Zetatrac™
Particle Size & ZetaPotential Analyzer

Model NPA152
Operation and Maintenance Manual

OM0013
Rev A
September 2008

Microtrac Inc.
A Unit of Nikkiso

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Revision A – 09/08

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CE **CE Conformity:** This product is in conformance with the protection requirements of the following European Council Directive: 89/336/EEC, the EMC directive, and 73/23/ECC, the Low Voltage Directive. Conformance of this product with any other "CE Mark" directive shall not be assumed.

WARNING

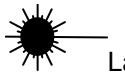
This is a class A product. In a domestic environment this product may cause radio interference in which case the user may be required to take appropriate measures.

SYMBOL DEFINITIONS


This CAUTION symbol on the equipment refers the user to the Product Manual for additional information. This symbol appears next to required information in the manual.


WARNING, risk of electric shock. This symbol warns the user of a potential shock hazard where voltages greater than 30 Vrms, 42.2 Vpeak, or 60 Vdc may be accessible.


Protective earth terminal. Provided for connection of the protective earth (green or green/yellow) supply system conductor.


Laser Radiation. Refer to Product Manual before removing or opening any covers.

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About This Document

Abstract

This manual describes the operation and maintenance of the Microtrac® Zetatrac™ Particle Size And ZetaPotential Analyzer.

Revision Notes

The following list provides notes concerning all revisions of this document.

Rev. ID	Date	Notes
A	09/08	Initial release.

References

Microtrac Documents

The following list identifies all Microtrac documents that may be sources of reference for the material discussed in this publication.

Document Title	ID #
Microtrac FLEX Software Operation Manual	SW0003

Contacts

The following list identifies important contacts within Microtrac.

Organization	Telephone	Address
Microtrac Technical Support	(727) 507-9770 Voice (888) 643-5880	12501-A 62nd Street North Largo, FL 33773

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1. Introduction

1.1 Purpose of the Manual

This manual describes the operation, normal maintenance, and servicing of Microtrac's Zetatrac™ Particle Size And ZetaPotential Analyzer. This manual also includes the following setup and maintenance information:

- Cleaning and routine maintenance
- Problem diagnosis and correction (Troubleshooting)

This manual is meant to be used in conjunction with SW0003, Microtrac FLEX Software Operations Manual. There are frequent references to SW0003 throughout this manual.

1.2 Product Description

The Zetatrac™ is a precise instrument that uses optical techniques, proprietary to Microtrac, to perform both size and zetapotential analysis of particle samples. The Zetatrac™ is an easy-to-use, compact, benchtop unit. It requires a typical PC computer, with data acquisition hardware and Microtrac's FLEX software installed, to complete the analysis task. The Zetatrac™ is a low-power instrument, consuming less than 5W of power under worst-case circumstances.

The industrial design of the Zetatrac™ makes it compatible for laboratory research, production quality control, process monitoring / control, and other applications. Zetatrac™ analysis can be performed on materials with particle size ranging from 0.0008µm (0.8nm) to 6.54 µm.

The Zetatrac™ analyzer is closely related to Microtrac's Nanotrac analyzer, which performs particle size analysis in the range of 0.8nm to 6.54 µm. Zetatrac™ utilizes and builds upon some of the same principles, techniques, hardware, and software as Nanotrac. However, Zetatrac™ provides extra functionality of zetapotential analysis, for those users who desire that feature.

Refer to Figs. 1-1 through 1-3 and the description of functions that follows:

Figure 1-1: Zetatrac™ Analyzer - Front View - Protective Lid Closed

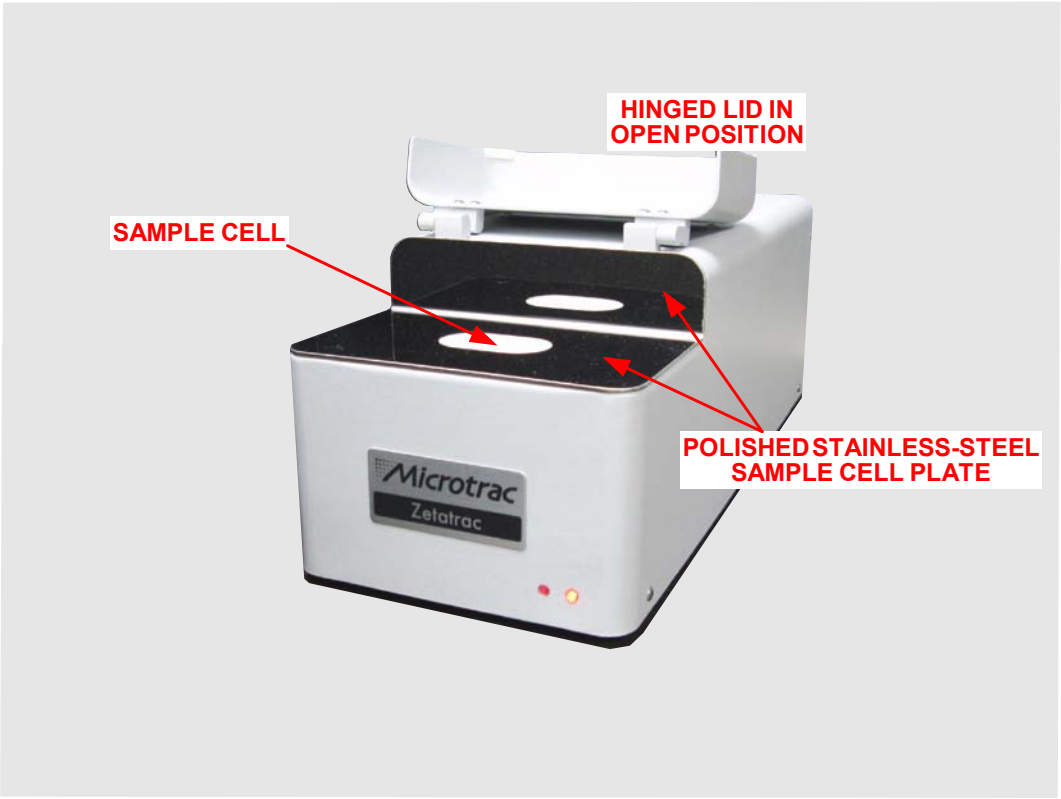


Figure 1-2: Zetatrac™ Analyzer - Front View - Protective Lid Open

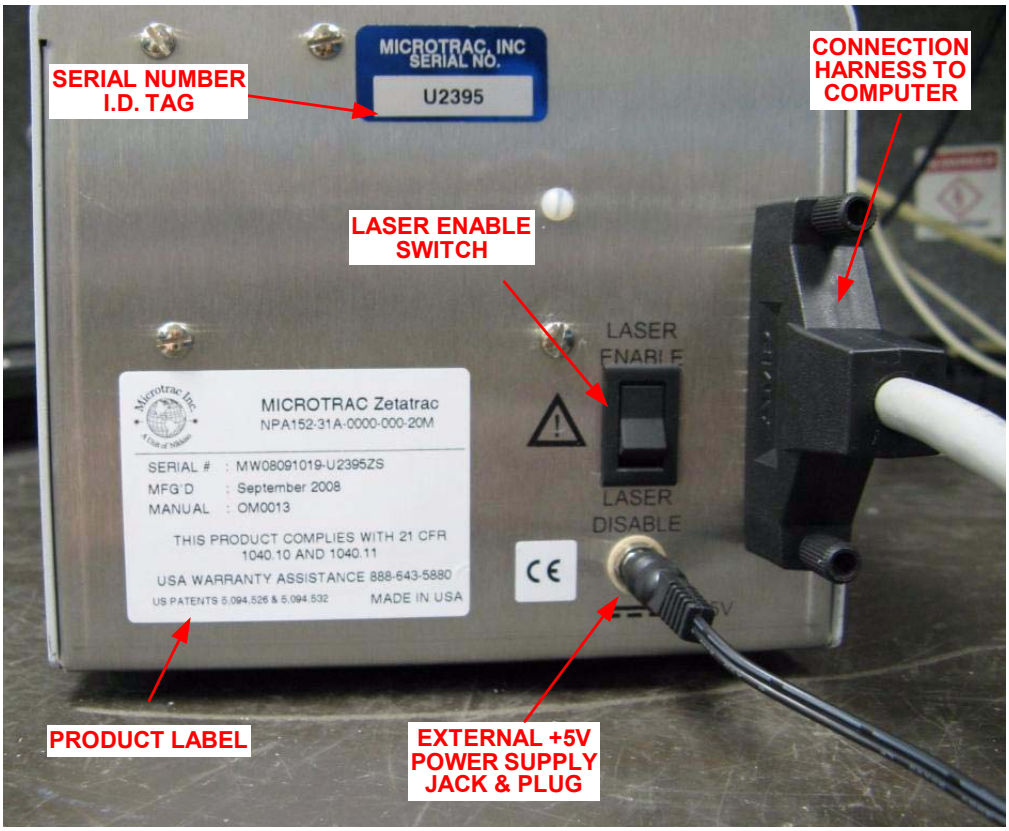


Figure 1-3: Zetatrac™ Analyzer - Rear View

Significant Features:

* Dual optical probe technology for analysis and determination of particle size distribution and electrophoretic mobility (from which zetapotential is ultimately derived). Optical light sources are dual solid-state laser diodes in 780nm (near-infrared) wavelength.
* Indicators mounted on the front surface provide indication of optical system activity.
* Chemical-resistant, easy-to-clean outer finish.
* Solvent-resistant Delrin sample-cell, with hinged lid for protection from sample contamination and for cell protection. Sample material to be analyzed is introduced into the sample-cell, where optical probes interact with the sample to perform the analysis.
* Mirror-polished stainless-steel plate for sample-cell mounting; provides easy cleanup from spills and daily use.
* Simple connections and operation - one connection to external DC power, and one harness to connect to the computer (both are provided with Zetatrac). An on/off switch on rear panel allows laser light-sources to be manually enabled or disabled for safety, maintenance, or diagnostic purposes.
* After the user manually introduces the sample into the sample-cell, the analysis is completely automated through use of Microtrac's FLEX Windows-based software. Full control and adjustment of analysis parameters, as well as selection of multiple data output formats, are available through the FLEX user interface.

The Zetatrac™ Optical Probes and Sample Cell

The Zetatrac™ performs particle size analysis and particle zetapotential analysis by the principle of dynamic light scattering (DLS). The purpose of the optical probe assemblies is to deliver laser-light to the sample, and simultaneously to collect the portion of this light that is scattered back from the sample's particles.

As shown in the following figures, the Zetatrac's two optical probes are horizontally mounted to the sample-cell, flush with the inside surface of the cell. The sample cell is mounted flush with a polished stainless-steel plate, to allow easy cleaning if spills should occur. As shown, sample is introduced into the sample area of the cell. The cutaway view demonstrates the minimum liquid required to perform an analysis - the probe surfaces must be completely submerged. This allows for an absolute minimum of sample to be used per analysis-run. Maximum amount of sample should be limited such that the cell does not overflow.

Also installed horizontally in the sample-cell are two electrodes. These are used during the zetapotential portion of the analysis. Note that the electrodes are paired with the optical probes, and note the gap between the probes and the electrodes. This configuration is crucial to proper zetapotential analysis operation.

The sample fluid temperature is measured by means of a thermistor, which is integrally mounted in one of the optical probe assemblies. This allows measurement of sample temperature with an accuracy of +/- 0.1ºC.

