SPECTREX

CORPORATION

INSTRUCTION MANUAL

SPECTREX LASER PARTICLE COUNTER MODEL PC-2000

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SPECTREX

CORPORATION

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1. Switch on **Particle Counter** (switch is located on the rear). 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. Press **Scanner** button to start scanner motor. This makes the laser beam rotate around its own axis. (This will probably already be turned on.)

3. Shake or stir container to ensure even particle distribution. Place **sample** firmly onto "V" block. Ensure that the container is extremely clean, with no smudges, fingerprints, or dust on the glass wall.

4. Set the **Threshold** setting to the 1µm level. This is normally either 10 or 11 on the threshold knob dial.

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 the size level specified in step #4 above.

IMPORTANT: If the counts go over 1,000/cc, then DILUTE the sample. "Coincident Counting", when the laser beam hits two particles at the same time, occurs at this concentration level. For the laser to hit one particle at a time, the count should be less than 1,000/cc.

Summary of Rapid Calibration with Sealed Standards

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Three calibrated suspension bottles containing polystyrene spheres in an alcohol/freon mix are provided with the Spectrex Particle Counter. This makes a very stable suspension with slow settling characteristics.

Red capped bottle:	Gives average count as stated on the label: with threshold setting corresponding to 1µm.
Green capped bottle:	Gives average count as stated on the label: find threshold setting corresponding to 1 µm that gives the number of Particles per ml marked on the bottle.
White capped bottle:	Gives average count less than 20 with threshold setting corresponding to 1 µm. This is the "ultra-clean" standard.

The suspensions provide a means of checking that the instrument is functioning properly. When the bottle with the WHITE CAP is placed on the "V" block, and the threshold setting is set for 1 µm, there should be less than 25-30 counts recorded.

Testing a bottle

1. Vigorously agitate the RED and/or GREEN capped bottles for approximately 15 seconds to produce a uniform suspension. (DO NOT SHAKE THE WHITE CAPPED BOTTLE!).
2. Place a bottle firmly on the "V" block and allow 15 seconds for air bubbles to disappear. Turn the dial to the proper threshold setting for the particle size to be checked, in most cases, 1 µm.
3. Press the **Count** button and wait for the red indicator light to go out (approximately 15 seconds). The number of particles per cc above the size selected will show in the display window and should match the number on the bottle to plus or minus 15%.

NOTE: In accordance with the laws of Sampling Statistics, repeated counts will most likely not show the exact same number. This holds, even when the particles are uniformly distributed throughout the container. When subsequent counts are take, motion of the liquid and the particles will present another sample to the sensitive zone of the instrument. The count will, therefore, vary as the sample changes.

For any particular calibration suspension bottle, the average of ten counts should be within plus or minus 15 % of the number written on the bottle. This indicates that the particles counter is calibrated correctly.

INSTRUMENT DESCRIPTION 3

The Model PC-2000 In-Situ Particle Counter is designed to permit thorough inspection of bottled liquids without breaking any seal or removing any of the contained liquid. 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.

A variable threshold is 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.

The instrument is typically used where critically clean liquids are needed. The bottle to be inspected is vigorously shaken to suspend any particulate material and is placed on the "V" block on the top of the unit. By pressing the **Count** button, a scan cycle can be initiated. During this cycle, the scanning laser beam counts all particles greater than the set **Threshold**. When a total volume of one milliliter has been scanned, the counter automatically stops and switches to hold the count.

The THRESHOLD setting may be calibrated in terms of equivalent optical diameter by direct comparison with known particles. By making a series of Count Measurements with different Threshold settings, it is possible to study the particle size distribution in liquid. *The computer attachment SPC-510 provides this particle distribution automatically.*

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). If you find it over 10%, the opacity lines on an optional calibration chart should be used.

Imperfections of the glass bottles and/or dirt on the outside of the bottles 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 and checking the count. The red circle caused where the laser enters the bottle should be as faint as possible. Wipe the bottle carefully 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.

The laser inspection technique is very sensitive and permits observation of very small particles. More than 1,000 particles being counted in one milliliter will cause coincident counts, where more than one particle is scanned at any one time. This will result in counting errors and should be avoided. Dilute sample with clean, particle-free diluent.

