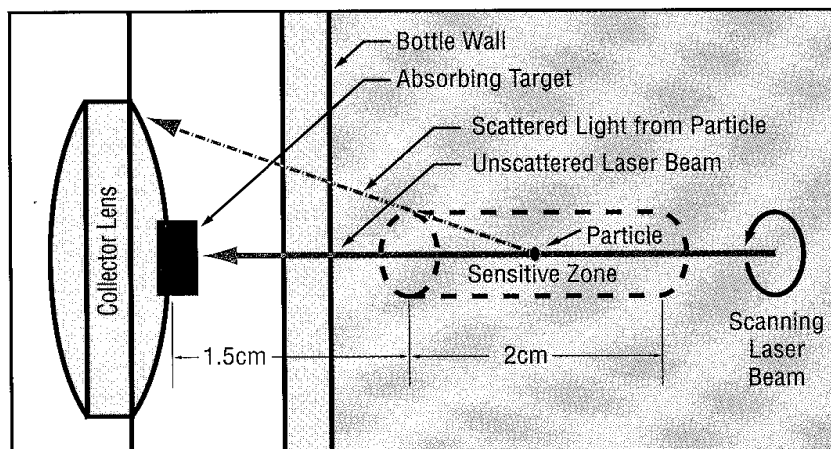


Fig. 1 Laser Beam Optics

As the illuminating beam sweeps through the liquid at a constant rate of speed, a definite period of time will correspond to a definite scanned volume. An electronic timer is provided to give an exact period and this is factory preset to give an output count in counts/mL.

The amount of light scattered by a particle in the sensitive zone of the optical system is a function of the scattering angle and the relative index of refraction of the particle. This instrument collects and averages light that has been scattered in a near-forward direction over a solid angle ranging from 4° to 19°. As particles may be at any location within the depth of field, a variation of collected light of approximately $\pm 15\%$ is to be expected on a single reading of one particle count. By averaging successive readings, the effect of these variations in sizing measurements can be minimized.

In-situ measurements are based upon a very brief illumination of each individual particle by the laser beam. The size of each particle is determined by the light scattered by the particle and the fraction of that light which reaches the photodetector. Individual measurements will vary around an average value as a result of optical and electronic noise, particle orientation, and particle position in the field of view. The greater the number of individual measurements which are averaged, the closer the average will be to the true value. Because the in-situ measurement is nondestructive, it is possible to repeat measurements any number of times so as to increase the accuracy of any test procedure.

All the particulate size measurements are based on the amount of scattered light reaching the collection system. Anything that attenuates the light will have some effect on the calibration. For most colorless liquids and clear bottles, the optical attenuation (opacity) is so small that it can be neglected. For colored liquids or bottles, the opacity should be checked with the opacity meter (available as an optional attachment).

Imperfections on glass bottles and/or dirt on the bottle exterior will also attenuate light to some degree and may influence the calibration. This can easily be checked by rotating the bottle so that the laser beam enters and leaves through a clean area. Wipe the bottle carefully with a lint-free cloth to remove any smudges and fingerprints. If the bottle is properly located on the "V" block, the walls of the bottle will be out-of-focus.

Ambient Light - The optical collection system collects the light pulses from the particulate matter suspended in the liquid. Light from other sources can possibly interfere if a sufficient quantity enters the collection system. Normal levels of incandescent illumination will not usually provide significant interference but high levels of illumination may cause trouble and should be checked. Fluorescent light, because of its high flicker content, are especially troublesome in this regard and should be avoided. A light-tight cover is provided to eliminate fluorescent light effects and is strongly recommended.

Beam Absorption - The projected laser beam can be absorbed or scattered by the container wall or the liquid under analysis. For most clean container walls and most relatively clear liquids, this absorption is negligibly small and will not affect the instrument calibration. If the liquid is optically absorbing or contains very fine particles (typically $< 1 \mu\text{m}$ in diameter) the amount of light scattered into the photodetector will be reduced. When measurements are made with such a liquid, the opacity meter is used to give precise opacity and dilution down to 10% opacity is recommended in order to detect particles down to $1\mu\text{m}$ in diameter.