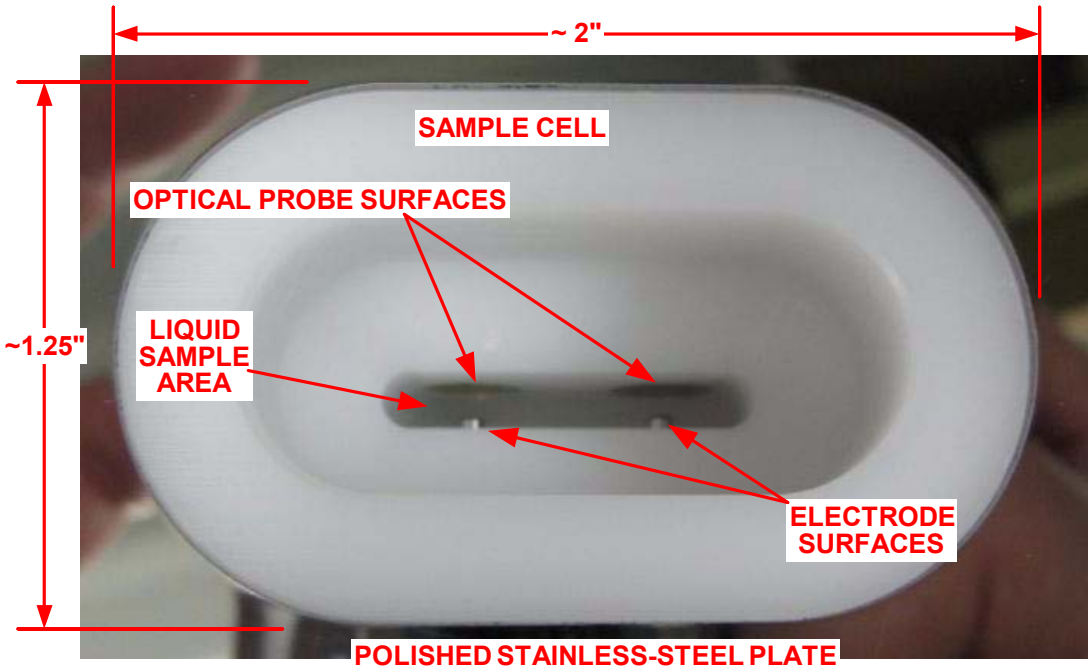


Figure 1-4: Magnified View Of Sample Cell, From Above

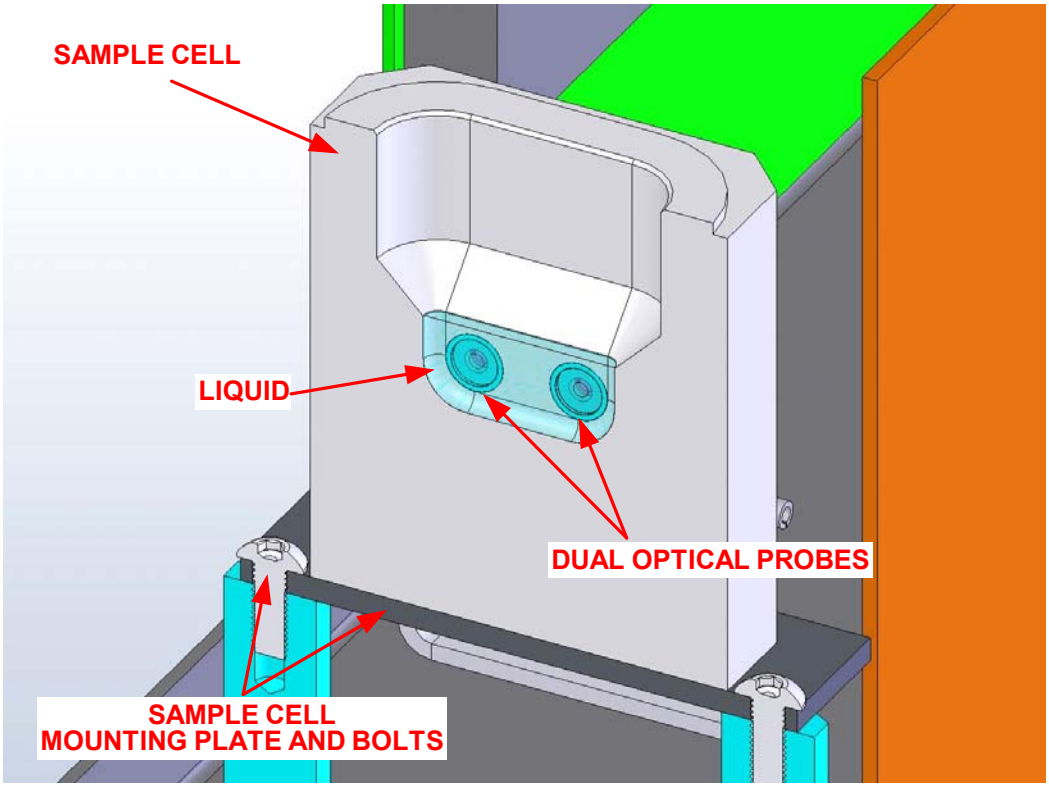


Figure 1-5: Cutaway View of Sample-Cell

Other Optical Components

Zetatrac optical system components and electronics components are connected together approximately as shown by the following diagram.

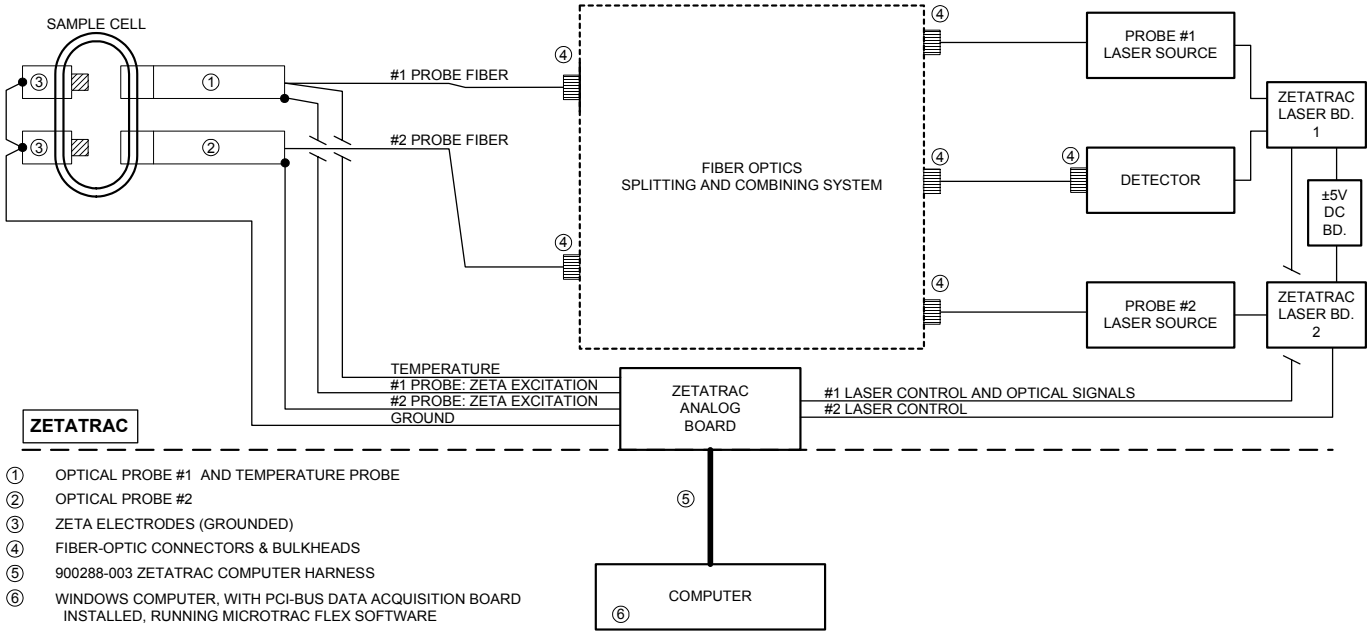


Figure 1-6: Zetatrac Schematic Diagram

1.3 Safety Information, Cautions, and Warnings

WARNING

Do not tamper with or attempt to defeat any safety feature. Use of controls or adjustments, or performance of procedures other than those specified by the manufacturer, may result in hazardous radiation exposure.

The Zetatrac contains no user serviceable parts. Zetatrac service or repair should be coordinated by contacting Microtrac Technical Support.

Electrical Safety

The Zetatrac's external AC-to-DC +5V power supply, provided with the system, must be plugged into AC power mains with an earth-grounded safety terminal.

WARNING

Use only AC Mains power system with protective-earth ground terminal. Never operate the unit from a power source that does not have a protective-earth terminal. Never attempt to defeat the protective-earth system of the power source.

Note: There are no high voltages present inside the Zetatrac.

Laser Safety

WARNING

Operation with cover off and disconnection of the optical system will present a laser radiation hazard. Users are not to attempt removal of covers or disassembly of optical components without specific approval from Microtrac. Contact your Microtrac representative if questions arise concerning operation, adjustment, or repair of the Zetatrac.

Laser safety labels are affixed at appropriate locations on the outside and inside surfaces of the Zetatrac™ The Zetatrac™ employs two solid-state diode lasers (Class IIIB) of 780 nanometer wavelength with a nominal optical power level of three milliwatts. When all covers are in place, and provided the Zetatrac™ is operated as stated in this manual, then operators of the Zetatrac™ cannot be exposed to direct laser radiation (Class I).

Zetatrac™ normal operation has one laser on and the other off. LED indicators on the front panel show this. To avoid all possible exposure to laser radiation, flip the Laser Enable/Disable switch to the 'Disable' position. Both optical-system-activity indicators should then be off.

Chemical Compatibility

Refer to the Specifications section for a list of wetted materials and chemical compatibility with those materials.

Questions concerning use of Zetatrac with solvents (organic, polar, non-polar), high-temperature applications, high-concentration acids / bases, etc., should be addressed to Microtrac Technical Support:

Microtrac Technical Support
12501-A 62nd Street North
Largo, FL 33773
(727) 507-9770

When using any volatile, flammable or caustic material, always use proper and adequate ventilation, and follow all other safe handling laboratory procedures.

1.4 Specifications

Mechanical	
Dimensions :	~ 6 ½" H x 6 ⅝" W x ~ 16" D
Weight :	~ 11.5 lbs (~ 5.2 kg)
Electrical	
AC-DC Power Supply :	*100-240VAC 47-63 Hz; +5VDC±1%; 10W max.
Zetatrac™ :	*+5VDC In; less than 5W power consumption
Solid-State Diode Lasers :	2 lasers; wavelength 780nm; max. 5mW optical output power; nominal 3mW output power
Computer	
System Requirements :	* Refer to Microtrac FLEX Operating Manual SW0003 for minimum system requirements
Special Hardware :	*One high-speed data acquisition board, requires one full-size PCI-bus slot on the PC motherboard
Environmental	
Ambient Temperature :	10 to 50°C
Humidity :	Up to 90%, non-condensing
Sample	
Volume :	0.7ml (700 µl) to 3 ml
Temperature	
Range :	10 to 80°C
Accuracy :	±0.1°C
Control :	Control of sample temperature is not available
pH Range :	3 to 10
Conductivity Range :	0 to 5 milliSiemen/cm
Size Analysis	
Particle Size Range :	0.0008µm (0.8nm) to 6.54 µm
ZetaPotential Analysis	
Particle Size Range :	0.0008µm (0.8nm) to 6.54 µm
Concentration :	0.1 vol% to at least 5 vol% (max. conc. can be higher; this is sample-dependent)
Electrophoretic Mobility[1]	
Range :	-10 to +10 µ/sec per volt/cm
Accuracy :	±0.3 µ/sec per volt/cm
ZetaPotential[1]	
Range :	-125 to +125 mV
Accuracy :	±3.8 mV
Chemical Compatibility :	Wetted surfaces may include: 316 stainless-steel, Inconel, oxide films, gold, titanium, Delrin, Teflon, sapphire, Hastelloy C. Recommended suspending medium is aqueous (water); other mediums (solvents, etc.) can be accommodated; contact Microtrac Technical Support for use with other mediums.

[1] Mobility and zetapotential specifications are at nominal sample temperature of +25°C.

2. Installation

2.1 Selecting A Location For The Zetatrac

The Zetatrac™ should be operated on a flat and level surface. Since the sample-cell is open, the surrounding environment should be such that sample contamination is minimized. The selected location should be as free from vibration as possible. If excessive vibration is present, additional steps should be taken to isolate the Zetatrac™ Contact Microtrac Technical Support for guidance on vibration isolation.

2.2 Setup for Operation

CAUTION

Before connecting or disconnecting any cables from the Zetatrac™ always insure that power to the computer is turned off. This will prevent possibility of damage to the Zetatrac™ or to the computer.

There are several actions that must be taken prior to connection and operation of the Zetatrac™ These actions follow this sequence:

* Install software drivers, for data acquisition hardware, onto computer. Follow Microtrac Service Instruction SI-900692.
* Install data acquisition hardware into computer. Follow Service Instruction SI-900692.
* Install Microtrac FLEX operating software into computer. Follow FLEX Manual SW0003.

Once these steps are complete, proceed to the next section.

2.3 Connecting And Disconnecting The Zetatrac

Included with your Zetatrac™ shipment are an AC-to-DC power supply and a computer connection harness, shown in the following figures:

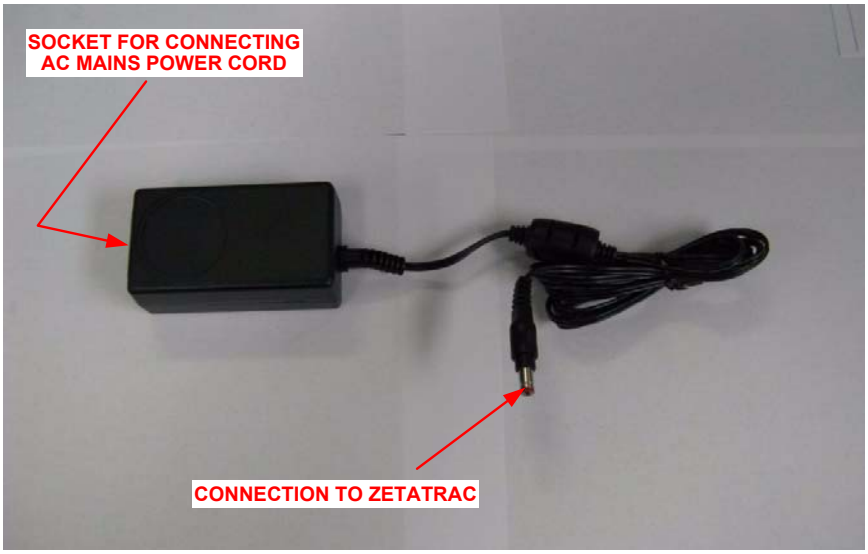


Figure 2-1: External AC-to-DC Power Supply For Zetatrac

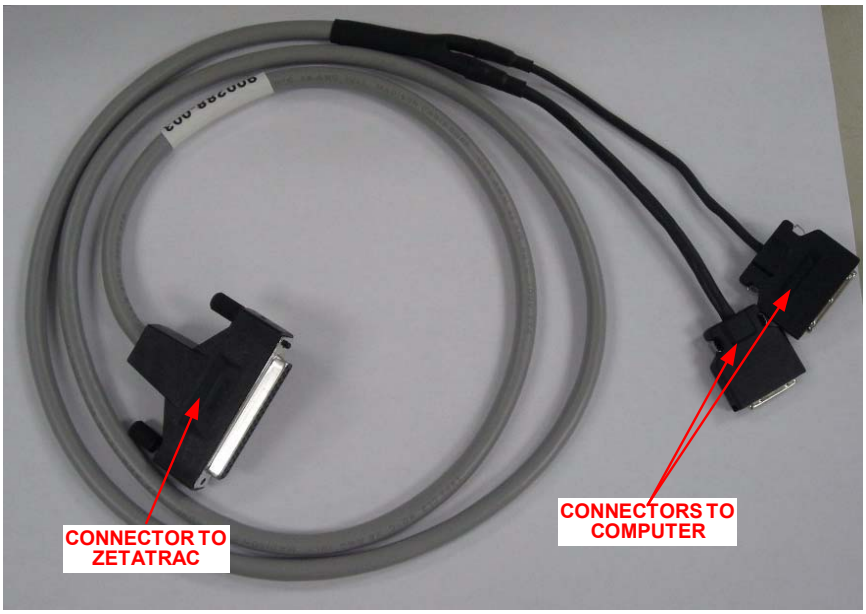


Figure 2-2: Harness Used to Connect Zetatrac™ to Computer

* Insure that power to the computer is off.
* Refer to Fig. 3 earlier in this document. Switch the Laser switch to 'Laser Disable'.
* Refer to Fig. 3. Connect the computer harness to the Zetatrac™ Secure it with the thumbscrews.
* See following figure to connect Zetatrac™ harness to previously-installed data acquisition computer board. Note that the computer connectors are in two sizes, one 50-pin connector, and one 68-pin connector; in addition, they are polarized. This means that the connectors can only be attached to specific places on the data acquisition board, with a specific orientation. Examine orientation of board connectors, and orient the harness connector to match it. Do not force connectors.