THE "V" BLOCK: This specially designed Delrin fixture is easily removed (by unscrewing (2) 8/32 screws) and will automatically hold in correct alignment any standard 150 ml beaker or calibration standard. The spring automatically pushes the bottle up into the "V" position.

* 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.

This instrument uses as its basic light source a laser diode (wavelength 670.8 nm). 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 Fraunhoffer diffraction. Much 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. The walls of a typical bottle placed on the "V" block on the top of this unit will be outside this zone and will therefore be out-of-focus. 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.*

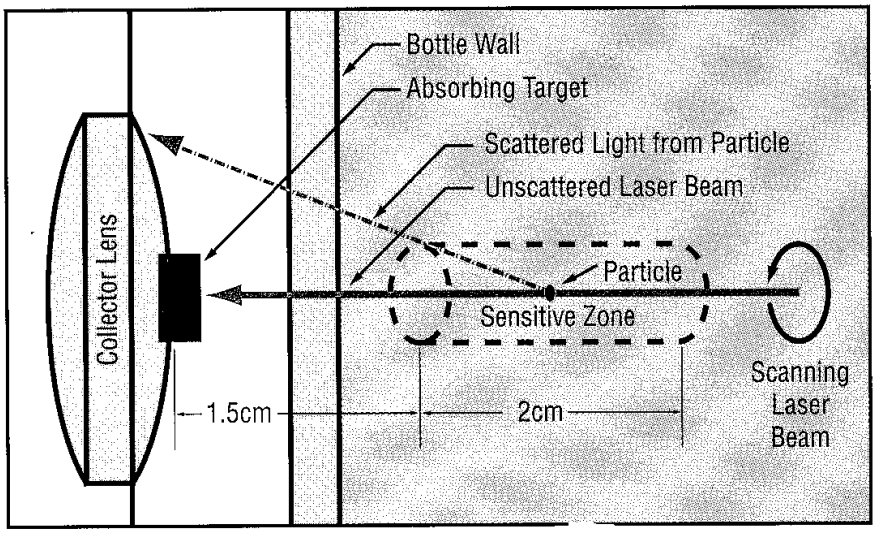

Fig. 1 Laser Beam Optics

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 therefore includes only those particles, which have been properly, illuminated and can therefore be sized correctly.

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 particles per milliliter.

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 plus or minus 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.

The PC-2000 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.

Ambient Light Considerations

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 lights,*** because of their 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.

The instrument should be connected to a suitable electrical outlet and turned on with the switch on the rear panel. The laser beam can be seen to come from the projection box and strike the black target on the receptor lens.

Optical Alignment

The PC-2000 is ruggedly constructed, with all the optical members mounted on one solid, aluminum base plate. Thus, it should never go out of alignment.

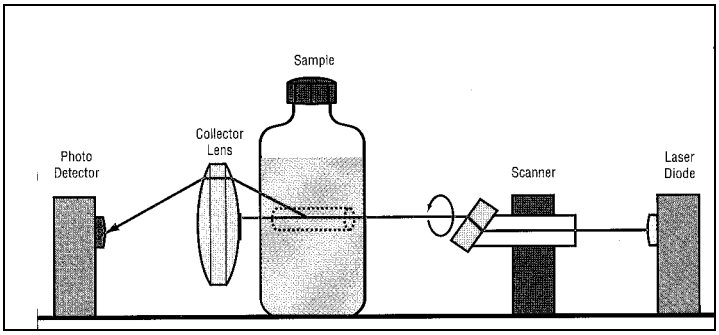

Fig. 2: Optical Schematic of the Laser Particle Counter

Checking Calibration of the Instrument

Three calibration standards in sealed bottles are provided with each PC-2000 Laser Particles Counter. Each standard contains 200 ml of **alcohol** and **freon** proportioned to each other to match the specific gravity of added polystyrene spheres. The liquid has been filtered through a 0.2 μm filter.

Standard #1 is a GREEN capped bottle and contains a precisely known number of suspended polystyrene spheres greater than 1 μm dia. Standard #2 is a RED capped bottle and contains a precisely known number of 5 μm dia. Spheres. Standard #3 is ultra-clean, particle free and thus provides a "clean" standard to establish accurate background levels. Each standard is sealed with inert, argon gas.

Stability

The RED and WHITE standards have proven stability of over ten years and the alcohol/freon suspension is miscable and stable.