Optical and Electrical Background - When viewing particles with an optical microscope, it is necessary to clearly distinguish particles from the background in order to get an accurate count. With the Spectrex PC-2300, it is similarly necessary that the instrument be able to clearly distinguish particles from the background in order that they are counted accurately. The background in this case consists of both optical and electronic noise and is referred to as the "background level".

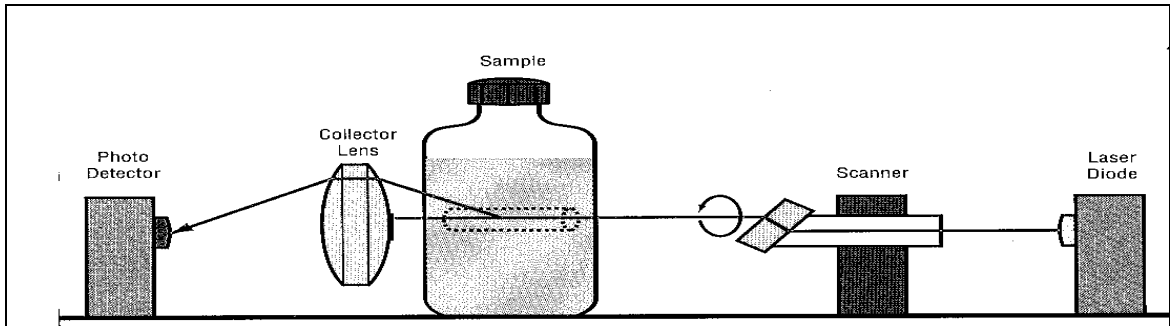


Fig. 2: Optical Schematic of the Laser Particle Counter

The PC-2300 is designed to be mounted on a bench or worktable at a height convenient to the operator. The instrument should be level and firmly seated on a rigid surface. It should not move or vibrate when a sample bottle is placed on its upper surface. It should also not be subject to mechanical vibration due to nearby machinery or processing equipment.

Auxiliary Output Connection – This is located on the front of the instrument. The DB 25 multi-pin connector interfaces the PC-2300 with any Windows based computer. Spectrex Corporation provides “SuperCount” software and an interface control board to fully automate the system.

SuperCount™ Software – Refer to the separate manual for information including the installation procedure.

Setting the Threshold Dial – The threshold knob located on the front of the instrument is set to 10 (“0” in the window and 10 marks on the circular dial) for a 1 μ m level.

Count Controls - Three calibration standards containing polystyrene spheres in water. The specially formulated diluent is miscible and creates a very stable suspension with slow settling characteristics. They provide a means of verifying that the instrument is functioning properly.

- Red capped bottle: This is the 4-5 μ m count control. It has a nominal concentration of 1000 particles/mL with a threshold setting corresponding to 1 μ m.
- Green capped bottle: This is the 1-2 μ m count control. It has a concentration of 300 - 500 particles/mL with a threshold setting corresponding to 1 μ m.
- White capped bottle: This is the “ultra clean” standard or blank. It gives average counts < 50 particles/mL with a threshold setting corresponding to 1 μ m.

The average of ten counts for any calibration standard should be within $\pm 15\%$ of the number stated on the bottle. This indicates that the particle counter is calibrated correctly.

Procedure:

1. Begin by choosing one of the three calibration standards. The Green and Red calibration standards should be resuspended before use by inverting and swirling gently 10 times. The White calibration standard should never be shaken.
2. Place the bottle firmly on the “V” block. Allow 1 minute for the red and green bottles to de-gas as bubbles may appear as statistical artifacts. Do not sonicate. Make sure the threshold knob is set to 10 (“0” in the window and 10 marks on the circular dial).
3. Press the Count button and wait for the red indicator light to go out (approximately 16 seconds). The number of particles/cc above the size selected will show in the display window and should match the number stated on the bottle to $\pm 15\%$.
4. Repeat until all three calibration standards have been tested.

NOTE: In accordance with the laws of sampling statistics, repeated counts will almost certainly not show the exact same number. This holds even when the particles are perfectly uniformly distributed throughout the container. When each subsequent count is taken, motion of the liquid and the particles will present another sample to the sensitive zone of the instrument. The count will, therefore, vary slightly as the sample changes.