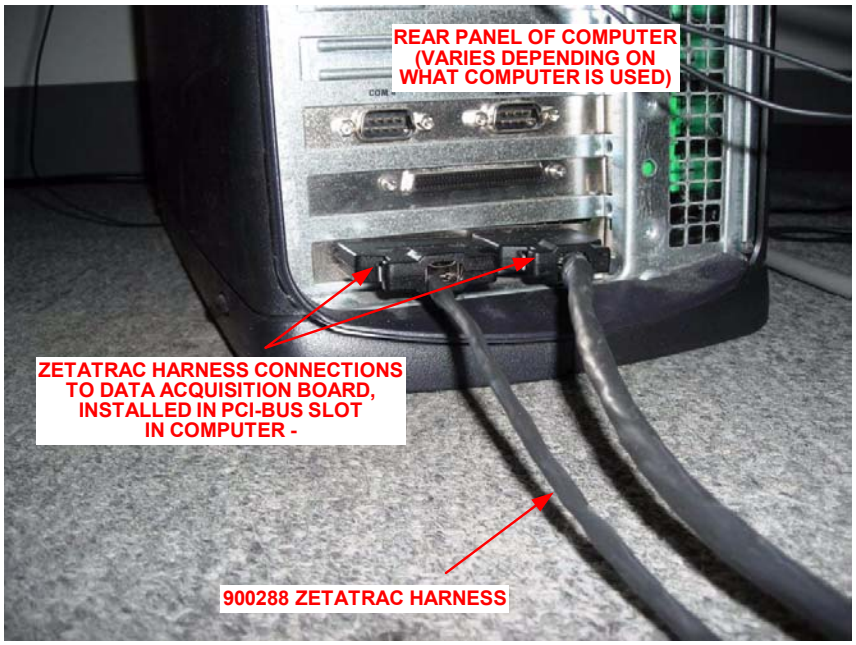


Figure 2-6: Harness Connections to Computer

* Connect the detachable power cord from the AC mains power source to the socket on the external power supply. Refer to Fig. 3; connect the power supply plug to the +5V jack on the rear of the Zetatrac; insert until the plug 'clicks' into place and is secure.

CAUTION

Use only AC Mains power system with protective-earth ground terminal. Never operate the unit from a power source that does not have a protective-earth terminal. Never attempt to defeat the protective-earth system of the power source.

Note that when the +5V power is connected, and the computer is off, that both optical system activity indicators (ref. Figs. 1 & 2) may be on at the same time. Typically only one or the other on. With the PC off it is acceptable that both indicators are simultaneously on.

* Power the computer. At this point, only one probe activity indicator should be on. Run the FLEX software and begin operating the Zetatrac™ See the 'Operation' section of this document, as well as FLEX Operating Manual SW0003, for guidance on using the Zetatrac™ for analysis.

If, for any reason, the Zetatrac™ must be disconnected from the computer, reverse the above instructions - close FLEX, shut down and power-off the computer, disconnect cables. When disconnecting harness connectors from the data acquisition board, be sure to grasp the connector body, and NOT the cable, when pulling the connector free.

3. Operation

3.1 Introduction

A complete Zetatrac™ Particle Size Analyzer system consists of the Zetatrac™ a computer with Windows operating system, a PCI-bus data acquisition board, and connecting cables. The Microtrac FLEX Software, installed on the computer, provides all operator interface functions, data acquisition and analysis commands, and data report formatting, as well as database data retrieval and supervisory control.

FLEX Operating Manual SW0003 contains detailed information on the use of FLEX to control the Zetatrac™ adjust analysis parameters, save and recall data, etc. The user should refer to this manual for details on Flex and it's features. Excerpts of SW0003 are given in this section of this document to help the user complete initial installation and to begin using the analyzer

3.2 Operation Summary

When Zeta Potential measurements are “Enabled” the measurement process contains additional data collections that consume more time than a Particle Size only measurement. Both Particle Size and Zeta Potential are measured and reported when Zeta Potential Measurements are “Enabled”. Total measurement time will be:

Total Test Time, Zeta Enabled = ('Run Time' * 2) + (Additional 20 to 100 seconds)

For example: a 60 second run time with Zeta Potential will take

60 + 60 + (20 to 100) = between 140 and 220 seconds

For size measurement only, disable the zetapotential measurement from the Measurement Setup dialog; insure that the 'Enable ZetaPotential' box on the 'Zeta Pot.' tab is not checked. With the Zeta Potential disabled, Zetatrac™ operates similar to Nanotrac™ Ultra analyzer (in other words, total test time = 'Run Time').

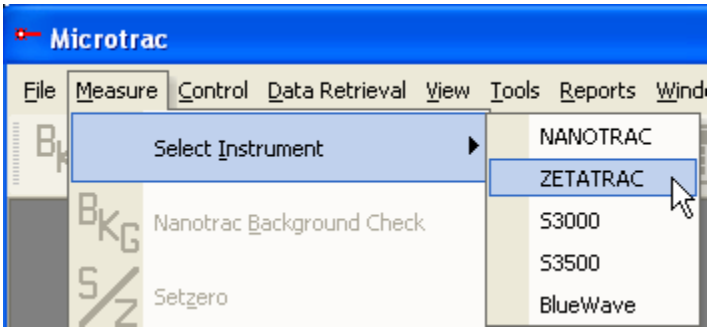
Reports that include Zeta Potential are selected from the “Reports Select/Design” dialog box. See SW0003 for information on customizing report content.

3.3 Setup

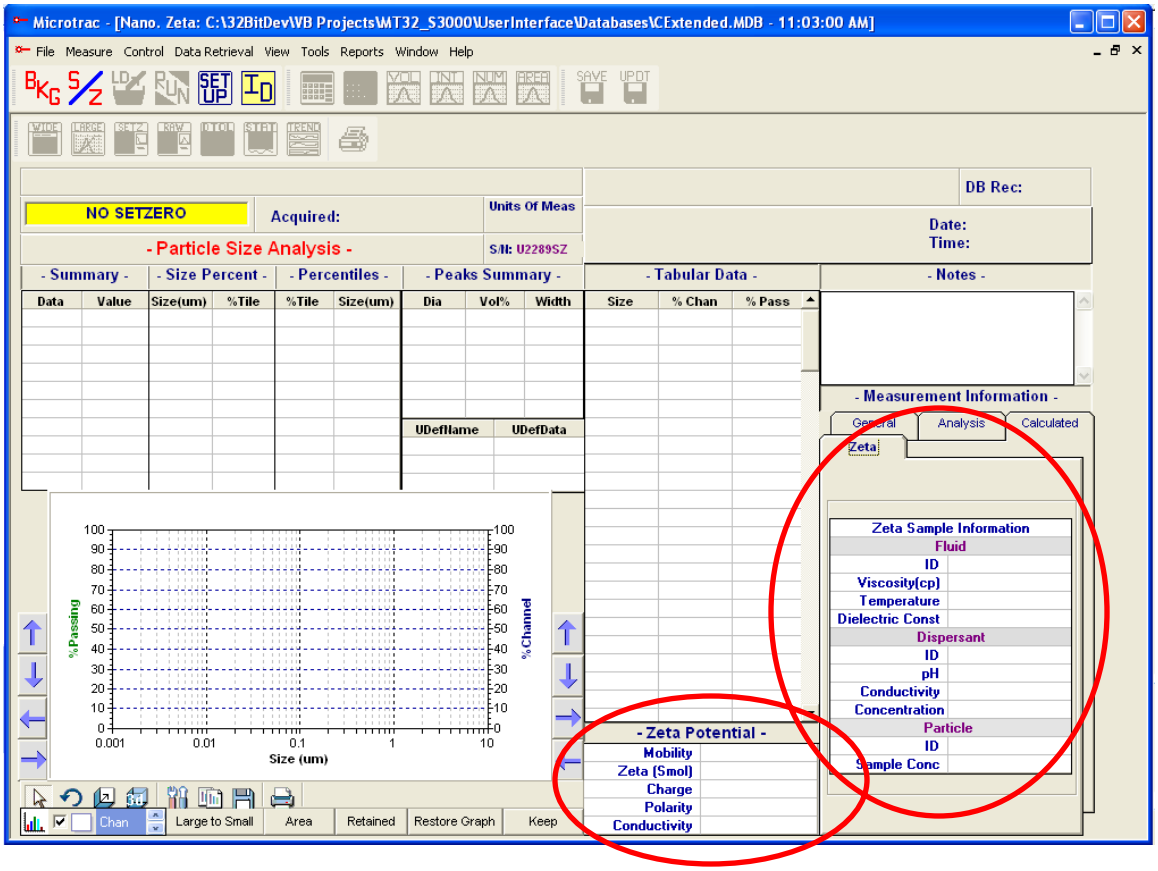
Starting The FLEX Software For Use With Zetatrac

Start the FLEX software from either the Windows Desktop icon or from the Windows 'Start' button.

Select Zetatrac™ from the 'Measure - Select Instrument' menu.

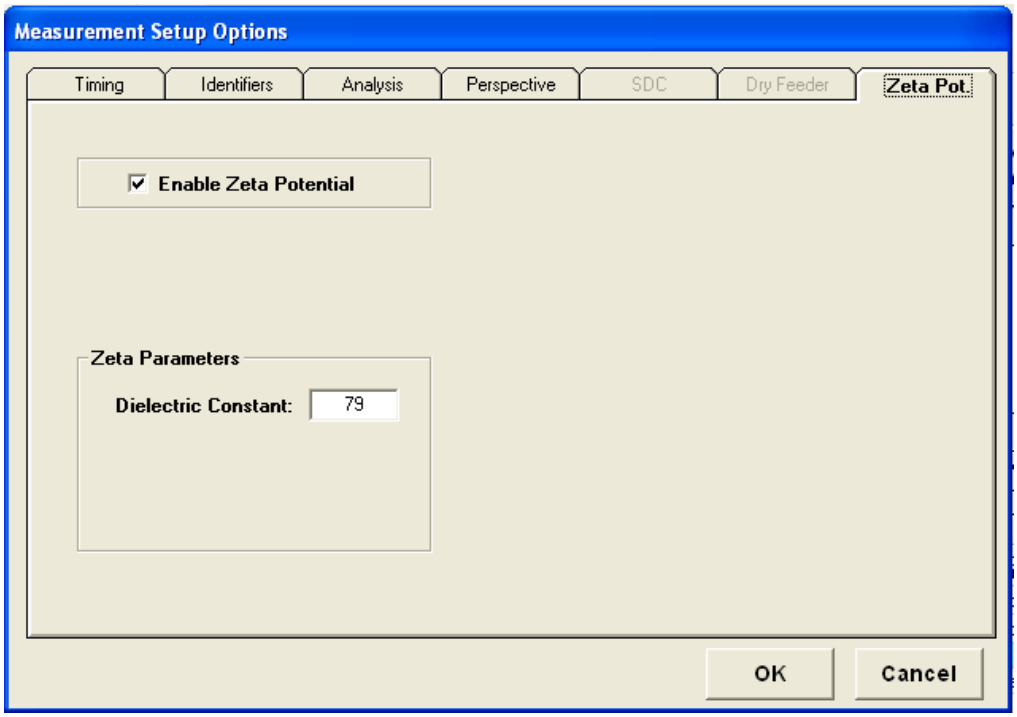


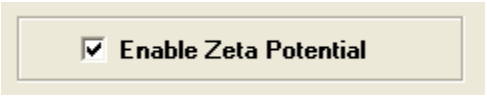
When the Zetatrac™ measurement window opens the screen will appear as shown below with a blank area for Zeta Potential data shown on the “Measurement Information” area in the lower right corner of the display.



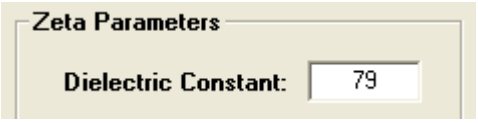
ZetaPotential Measurement - Setup Parameters

To access Zeta Potential measurement setup parameters, click the 'Setup' toolbar button, then click 'Options', to open the Measurement Setup Options dialog, and select the 'Zeta Pot.' tab.



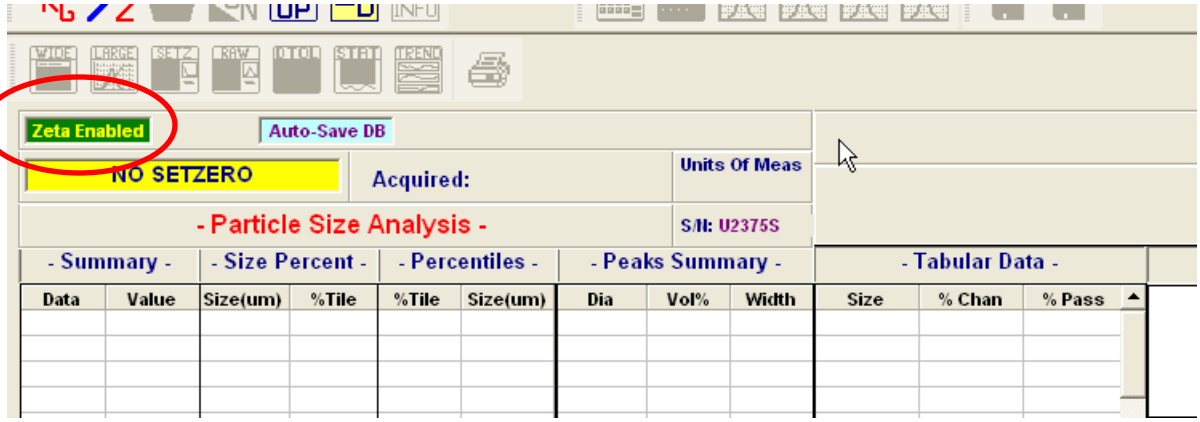


Check the 'Enable Zeta Potential' box to instruct the instrument to include the Zeta Potential function when sample measurements are performed. If this item is NOT checked, then only the particle-size measurement function will be performed.