The GREEN, 1 μm, standard has a one year stability warranty as Spectrex has not had enough time to evaluate its long term stability. This is a very important standard as the sizing "structure" software is built up from the 1 μm level. We suggest you order a replacement twelve months from the date you received your Laser Particle Counter.

Setting the Threshold Dial

After shaking, the GREEN, 1 μm, standard is placed on the particle counter. **Set the Threshold dial to the setting number marked on the bottle.** This then is the setting on the dial to give the total number of particles greater than 1 μm. From time to time, once a month suggested, this setting should be checked.

Three calibrated suspension bottles containing polystyrene spheres in an alcohol/freon mix are provided with the Spectrex Particle Counter. This makes a very stable suspension with slow settling characteristics.

Red capped bottle: threshold	Gives average count as stated on the label: with setting corresponding to 1 µm.
Green capped bottle:	Gives average count as stated on the label: find threshold setting corresponding to 1 µm.
White capped bottle:	Gives average count less than 20 with threshold setting Corresponding to 1 µm. This is the ultra-clean standard.

The suspensions provide a means of checking that the instrument is functioning properly. When the bottle with the WHITE cap is placed on the "V" block and the threshold settling is set for 20 µm, there should be no counts recorded. When the threshold is set for 5 µm, there should be less than 10 counts recorded.

Testing a bottle

1. Vigorously agitate the RED and/or GREEN capped bottles for approximately 15 seconds to produce a uniform suspension (DO NOT SHAKE THE WHITE CAPPED BOTTLE!).
2. Place a bottle firmly on the "V" block and allow 15 seconds for air bubbles to disappear. Turn the dial to the proper threshold setting for the particle size to be checked as marked on the bottle.
3. Press the **Count** button and wait for the red indicator light to go out (approximately 15 seconds). The number of particles per cc above the size selected will show in the display window.

NOTE: In accordance with the laws of Sampling Statistics, repeated counts will most likely not show the exact same number. This holds, even when the particles are uniformly distributed throughout the container. When subsequent counts are taken, the motion of the liquid and the particles will present another sample to the sensitive zone of the instrument. The count will, therefore, vary as the sample changes.

For any particular calibration suspension bottle, the average of ten counts should be within plus or minus 15% of the number written on the bottle. This indicates that the particle counter is calibrated correctly.

A. 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. A poor quality beaker should be avoided. 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 Corporation makes available a high quality Pyrex glass bottle, of the type used for calibration standards and these can be purchased off the shelf.

It is also possible to use flow-through cells. Two different models are available from Spectrex upon request.

Custom cells are also available and Spectrex Corporation has designed one with sapphire windows and nickel alloy walls to withstand extreme temperatures and pressures. However, when there is the possibility that the windows will touch the sensitive zone, it is wise to stop the laser scanning and depend upon the movement of the particles flowing through the cell to generate the near angle light pulses. This flow rate has to be well controlled. Spectrex will help you in the design of specialty cells for your specific needs and has standard flow-through cells available.

B. Count Procedures

Simple Five Step Counting Procedure using the PC-2000 Particle Counter without a computer attachment.

1. Switch on **Particle Counter** (switch is located on the rear). 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. Press **Scanner** button to start scanner motor. This makes the laser beam rotate around its own axis. (This will probably already by turned on.)
3. Shake or stir container to ensure even particle distribution. Place **sample** firmly onto "V" block. Ensure that the container is extremely clean, with no smudges, fingerprints, or dust on the glass wall.
4. Set the **Threshold** setting to the 1µm level. This is normally either 10 or 11 on the threshold levels dial.
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 the size level specified in step #4 above.

When direct comparisons are to be made between in-situ particle counts and manual microscope counting of membrane-deposited particles, the following points should be kept in mind:

Sampling

A single measurement with a PC-2000 Particle Counter will give the number of particles in one milliliter of the sample fluid. Assuming that the sample bottle contains 100 ml, this will provide a one per cent sample. If ten successive readings are taken and average, this will provide a more representative sample as ten per cent of the sample has been viewed.

In a similar fashion, if a membrane count is performed by passing 100 percent of the sample through a filter membrane and microscopically counting one percent of the membrane surface, only one percent of the suspension has been sampled. A more representative sample would result by counting ten percent of the membrane surface for instance.