Select a Container for the Sample - A clean, high quality glass container such as a 120 mL beaker (Grade A quality) or extruded glass bottle of no less than 2 ¼" in diameter should be used. The quality can be checked by holding it up to the light. If it has the smooth appearance of a sheet of window glass, it is suitable. If it has many striations parallel to its base, it should be rejected. (Spectrex has high quality beaker sets and glass bottles that may be purchased.)

Simple Five Step Counting Procedure (*w/o computer attachment):

**Refer to the SuperCount™ Software Manual supplement for procedure w/ computer attachment.*

1. Turn on the Particle Counter. A laser beam will appear from the projection box toward the front of the instrument. Use the light-tight cover to protect from fluorescent light.
2. View the red circle, approximately 3 mm in diameter, projected in the center of the black target in the middle of the collector lens.
3. Ensure the threshold setting is at the 1µm level. This is normally either 10 or 11 on the threshold knob dial.
4. Resuspend the sample by swirling/stirring to ensure a homogenous suspension. Place the sample firmly onto "V" block. The container should be extremely clean, with no smudges, fingerprints, or dust on the glass wall.
5. Press the Count button. The readout in the display window, when the counting has stopped, indicates the number of particles per cubic centimeter (milliliter) above 1 µm.

YOU ARE NOW READY TO RUN THE PARTICLE COUNTER WITH THE COMPUTER. FOLLOW THE INSTRUCTIONS IN THE SOFTWARE MANUAL.

CALIBRATION CHANGE - The optical elements may collect a film of dirt from the atmosphere, and the exposed optical elements may be splashed with liquids. In both of these cases, the optical efficiency of the unit will be reduced, and counts may change if a large reduction in optical efficiency occurs. Such a change in counts can be detected by a careful test with the calibration standards.

If such a test does indicate a count change, the collector lens can be cleaned. This can be done using lens cleaning tissue such as is used for eyeglasses. When cleaning the lens surface, be careful not to disturb the black light absorbing target in the center of the lens.

LASER NO LONGER ROTATING

1. Remove the black laser/scanner cover at the rear by unscrewing the two black, flat-head Allen screws.
2. Check that the drive belt has not fallen off the two pulley wheels. If so, refit to pulleys.
3. Check that the motor is rotating. If not, contact Spectrex for fixing.

WHITE STANDARD COUNTS TOO HIGH

1. Let stand for 12 hours.
2. Slowly rotate bottle while counting to find position for minimum counts. Repeat count in this position.

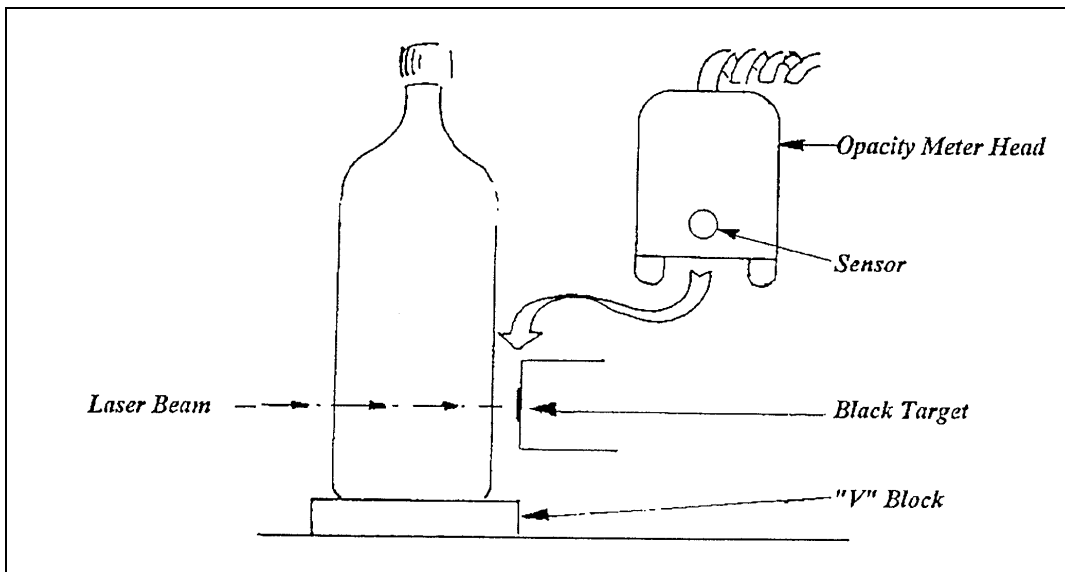
GREEN OR RED STANDARDS COUNTS TOO LOW - Sonicate for 5 seconds (to break up agglomerates).