Enter the Dielectric Constant for the suspending fluid. This value is required for the Zeta Potential calculation. Default is 79 the dielectric constant of Water.

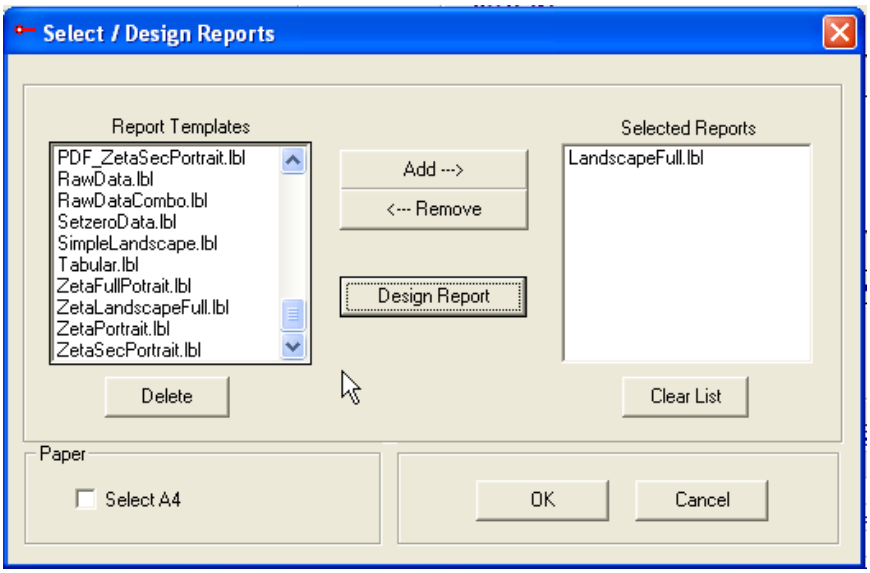
Click 'OK', then 'Close' to close the Measurement Setup Options dialog, and to save the ZetaPotential settings. Note that there is now an enunciator on the Measurement window, telling the user that ZetaPotential measurement function is enabled.



Selecting ZetaPotential Reports

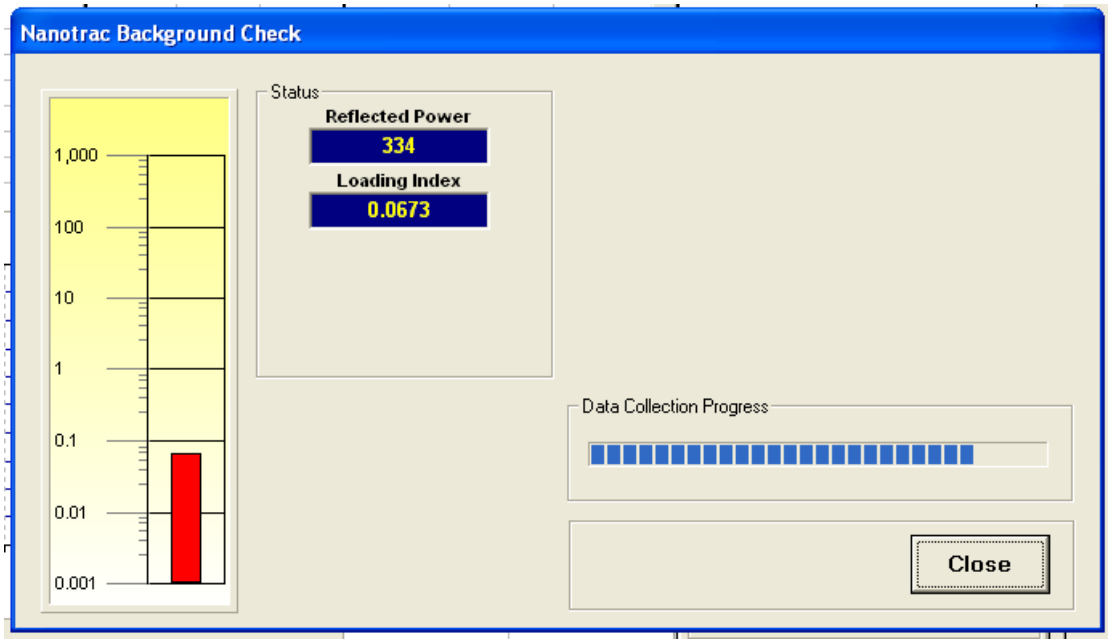
For ZetaPotential data to be presented in printed reports, several pre-designed reports have been provided. The user must select one or more of these reports for printed ZetaPotential results to be produced. To select one or more of these reports, click the 'Reports - Select/Design' menu.

Select one of the reports whose name begins with 'Zeta', and click 'Add', to add this formate to the 'Selected Reports'.

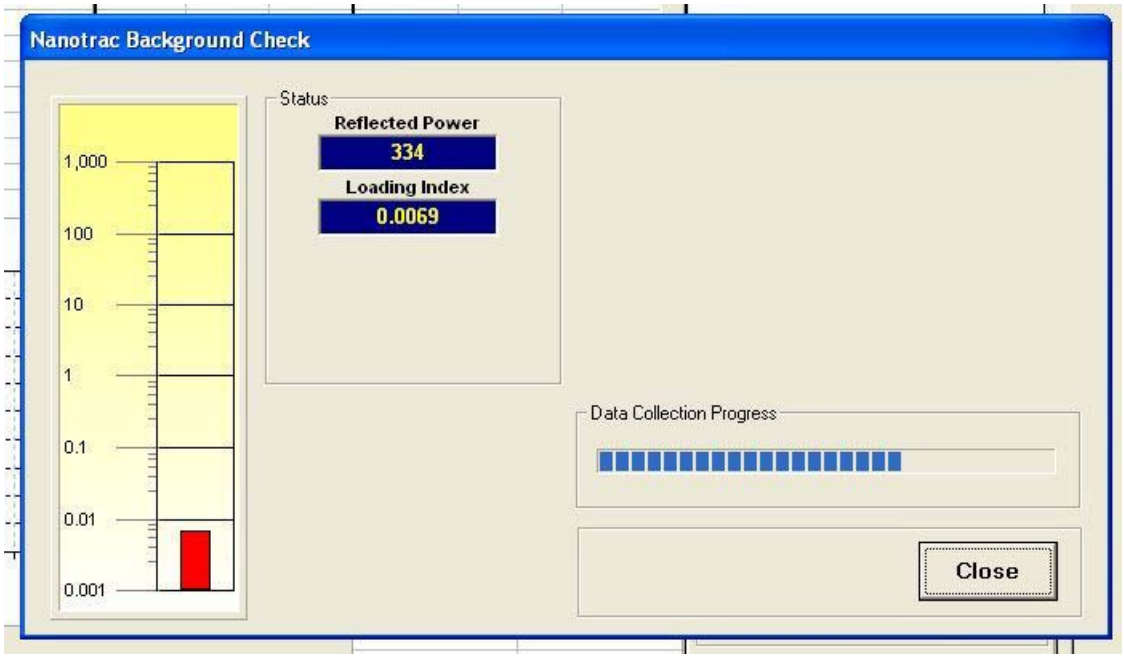


3.4 Performing Zeta Potential Measurement

Add clear fluid to the sample cell and click the 'BKG' toolbar button, to perform a background check. This check can be used to determine the level of particulate in the cell prior to the start of a SetZero or a measurement Run. A Background Check screen appears; a desirable goal is to have Background ≤ 0.01 prior to SetZero or Run. Refer to the following figures.



Zetatrac/Nanotrac Background Check Screen - Additional cleaning is needed



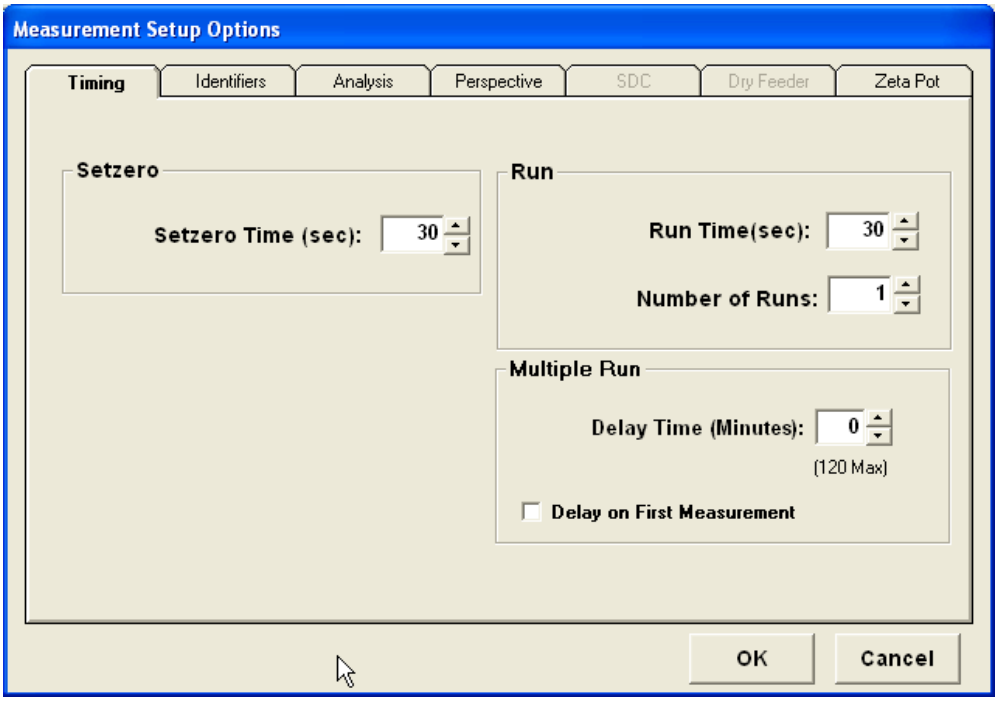
Zetatrac/Nanotrac Background Check - Clean, ready to perform SetZero or Run

The Setzero (Background Measurement) is performed by clicking  on the FLEX toolbar. Note the color of the SetZero enunciator on Measurement window:

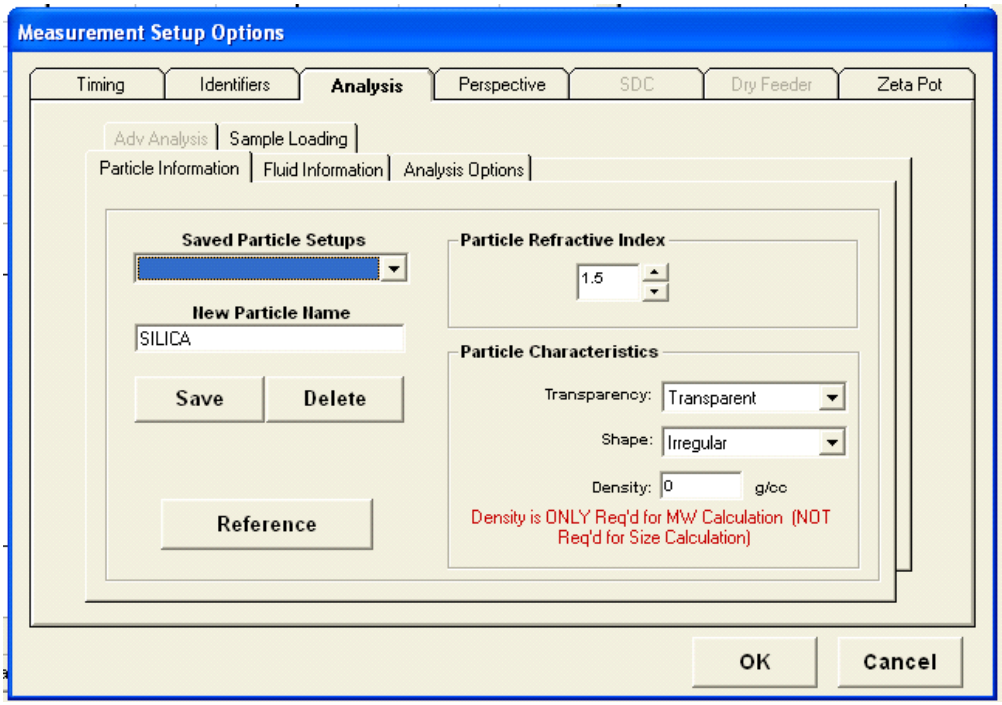
* Yellow with 'No SetZero': Remains like this until a successful SetZero is completed;
* Green with Date and Time: A successful SetZero has been completed;
* Red with Date and Time: SetZero attempt was not successful; run Background Check.

When the setzero is successfully completed, extract the fluid if desired, to prevent sample dilution. Add sample material to the sample cell; add sufficient sample to immerse the optical probes.