Because it is not possible to repeat the membrane filtration of a test suspension without introducing additional sampling and contamination errors, it is usually necessary to take the in-situ measurements first and then perform the membrane filtration as a second operation on the same suspension.

Size Determination

When a membrane is selected for the filtration, the pore size should be 1/3 to 1/10 the particle size of interested so that no irregular shaped particles will be lost in the pores, and all significant particles in the population are available for microscopic viewing. A calibrated reticule can then be used to estimate the size of each particle and a decision made to include or exclude the particle in the total count.

The 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.**

Effects of Optical and Electronic 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-2000, it is similarly necessary that the instrument be able to clearly distinguish particles from the background in order that they be counted accurately. The background in this case consists of both optical and electronic components and is referred to as the BACKGROUND LEVEL.

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 particle (typically less then 1 µm in diameter), the amount of light scattered into the photodetector will be reduced and the calibration curve will be shifted accordingly. When measurements are to be made with such a liquid, the opacity meter is used to give precise opacity and then the correct opacity level (arranged as diagonal, parallel lines on a calibration chart) is chosen and used to set the threshold level. This chart is available upon request.

Auxiliary Output Connection

(on the rear of the instrument)

This DB 25 multi-pin connector interfaces the PC-2000 with any windows based computer. Spectrex Corporation provides special "Supercount" software and an interface control board to fully automate the system.

Optional Attachments

1. OPACITY METER: this provides a rapid means of measuring the opacity of a sample. This is important as a correction needs to be made on the threshold dial to compensate for opacity. Approximately 30% opacity will set the lower limit of sensitivity to 5 µm instead of the standard 1 µm. The calibration chart provides the corrected setting.
2. SMALL VIAL ATTACHMENT: This permits the counting and sizing of particles in small vials and ampules down to a 5mm-vial volume. It is easy to fit and has a separate instruction manual.
3. FLOW THROUGH CELL: This attachment provides monitoring continous flow of liquids. When the Windows® Software is used, the **Automode Function** gives continous 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.

As the basis circuit of the PC-2000 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 check suspensions.

If such a test does indicate a calibration change, the external optics should be cleaned. This can be done using lens cleaning tissue such as is used for eyeglasses. *Clean only the exposed optical surfaces, the projector window, and the collector lens surface.* When cleaning the collector lens surface, be careful not to disturb the light absorbing target in the center of the lens.

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 yours should not need replacement for over 15 years.

All maintenance and service question should be directed to the service department.

Call 1-800-822-3940

The Model PC-2000 Particle Counter provides all of the facilities needed for rapid and thorough in-situ 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. By taking sequential measurements, size distribution data can be developed, or the size selector can be locked to provide statistical quality control data. As the bottled liquid is tested in-situ, repeat measurements can be made at any time for certification of observations. Rear-panel output to any Windows® based computer provides complete sizing and storage capabilities.

Minimum Detectable Particle Diameter	Continuously adjustable through two ranges from 0.5 µm to 100 µm.
Volume Scanned	1 cubic centimeter
Bottle Material	Transparent glass (Note: unscratched Polystyrene can be used for Hydrofluoric Acid)
Bottle Size	50 mm dia. To 200 mm dia.
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	7″ x 6″ x 15″
Weight	16lbs.

Patents Awarded

Spectrex has manufactured this instrument with emphasis on first-class quality and careful workmanship. Because of this, you can expect many years of uninterrupted service from this unit.

During the first year of service, our guarantee will include replacement (material and labor) at the factory of any faulty components, 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.