GREEN OR RED STANDARDS COUNTS TOO HIGH - Let stand for 24 hours. Swirl 20 times (only).

1. OPACITY METER This provides a rapid means of measuring the opacity of a sample. Approximately 30% opacity will set the lower limit of sensitivity to 5 μm instead of the standard 1 μm . Dilution down to 10% opacity is needed to measure particles down to 1 μm (ISO-4406) or 4 μm (ISO-11171).

Installation:

1. Slide Opacity Meter “Head” in front of the receptor lens with the black target. The circular sensor aperture should be facing the front of the instrument, and the red laser beam should hit it fully.



2. With the dial knob, set the indicator on the Opacity Meter to “CAL”.
3. Place the beaker or bottle containing the liquid to be tested into the “V” Block.
4. Note where the indicator on the Opacity Meter dial points. This is the opacity of the sample.

2. **SMALL VIAL ATTACHMENT** This permits the counting and sizing of particles in small vials and ampules down to a 0.8 inch diameter. It is easy to fit and has a separate instruction manual.

Installation:

1. Remove the Light Tight Cover.
 2. Remove the "V" Block by unscrewing the two (2) Philips-headed screws holding it to the base plate.
 3. Fit the Collector Lens Assembly over the lens with the black target in its center. Rotate the assembly until the red laser circle is closest to the center of the black target. Tighten it with the Hex Driver provided.
 4. Replace the "V" block with the Small Vial Stand provided. Do a final check that the red laser circle is close to the center of the black target.
 5. Replace the Light Tight Cover. The Small Vial Attachment is now ready for use.
3. **FLOW-THRU CELL** This attachment provides monitoring continuous flow of liquids. When the Spectracount Software is used, the **Automode Function** gives continuous automatic monitoring. Two different models are available from Spectrex, a short path-length design for high concentration liquids and a long path-length design for low concentration liquids. (The long path-length model is the one normally supplied by Spectrex.)

Installation:

1. Hook up the flow-through cell to your flow system and check for any leaks. If so, then tighten the sealing rings on each end. Remove any air bubbles in the cell by undoing the screw on the top of the cell and letting them escape.
2. Thoroughly inspect the windows of the cell to make sure that there are no fingerprints or other contaminants on the cell windows.
3. Using a Philips screwdriver, remove the two screws that hold the V block which locates the standard bottle or beaker on the main plate of the particle counter.

4. Place the black anodized flow-through cell on the main plate, in the same location as the V-block, with the end closest to the cell mounting bracket as close as possible to the collector lens.
Note: *the collector lens is the one with a black target in its center.*
5. Secure the cell on the main plate with the screws supplied.
6. Start the liquid to be tested flowing through the cell and double check for any leaks.
7. Preferably set the flow of the liquid to 100 ml/min, with a flow control downstream from the cell. This avoids air bubbles forming in the cell.
8. Set the software on the computer connected to the particle counter to Automode and follow detailed instructions.
9. Contact Spectrex to obtain Spectracount™ software for Automode operation to continuously monitor flowing samples over time.

As the basic circuit of the PC-2300 is solid state, very little maintenance should be required to keep it in top operating condition. The optical elements may collect a film of dirt from the atmosphere, and the exposed optical elements may be splashed with liquids. In both of these cases, the optical efficiency of the unit will be reduced and calibration may change if a large reduction in optical efficiency occurs. Such a change in calibration can be detected by a careful test with the calibration standards.