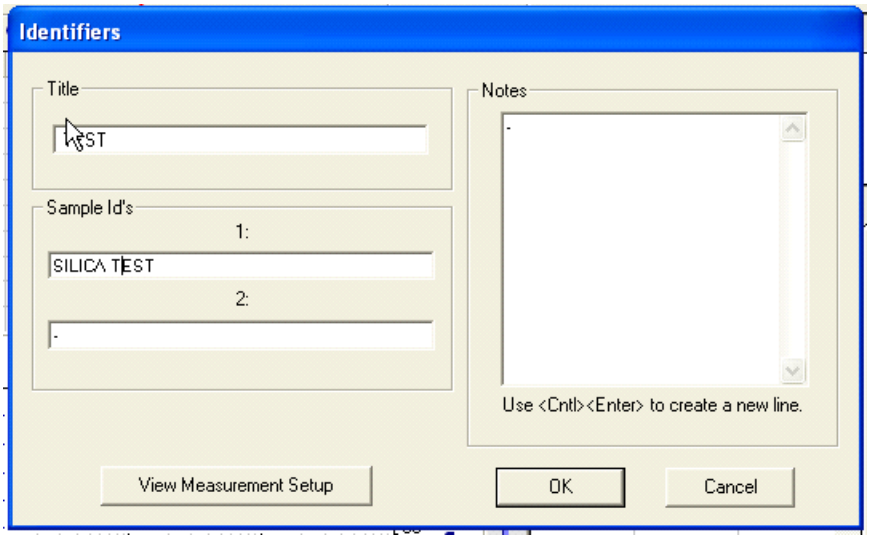
Click the 'Setup' toolbar button to adjust the measurement parameters. Refer to the following figures. Refer to other sections in this manual for guidance on setting parameters.



Measurement Setup – Timing Parameters



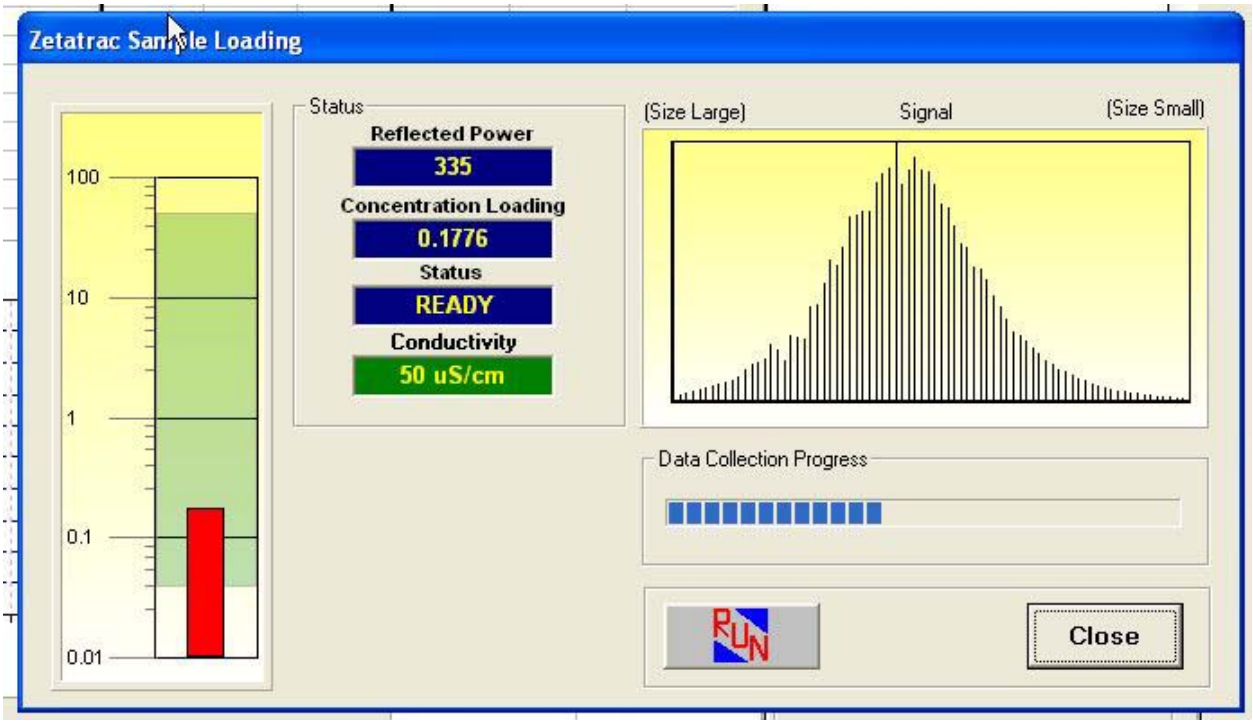
Measurement Setup – Analysis Parameters



Measurement Setup – Sample Identifier Parameters

Introducing Sample to the Analyzer and Loading Check

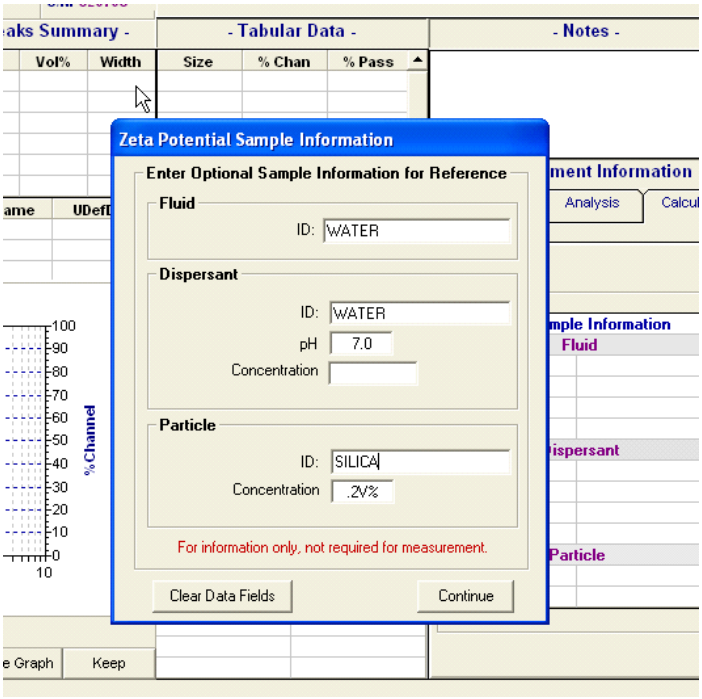
Click on 'LD' toolbar button to launch the Zetatrac loading screen. Note that this is a measure of sample concentration. Accurate refractive indices for particle and fluid are necessary for correct calculation of concentration here. When the 'loading bar' is within the acceptable 'green zone' limits, and the Status enunciator indicates 'Ready', it is ready to proceed with a measurement run, which will use the previously-entered parameters. If sample concentration is too high, the loading bar will out of the green zone, and the Status should show 'Dilute', indicating the action to be taken. If concentration is too low and the loading bar is below the green zone, the Status indicator should show 'Add Sample'.



Performing the Measurement ('Run')

Click  to perform the Particle Size and Zeta Potential measurement.

Complete the Optional Zeta Potential Sample Information form (shown below),. and click  to perform the particle-sizing and zetapotential measurement functions.



ZetaPotential Sample-Information Fields

The information in these fields *DO NOT* directly influence Zeta Potential measurements and are for informational purposes only.

Fluid ID: **Enter an Identifier for the Fluid (carrier) (this is a text field)**

Dispersant ID: **Enter Identifier for the Dispersant ID (if used) (this is a text field)**

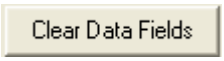
Dispersant PH: **Enter the sample pH (this is a number field)**

Dispersant Concentration: **Enter the dispersant concentration (this is a text field)**

Particle ID: **Enter an Identifier for the Particle (this is a text field)**

Sample Concentration: **Enter the sample concentration (this is a number field)**

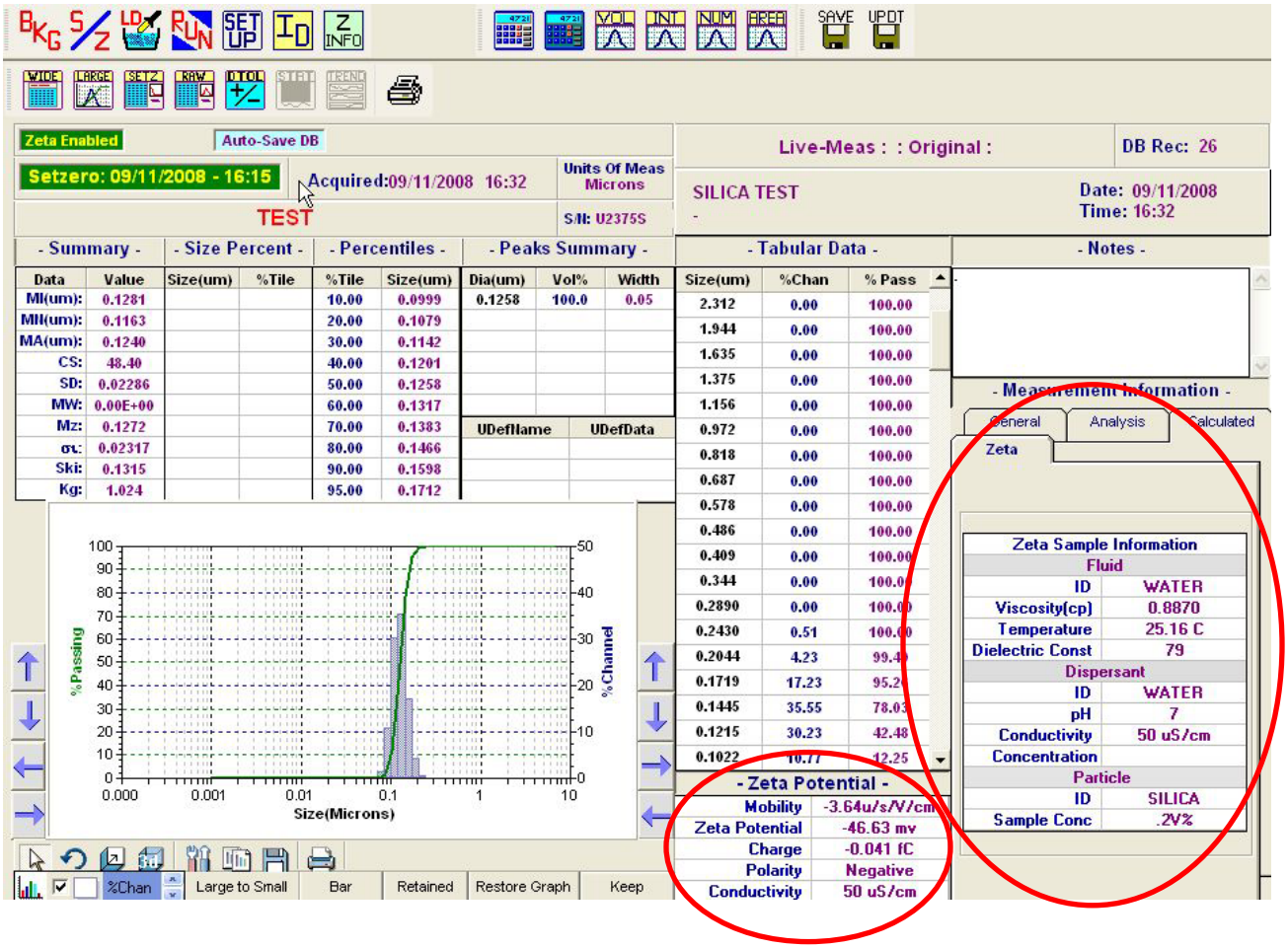
NOTE 1: Any previously-entered values will be kept and shown for each future sample. Be sure to change these entries as required for each sample.

NOTE 2: Clear any unwanted sample information from this form if no information is desired. The information in this form WILL be saved and displayed with the results of the measurement. All fields can be cleared simultaneously by clicking the  **button.**

NOTE 3: Particle Size Data will be displayed PRIOR to the Zeta Potential measurement being performed.

Click 'Continue' and the measurement run will begin. Throughout the run, the progress of the measurements is indicated in the Measurement window.

When all measurements are finished, the completed measurement window appears, similar to that shown here, with zetapotential measurements included.



NOTE: Zeta Potential Sample Information can be changed from the completed data display (measurement or database recall) by clicking the Z INFO button. This will open the Zeta Potential Sample Information dialog, and if you exit by clicking the Update button, the data display will be updated to the new values. If the data has been saved to a database record, then it is the user's responsibility to "Update" UPDT the full record, to reflect the zetapotential sample information changes that were made.

3.5 Zetatrac™ Testing Guidelines & Information

Fluid Temperature

The Zetatrac™ will measure samples that are at temperatures other than room temperature. It is advisable that the temperature of the fluid used to perform a SETZERO be at the same as the temperature of the sample used for a measurement RUN.

SetZero ('SZ')

Setzero is a procedure for measuring the background or steady-state noise of the system. Some of this noise can originate from minute contamination that may be present in the clean dispersing fluid, but the bulk of it is electrical noise inherent in the electronics of the system. Noise characteristics can vary from unit to unit, but are inherently constant in a given unit and are always present, whether clean fluid or a sample is being measured. Therefore, it is legitimate to treat contamination and noise as a background. This background is measured with clean fluid in the cell, stored, and then subtracted after each sample measurement.

* Because some of the background noise can be due to conditions of the fluid or fluid-probe interface, perform a SetZero whenever there have been changes in the environment or operating conditions, such that the background may have changed. For instance, SetZero must be performed whenever a new solvent or dispersing fluid is used.
* Perform a SetZero whenever the type of sample being measured is changed dramatically (for example, changing from a mineral slurry to a paint emulsion).
* A SetZero must be performed after a power-up, before making any sample runs.
* A SetZero must be performed after opening a Zetatrac™ measurement window in the Microtrac® Windows Software.
* Before performing a SetZero, use a clean pipette (provided with system, or supplied by the user), filled with appropriate fluid, to flush out the cell and remove all particles. Fill the sample cell with clean dispersing fluid. Perform a SetZero using the Microtrac FLEX Software Program.

CAUTION

Do not move, bump, touch or otherwise disturb the Zetatrac™ during the SetZero procedure.