For more information, please contact

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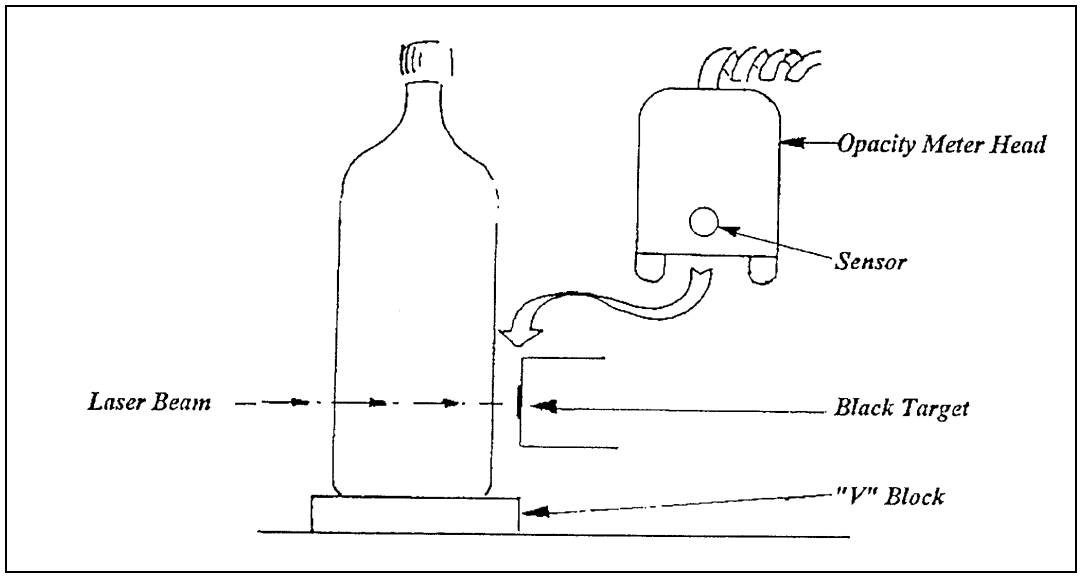
Operating Instructions

Step 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.



Step 2
With the dial knob, set the indicator on the Opacity Meter to "CAL".

Step 3
Place the beaker or bottle containing the liquid to be tested into the "V" Block.

Step 4
Note where indicator on the Opacity Meter dial points. This is the opacity of the sample.

Step 5
Make necessary corrections for Threshold Setting using the calibration chart.

DILUTION PROCEDURES FOR PARTICLE SIZE/COUNT ANALYSIS

Appendix B

Introduction

Because the path length of the laser through the sample is at least 2 inches, the sample may have to be diluted in order to avoid coincident counts. Ideally, one should aim at well less than 1,000 particles per ml. A fairly simple procedure is outlined here which makes dilution easy.

Pour the raw sample into a 140 ml beaker and place on the particle counter. If the counts exceed 8-900 at 1 µm, use the following dilution procedure:

STEP 1 Fill a clean 140 ml beaker with 100 ml of clean diluent and take a count. There should not be more than 50 particles greater than 1 µm per ml.

STEP 2 Thoroughly shake sample (sonicate to break up agglomerates if necessary)

STEP 3 Draw 1 cc 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 ½ ml addition takes the counts above 1,000 we suggest you use "double dilution". This procedure is outlined later.

STEP 5 If Step 3 is done carefully, the counts should come up by 100-200. Add more sample until total counts are 5-600. This is a good place to stop and indicates that you are safely away from the "1,000 count", coincident count state.

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

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

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

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

The equation is very simple:

Dilution Ratio = $\frac{\text{Number of ml diluent}}{\text{No. of ml sample added}}$ Then add 1 to get Dilution Factor

DOUBLE DILUTION PROCEDURES

Appendix C

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 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 50 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
Dilution ratio is $\frac{99:1}{1}$ (Dilution factor is 100:1 for Sample "A")

Final dilution $\frac{100x100}{\text{Number of ml added}}$

Example

If 1 ml of "A" is added in second dilution state, dilution factor is $\frac{100 \times 100 = 10,000}{1}$

If 2 ml of "A" are added, dilution factor would be $\frac{100 \times 100}{2} = 5,000$

IF YOU HAVE ANY QUESTIONS OR NEED ADDITIONAL INFORMATION ABOUT THESE PROCEDURES CALL 1-800-822-3940.

INSTRUCTIONS FOR INSTALLING THE FLOW-THROUGH CELL

Appendix D

Step 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.

Step 2 Thoroughly inspect the windows of the cell to make sure that there are no fingerprints or other contaminates on the cell windows.

Step 3 Remove the light-tight cover from the particle counter.

Step 4 With a Philips driver, remove the two screws that hold the V block which locates the standard bottle or beaker on the main plate of the particle counter.

Step 5 Place the brass 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.

Step 6 Secure the cell on the main plate with the screws supplied.

Step 7 Start the liquid to be tested flowing through the cell and double check for any leaks.

Step 8 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.

Step 9 Set the software on the computer connected to the particle counter to **Automode** and follow detailed instruction