If the laser diode fails, it is recommended that the unit be returned to the factory or that the replacement be done (if necessary) by a factory trained service technician. However, laser diodes have an incredibly long life and should not need replacement for over 15 years.

- * Quality control of hydraulic fluids and oils.
- * De-ionized water and acid testing for semiconductor manufacturing.
- * Vial and ampule inspection for pharmaceuticals.
- * Silt and sediment sizing.
- * Oceanographic particles.
- * Sizing for corrosive chemicals and solvents.
- * Cell counting where physical force would damage particles.
- * Particle agglomeration studies.
- * Water treatment plants.
- * Filter efficiency control.
- * Powdered solids manufacturing.
- * Quality control for solvents for liquid chromatography.

During the first year of service, our guarantee will include replacement parts and labor of any faulty or failed components not caused by misuse at no extra cost to the user. After the first year, any factory repairs or calibration will be done for a nominal charge.

The software program for dilution (2nd icon from the right on the SuperCount homepage) makes it extremely easy to enter dilution factors. See manual for more details.

ONE STEP DILUTION

Since the path length of the laser through the sample is at least 2 inches, the sample may need to be diluted in order to avoid coincident counts. Ideally, one should aim at < 1,000 particles per mL. A fairly simple procedure is outlined here.

Pour the raw sample into a 140 mL beaker and place on the particle counter. If the counts exceed 800-900 particles/mL at 1 μm , use the following dilution procedure:

STEP 1 Fill a clean 140mL beaker with 100mL of clean diluent and take a count. There should not be more than 30 particles/mL greater than 1 μm .

STEP 2 Thoroughly redisperse the sample.

STEP 3 Draw 1 mL of sample by glass pipette and begin adding a controlled proportion of sample to the diluent. Stir carefully with clean spatula or magnetic stir bar.

STEP 4 Take a count. If a 0.5 mL addition takes the counts above 1,000/mL we suggest you use the two step dilution procedure (below).

STEP 5 If Step 3 is done carefully, the counts should increase by 100-200 particles/mL. Add more sample until total counts are between 500-600 particles/mL. This is a good place to stop and indicates that you are safely below the “1,000 count”, coincident count state.

STEP 6 Calculate the dilution ratio as follows: (assuming you start with 100mL diluent).

If 1mL of sample is added, dilution ratio is $\frac{100}{1} = 100:1$ (Dilution factor is 101)

If 0.1mL of sample is added, dilution ratio is $\frac{100}{0.1} = 1000:1$ (Dilution factor is 1,001)

If 5mL of sample are added, dilution ratio is $\frac{100}{5} = 20:1$ (Dilution factor is 21)

The equation is very simple:

$$\text{Dilution Ratio Factor} = \frac{\# \text{ of mLs of diluent}}{\# \text{ of mLs of sample added}}$$

Then add 1 to get the Dilution Factor

DOUBLE DILUTION

For high-concentration samples, this procedure will ensure that a large enough, representative sample is taken to provide a realistic size and count analysis.

STEP 1 Start with 99 mL of particle free diluent in the beaker. (Note: it greatly simplifies calculations by using 99 instead of 100 mL).

STEP 2 Add 1 mL of sample and thoroughly mix. Call this Sample "A".
(Note: Dilution factor is 100:1).

STEP 3 Place another beaker of 99 mL of diluent on the particle counter and check that it is clean, with less than 30 counts >1 μm

STEP 4 Add 1 mL of Sample "A" and stir.

STEP 5 Continue to add precise amounts until total counts >1 μm are 5-600/mL

STEP 6 Calculate dilution ratio as follows (for Sample "A"):

First dilution

$$\text{Dilution ratio is } \frac{99+1}{1} \quad (\text{Dilution factor is 100:1 for Sample "A"})$$

Final dilution

$$\frac{100 \times 100}{\text{Number of mLs added in Step 5}}$$

Example:

If 1 mL of "A" is added in second dilution state, the dilution factor is

$$\frac{100 \times 100}{1} = 10,000$$

If 2 mL of "A" are added, the dilution factor is

$$\frac{100 \times 100}{2} = 5,000$$