At the end of the SetZero procedure the computer screen will display the SetZero Status. If the SetZero Status is BAD, then thoroughly clean and rinse the Sample Cell. Fill the sample cell with clean fluid and use the Background function on the Microtrac FLEX Software Program to check the Loading Index. The Loading Index should be less than or equal to 0.010 to be acceptable to the system as background.

After flushing all particles from a cell, you can gauge its cleanliness by comparing the measured Loading Index to the loading level currently being utilized. Flushing should proceed until the loading level clean is 1/100 to 1/1000 the expected loading level with current sample being run. For example if expected loading is 50 clean to 0.05 to 0.5, if expected level is 2 clean to 0.002 to 0.02.

Microtrac recommends that a SetZero be performed at startup each day. Then the SetZero procedure should be periodically performed throughout the day. For optimum performance, especially if using sample with low concentrations, Microtrac recommends performing SetZero with clean liquid before every sample run.

Sample Dilution

Dilution is normally necessary because most processes provide fairly concentrated samples. The sample acquired from the process must be diluted using a solvent or dispersing fluid compatible with the particles and the Microtrac® system. The SetZero measurement must be performed with the chosen fluid in the cell. When choosing the fluid, its viscosity and refractive index must be known and entered into the fluid parameter menu.

'Multiple Run' Selection

Multiple Runs may be chosen to provide multiple measurements and average measurements of the same sample. Total time of Zetatrac Multiple Run sequence will depend on the 'Run Time' entered by user, and will also depend somewhat on the sample itself. A Zetatrac measurement run consists of 1) a sizing measurement, 2) and zetapotential magnitude measurement, and 3) a zetapotential sign measurement. If 'n' Multiple Runs are chosen, the overall sequence is as follows:

1) Size measurement 1; time = Run Time;
2) Zeta magnitude measurement 1; time = Run Time;
3) Zeta sign measurement; time = 20 to 100 seconds, depending on sample;
4) Size measurement 2; time = Run Time;
5) Zeta magnitude measurement 2; time = Run Time;

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.
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*) Size measurement 'n'; time = Run Time;
*) Zeta magnitude measurement 'n'; time = Run Time;

Multiple Measurements of the Same Sample

The Zetatrac's zetapotential measurement operation has the possibility of altering the particle distribution throughout the sample's volume. The possibility that this alteration will happen is sample-dependent. Therefore, Microtrac recommends that if repeated measurements are to be made of the same sample, with 'Zetapotential' enabled, that in-between measurements the sample should be agitated to redistribute particles throughout the sample volume. For instance, using a clean micropipette, the sample could be extracted and reinserted several times; this will perform the redistribution action.

Note: this does not imply that any user action is needed during 'Multi Run' measurements; agitation action may only be necessary after the entire run is complete.

Fluid Compatibility

The Zetatrac™ has been designed for use with a wide range of fluids. However, when using non-aqueous samples (polar solvents, non-polar solvents, organic solvents, pH

outside of stated range, etc.), the user is recommended to contact Microtrac Technical Support. Refer to Specifications section for details.

Fluid Viscosity

Fluid viscosity must be known accurately at two temperatures within the 10^{o} to 80^{o}C operating range. The operator enters these two temperatures and corresponding viscosity values into the Microtrac® FLEX software. FLEX uses these values and the measured cell temperature to compute actual fluid viscosity at the measurement temperature.

Viscosities from 0.3 to 3 cp are generally preferred. Higher viscosities cause slower particle velocities and, therefore, lower frequencies in the detected signal. The upper size limit will be determined by the viscosity -- the higher the viscosity the lower the upper size limit. In water (1cp) the size limit is 6.4 microns. At a viscosity of 10cp the limit would be 0.64 microns.

A signal with a frequency spectrum mainly in the low frequency range requires longer sampling time, with the time increase proportional to the viscosity increase above that of water. For example:

* A 500 nm sample has normal run time of 30 to 60 seconds in water, 1 cp fluid viscosity.
* A 500 nm sample in a fluid of 3 cp viscosity exhibits a frequency spectrum similar to that of a 1,500 nanometer sample in water. Thus, a 500 nm sample in a fluid of 3 cp viscosity must use a run time of 90 to 180 seconds or more.
* The Zetatrac™ has a particle size range of 0.0008 (0.8nm) to 6.54 microns in water. Higher viscosity changes the upper size limit. The effect can be estimated as follows:
  - The product of [Viscosity (cp) * Particle Diameter (microns)] should be between 0.0008 (0.8nm) and 6.54;
  - A 1 micron sample in 3 cp fluid is equivalent to a 3 micron sample in water (1 cp);
  - A 2 micron sample in 3 cp fluid is equivalent to a 6 micron sample in water.

Refractive Index

Particles are visible to the instrument only if their refractive index is different from that of the suspending fluid. Scattered power is reduced as the particle index approaches the fluid refractive index. Refer to SW0003 Flex Software Operations Manual for additional guidance on how to enter refractive index information for fluids and for particles. Use the SAMPLE LOADING function and the procedures outlined in the next section, to determine if the fluid/particle combination gives adequate Loading Index (LI).

Sample Concentration

The Loading Index is given at the end of a measurement and in the Sample Loading window; refer to SW0003 Flex Software Operations Manual for details. The Loading Index is a measure of the total AC signal obtained from the light scattered from the moving particles.

Particles with a high refractive index relative to the fluid index tend to give larger signals. However, there can be significant variation of scattering efficiency as a function of particle size. Efficient scatterers can give adequate signal at concentrations as low as a few parts per million (ppm), while inefficient scatterers can require concentrations of several thousand ppm.

Scattering efficiency makes concentration important for some samples. Overloaded samples can create optical signals which could create artificial 'modes' or peaks in the size distribution. Scattering efficiency needs to be considered when sample concentration is determined.

Concentration limits can be broken down into three ranges depending on the Loading Index.

Safe Range: Loading Index = 0.1 to 100.0

This range of Loading Index can be used without concern for instrument or sample limitations.

Caution Range: Loading Index < 0.1

Use very small particle or very low concentrations of particles with caution. There is no absolute lower-Loading-Index limit, but the precautions observed become more severe depending on the Loading Index utilized. Concerns include:

- Contamination of small particles with low concentrations of large particles.
- Setzero Background Index should be less than 0.010
- Interference of the measurement with environmental changes, such as bench vibration or rapid temperature changes should be avoided.

Caution Range: Loading Index > 100.0

High Loading Index values are also to be used with caution. At high sample concentrations, particle interactions become more probable, and results are subject to interpretation. Agglomeration, changes in the effective dispersing fluid viscosity, and changes in effective dispersing fluid index can result from high sample concentration and particle interactions. These effects can shift or distort measured distributions. The individual sample chemistry will determine the Loading Index limit. Determination of the limits must be made on a sample-by-sample basis.

3.6 Sample Preparation

Some applications require the measurement of particles that are normally dispersed. These samples require no preparation and can be added directly to the Sample Cell. Other applications require the addition of surfactants or other dispersing agents, in addition to mechanical energy, to disperse the sample into individual discrete particles. Samples must be representative of the entire product lot or batch. This determines which, if any, sample preparation techniques are required. Sample preparation techniques presented in this section are of a general nature; address specific questions about particular sample preparation techniques to:

Microtrac Technical Support
12501-A 62nd Street North
Largo, FL 33773
(727) 507-9770

Sample Preparation Techniques

Two steps, wetting and dispersion, are normally required to achieve a non-agglomerated sample.

* Wetting uses water, other suspending media, and/or chemicals to reduce surface tension to promote mixing and diluting a sample in a suspending fluid. A wetted sample mixes freely.
* Final dispersion (de-agglomeration) may require energy to act mechanically on the suspension.

Wetting agents include water, surfactants, dispersants, and solvents.

NOTE: Surfactants or dispersants used in excess can cause formation of bubbles that may affect the measurement. Excess surfactant can also lead to re-agglomeration when reaching the surfactant critical micelle concentration.

ZETATRAC™ NOTE: Use of surfactants and dispersants may affect the accuracy of Zeta measurements.

Dispersion

Dispersion requires energy input from one of several devices. Most commonly, ultrasonic energy is employed in the form of a bath or probe.

High shear devices such as tissue homogenizers should not be used because they tend to produce artificial distributions and a non-representative sample.

Germicides

Microscopic organisms in the sample or the dispersing fluid can be read as particles. Use an appropriate germicide if the fluid is conducive to microbial growth. Such growth is typically slow. In clean water, bacteria can take several weeks to grow to a noticeable size and/or concentration.

Particle Size Measurement

Once the sample has been diluted to a concentration between the upper and lower boundaries of the loading screen, it can be run. Follow the instructions above, and in the SW0003 Microtrac FLEX Software Manual, to conduct a run.

Run Time

Optimum measurement (run) time depends on the particle size. Small, fast-moving particles can be measured in short times, while larger slower moving particles require longer times. Use the following guidelines to determine minimum measurement time. Measurement times needed to achieve maximum repeatability are determined experimentally by calculating statistical repeatability (for example mean and standard deviation) at several measurement periods

Table 3-1 Run Time

Particle Size Range (Nanometers)	Minimum Run Time (Seconds)
Below 60	30
60 to 300	90
300 to 900	120
Above 900	180

Run time can always be increased above these values. Longer Run Times may provide more repeatable data.

After completing a sample measurement, thoroughly flush the sample cell to avoid accumulation of contaminating particles. Refer to the Maintenance section for additional guidance on cleaning the sample-cell. Do not leave sample material in the cell. The fluid can evaporate and deposit particles on the probe face and other cell parts. Dried particles are more difficult to remove than suspended particles.

If particles dry onto the sample cell surfaces, wet-swab the entire sample cell. Then rinse the sample cell two to three times with clean fluid to remove any residual contaminates.

4. Principles of Operation

4.1 Introduction

The Zetatrac™ measurement technique is that of dynamic light scattering. The velocity distribution of a sample of particles suspended in a dispersing medium is a known function of particle size. Light from a laser diode is coupled to the sample through an optical power splitter/probe assembly. Light scattered from each particle is Doppler-shifted by the particle motion (Brownian motion). The Doppler-shifted scattered light is mixed with coherent, un-shifted light; and the optical system sends these mixed signals to a silicon photo-detector. The detector output signal is then amplified, filtered, digitized, and mathematically analyzed by the Microtrac® FLEX Windows Software, using proprietary algorithms, to provide the particle size distribution.

Zetatrac™ extends the dynamic light scattering measurement function of Microtrac's Nanotrac™ particle size analyzer, to measure both the particle-size-distribution, and the zetapotential of suspended particles. Added to Nanotrac™ is the ability to apply an electric field to a particle suspension and simultaneously view the resulting particle motion. Zetatrac™ analysis of the particle motion determines the particle charge, electrophoretic mobility and zeta potential. In addition to the Zetatrac™ analysis all measurements include the standard Nanotrac™ particle size distribution.

4.2 Physical Principles

Brownian Motion

Particles suspended in a dispersing fluid are subject to random collisions with thermally excited molecules of the fluid. The velocity and direction of the resulting motion are random, but the velocity distribution of a large number of mono-sized particles averaged over a long time will approach a known functional form.

The velocity distribution is a known function of particle size. Figure 4-1 shows that a sample of small particles has a higher median velocity than a sample of large particles, where the median velocity is inversely proportional to the particle size. Velocity distribution is also a function of fluid temperature and fluid viscosity.

If the fluid molecules have higher average thermal energy (higher temperature), they will impart higher velocities to the particles with which they collide. Median particle velocity is directly proportional to the absolute (Kelvin) temperature of the fluid. A viscous fluid slows the energized particles. Particle velocity is inversely proportional to fluid viscosity.

Viscosity itself is a complex but predictable function of temperature. If the operating temperature is known, the viscosity at that temperature can be computed. Thus, compensation can be made for both temperature and viscosity effects. With compensation, the velocity distribution becomes a unique function of particle diameter.

Viscosity entered into the Flex software must be of the clean fluid only; considerations of the viscosity of the mixed sample, especially if that mix is considered to have non-Newtonian behavior, must be avoided.

Figure 4-2 shows the general behavior of particle velocity distributions as a function of particle diameter, fluid temperature, and fluid viscosity. The median particle velocities range from 5 micron/second to 6000 micron/second.

Doppler Effect

Light from the laser is coupled through one of the fiber optic tails, through the sapphire window and into the fluid. A small percentage of the light is reflected from the sapphire/fluid interface and travels back through the fiber and into the photo-detector mounted on the Laser/Detector board. Doppler-shifted light scattered back from the particles enters the probe at the tip, mixes with the reflected light and travels back to the photo-detector.

Light incident on a particle scatters in all directions. If the particle is stationary the scattered light is of the same frequency (or wavelength) as the incident light. If the particle is moving at some velocity relative to the light source, the scattered light is shifted in frequency by an amount proportional to the particle velocity. An ensemble of particles with a certain velocity distribution will thus have a unique distribution of frequency shifts.

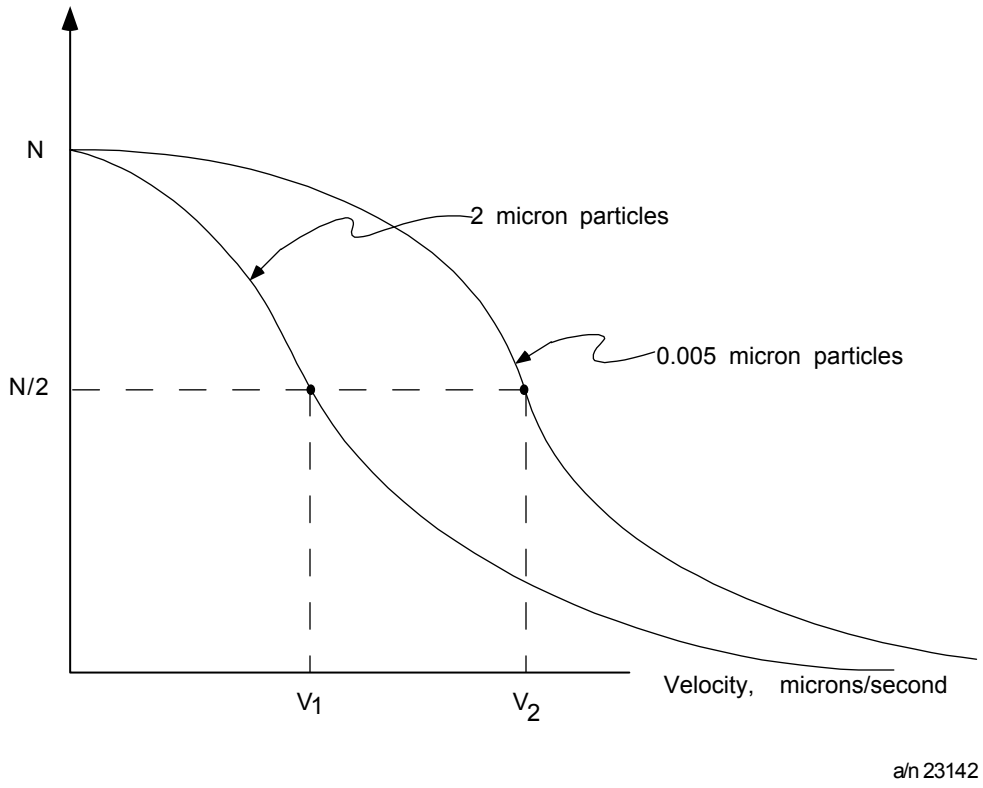


Figure 4-1: Velocity Distribution, Suspended Particles in Brownian Motion

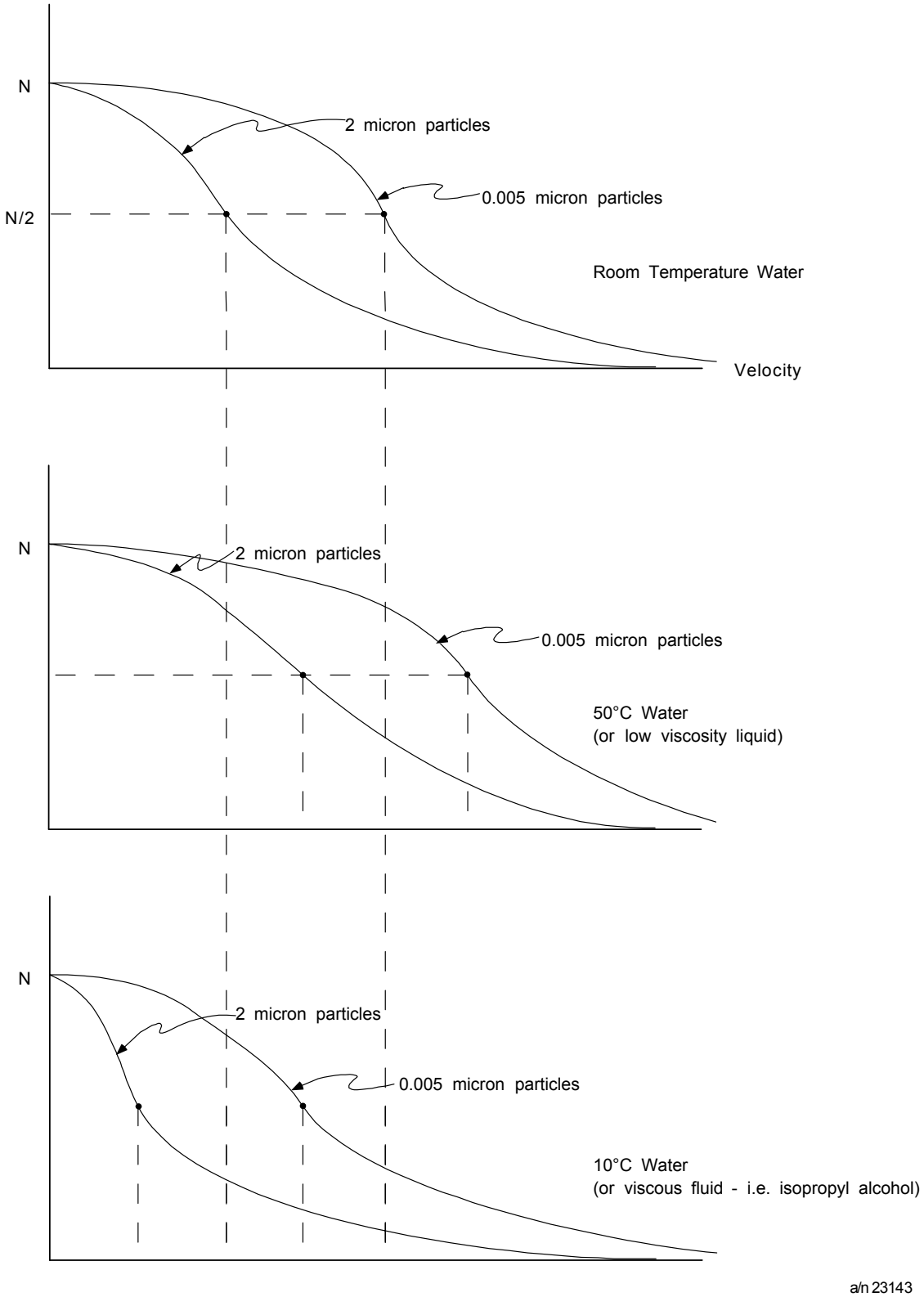


Figure 4-2: Velocity Distribution, Temperature and Velocity Effects

Heterodyne Detection

Heterodyne detection involves combining Doppler (frequency) shifted light scattered from moving particles with a reference beam of light from the same source, not Doppler shifted, reflected from a stationary surface; see Figure 4-3.

The particle velocities are so small compared to the velocity of light that the Doppler frequency shifts can only be detected using frequency-beating techniques. Light reaching the photo-detector consists primarily of a large amplitude component at the transmitted frequency, F_t, and much smaller amplitude components at the frequency of the Doppler-shifted scattered light, F_1 and F_2.

Because the laser output and the reflection at the probe sapphire/fluid interface are constant, the reflected component, F_t, will be large and constant. The components F_1 and F_2 will be small and varying as the particles continuously change direction and velocity. Thus the detector will generate:

* A large dc output component proportional to the reflected light
* Smaller ac output components at the heterodyne difference frequencies, (F1 - Ft) and (Ft - F2)
* Much smaller ac output components at the self-beating frequency, (F1 - F2)

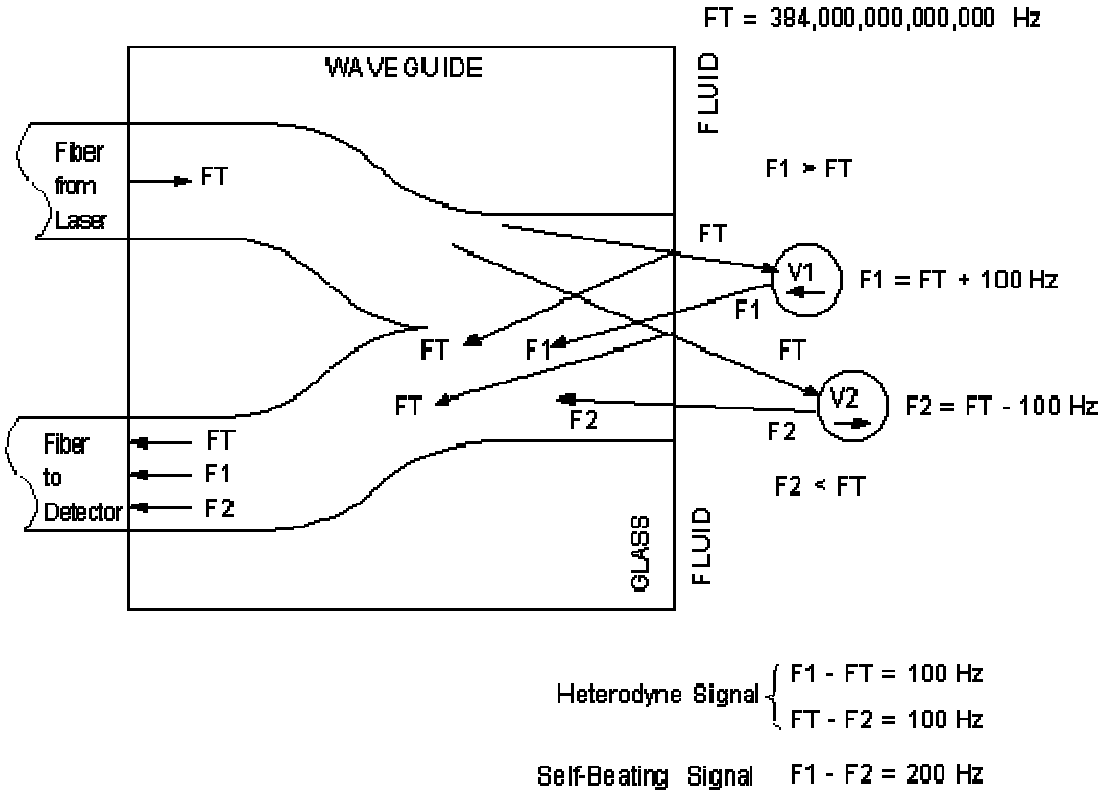


Figure 4-3: Heterodyne Detection of Scattered Light

The self-beating or homodyne frequencies are twice the value of the heterodyned frequencies and can be considered interference. The Zetatrac™ Analyzer makes use of

the heterodyned signal, which will be the major signal over a very wide range of particle concentrations.

Frequency Spectrum

In a real measurement situation, large numbers of particles move randomly in the vicinity of the probe tip, generating an equal number of Doppler-shifted scattered light signals. These signals of various frequencies combine with the reflected signal of un-shifted frequency to generate a wide spectrum of heterodyne difference frequencies. The resulting output of the photo-detector is thus a random signal with a frequency spectrum determined by the velocity distribution of the particles in the sample.

The random signal from the photo-detector is sampled and digitized by the analog-to-digital converter board (data acquisition board). The resulting stream of numbers then undergoes a complex series of mathematical operations designed to compute the frequency spectrum of the original random signal.

Frequency shift is directly proportional to particle velocity, so the shape of the frequency spectrum as a function of particle size, temperature, and viscosity is very similar to the velocity distributions shown in Figure 4-1. Spectra from small particles have more high frequency components than those from large particles. A higher fluid temperature (or a lower viscosity) will cause increased high frequency components in the spectrum.

Particle Distribution

The frequency spectrum is uniquely determined by the particle velocity distribution that, in turn, is uniquely determined by the particle size distribution. Using proprietary signal-processing algorithms, the particle distribution is computed directly from the measured frequency spectrum recovered from the Doppler-shifted scattered light.

Interference Effects and Scattering Efficiency

Transparent particles give rise to optical interference effects; many particles in the Zetatrac™ size range are transparent. These effects can be visualized by reference to Figure 4-3**.**

What is not shown is that a portion of the incident light, at the Doppler shifted frequency F_1, enters the particle and travels inside the particle material at a velocity determined by the index of refraction of the particle material. After traveling through the particle, the light hits the "far wall" and some of it "bounces" and travels back through the particle. A portion of this bounced light then exits at the front wall and travels toward the probe on the same path as the light scattered from the front wall. The frequency of both waves is the same but the phase is not. Since the bounced wave has been delayed by traveling back and forth through the particle, its' phase fronts (or minima and maxima) cannot align exactly with the phase fronts of the scattered wave. The difference in the alignment of phase fronts is called *optical interference*, which can be constructive or destructive:

In the case of constructive interference, the phase fronts align exactly:

* The combined wave has a higher amplitude;

* The particle is an efficient scatterer;

In the case of destructive interference the Maxima of one wave align with minima of the other wave:

* The combined wave has a lower amplitude;

* The particle is an inefficient scatterer

The amplitude of the signal recovered at the photo-detector will be larger or smaller depending on whether the particles cause constructive or destructive interference. The size of the particle and its refractive index will determine the delay between the two waves and therefore the nature of the interference and scattering efficiency.

Figure 4-4 is a plot of scattering efficiency versus particle size, for a given combination of particle index and fluid index. Figure 4-5 gives an expanded view of the scattering efficiency in the region of 0.0 to 0.1 micron.

Other fluid/particle combinations will yield a similar plot but the peaks and valleys will be at different values of particle size.

If the particle distribution in a sample consists of a single particle size, optical interference is not a problem. The shape of the frequency spectrum is unaffected, resulting in the proper computation of particle size.

Optical interference effects can be significant if the particle distribution in a sample is bimodal, or a broad distribution of sizes. The computed particle size distribution would be distorted, skewed in favor of the more efficient scatterers. Note that in the Rayleigh region (up to about 200 nm) the change in scattering efficiency varies rapidly with particle size.

When the distorting effects of the scattering efficiency function are properly compensated, the computed particle size distribution is a true Volume Distribution. If the distorting effects of the scattering efficiency function are uncompensated, the resulting computed particle size distribution is more properly termed an intensity Weighted distribution.

Instruments based on older technologies were unable to make the necessary compensation and presented only Intensity-Weighted particle size distributions. Microtrac® Particle Size Analyzers compute true Volume Distributions in the standard mode of operation, and offer the user the alternative of selecting Intensity-Weighted or Mono-Disperse modes of operation. Refer to SW0003 Flex Operations Manual for details on how to selection the mode of operation.

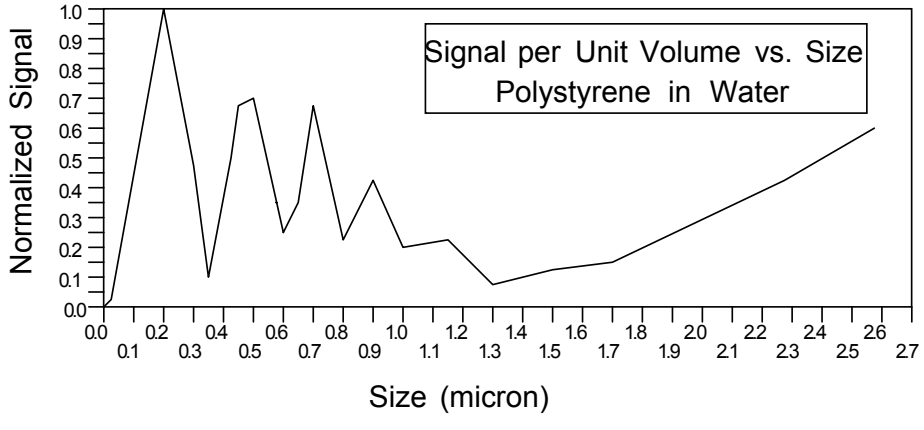


Figure 4-4: Scattering Efficiency, Full Range

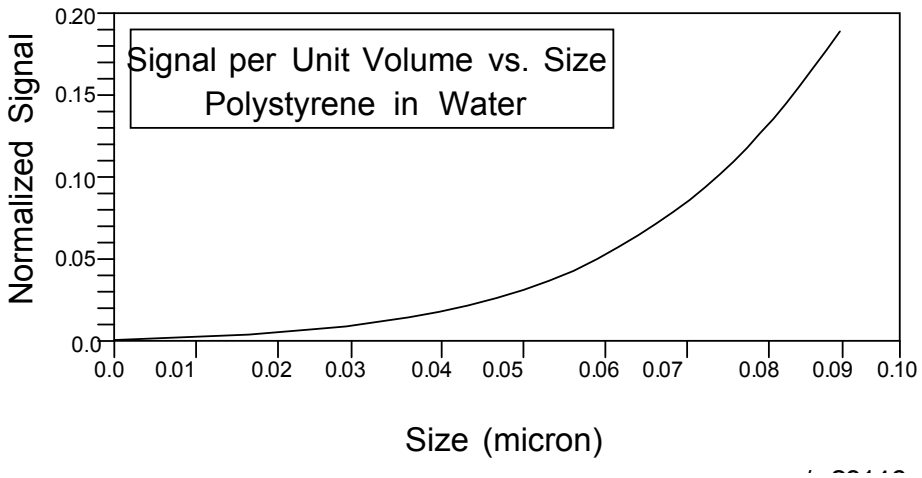


Figure 4-5: Scattering Efficiency, Rayleigh Range

4.3 Zetapotential Measurement

As discussed in previous sections, the Zetatrac™ has all of the features of Nanotrac™, with additional zetapotential measurement capability provided by new hardware:

1. A voltage source with programmable amplitude and wave form.

2. An insulating sample cell, with optical probes opposed by electrodes, forming cell which makes zetapotential measurement possible.

3. Optical probes, with sample interface window consisting of typical sapphire, but having specialized metallic and semiconductor optical coatings applied.

4. Coated-window optical probes are paired with their opposite electrodes. Excitation of cell, from the above voltage source, applied between the optical probe and it's electrode, creates electric fields. Particle motion is analyzed while under the influence of the fields.

As mentioned previously, particle size distribution is determined from the velocity distribution of particles suspended in a dispersing medium, using the principles of dynamic light scattering. The Zetatrac™ analyzer measures the additional velocity imparted to the charged particles when placed in an electric field. Particle electrophoretic mobility is calculated from this additional velocity component. Zeta potential is calculated from mobility using accepted relationships between mobility and zeta potential. In the limit of high concentration of electrolyte, the relation between zeta potential and mobility is given by the Smoluchowski equation:

$$\zeta = \mu\eta/\varepsilon$$

ζ = zeta potential, μ = mobility, η = viscosity, ε = dielectric constant

For water at 25degC, Zeta potential(mV) = 12.8 x Mobility(μ/sec/volt/cm)

5. Maintenance

5.1 Introduction

This chapter contains the following information:

- Routine Maintenance
- Troubleshooting
- Safe Maintenance Practices

5.2 Safe Maintenance Practices

Observe all safety notices posted on the Zetatrac™ and throughout this manual.

WARNING

The Zetatrac™ must be serviced by a qualified and authorized service technician. Operation with cover off will present a laser radiation hazard. All personnel in the area must have suitable eye protection. Do not attempt service procedures not described in this manual. Contact your Microtrac representative if questions arise concerning operation, adjustment, or repair of any particle size analyzer.

5.3 Routine Maintenance

A program of regular inspection and maintenance helps to ensure continued optimum performance of the Zetatrac™ Particle Size Analyzer. The user can perform the routine maintenance procedures in this chapter at intervals that are determined by the amount and severity of use of the Zetatrac™.

Routine maintenance involves cleaning the sample-cell, and the optical probes.

Cleaning the Sample Cell and Optical Probes

To clean the sample cell and probe:

1. First thoroughly flush out all particles from the cell. The user should be especially careful if the particles are abrasive; in these cases, do not wipe the optical probe tips until the cell is generously flushed.
2. Wipe the probe tip gently to loosen accumulated material. Supplied with Zetatrac are either *'Microbrushes', *Chamois swabs, or both. Moisten brushes or swabs with deionized water.

   See the figures that follow. Note that the chamois swabs have a cut-out area on one side; insure that this side faces the 'electrode'. The swab will then fit between the electrode and the face of the optical probe, allowing for gentle cleaning of the probe. Use up-and-down motion, applying gentle pressure to the probe face.

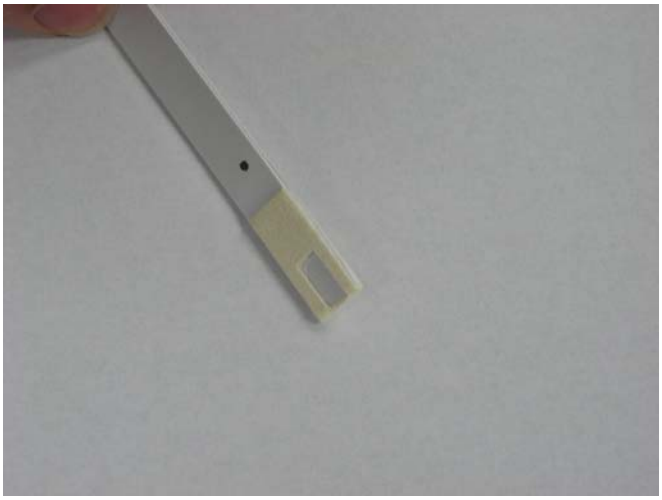
   When using microbrushes, again, apply gentle pressure to the face of the optical probe.

   Clean each optical probe individually using these techniques.

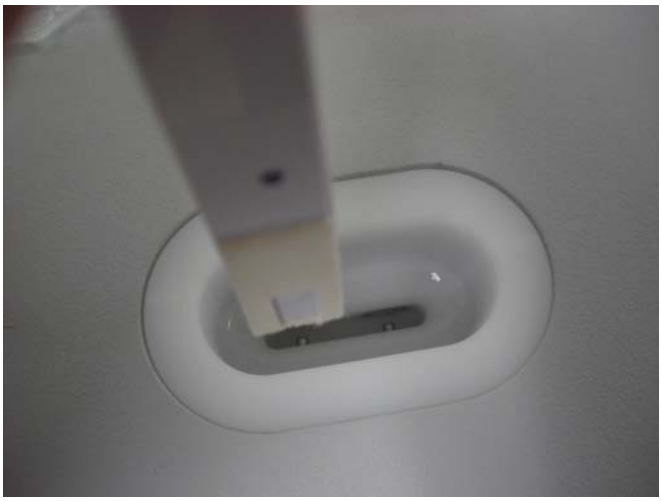
   Swabs and microbrushes are re-usable. Microtrac recommends using one swab or brush for each type of sample to be tested, and to regularly use new swabs/brushes, and dispose of used ones.

Flush the cell again several times to remove loosened material. The FLEX software can be used to judge the cell cleanliness; use the 'Background BKG' or 'Loading LD' toolbar buttons to activate a loading screen; these displays can be used as diagnostic aides to determine the quantity of particulate in the cell. See previous sections of this manual, and refer to Flex Operating Manual SW0003, for further use of these functions.

3. Other solvents may be more appropriate for cleaning, depending on the nature of the particles or contaminating materials. The user should consult Microtrac® Technical Support for use of other solvents.



Close-up view of chamois swab



Orientation of swab to cell for cleaning

5.4 Troubleshooting

The need for troubleshooting arises under the following conditions:

- Warning or error messages occur during operation.
- Spurious or inconsistent data.

Should these situations occur, the user may be able to troubleshoot and fix the problem. Before contacting Microtrac, the user should follow the steps below.

Troubleshooting: Is The Zetatrac Properly Connected, And Does It Power-Up Correctly?

Refer to previous sections on installation and connection; check that all connections are correct, and that connectors are fully inserted. Check for any visible damage to any connectors (bent connector, damaged connector housing, etc.). Note: The user should avoid connecting or disconnecting any Zetatrac connections or computer connections without first powering down the system. If a connector is found to be loose or disconnected, first power down the system, then reconnect the connector, then power up the system again.

Check that the AC power source is with specification, both for Zetatrac DC power supply and for computer.

Observe the Zetatrac front-panel optical system activity indicators:

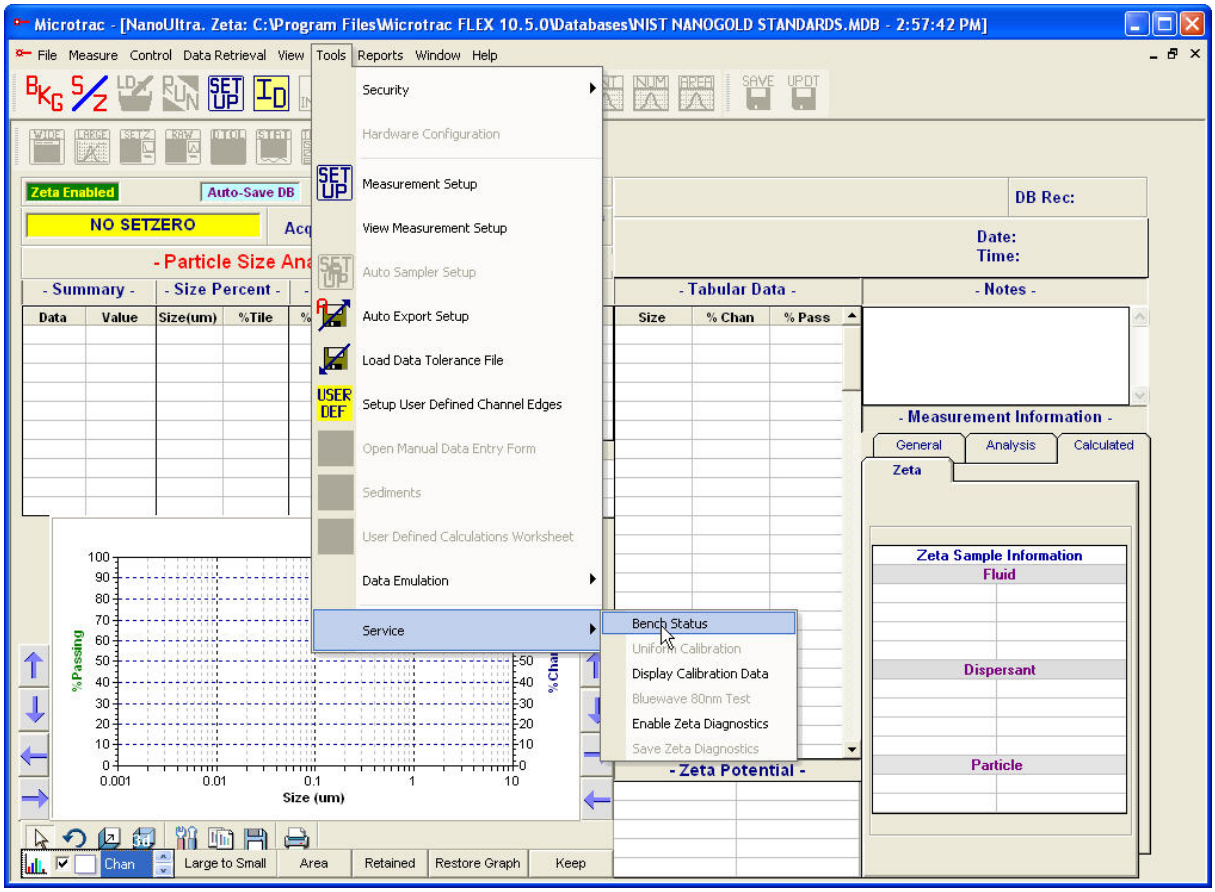
* With DC power supply plugged into Zetatrac, but with computer turned off, both indicators may be on, that is acceptable (it is advised that the laser switch on the rear of the Zetatrac be switched to 'Disable' if the unit is be left in this state overnight or for extended period of time);
* On power-up of computer, both indicators may turn off; that is acceptable; both may stay off until Flex software is opened;
* On opening of Flex software, only one indicator should be on at any one time. Indicators will change state during the course of a zetapotential measurement; this is acceptable. If, after Flex is opened, both indicators are off or both are on at any time, then there may be a hardware fault. Contact Microtrac® service for more information.

Troubleshooting: What Are Results of 'Bench Status' Check?

Zetatrac performance can be checked with Flex 'Bench Status' diagnostic function..

Prior to running Bench Status, if necessary, flush and clean the sample-cell as described in Maintenance section. Fill the sample-cell with clean fluid. Click 'Tools - Service - Bench Status' as shown below. Note: A Zetatrac 'Measurement' window must be open, and must be the active window, as shown below, in order to access the Bench Status function.

Note: At start of Bench Status function, both of the optical-system-activity indicators on the front of the Zetatrac should turn off. They should then recover function on completion of Bench Status, with only one of the indicators on. This is normal. IF the Bench Status function 'Cancel' button is clicked during the first portion of the Bench Status check, then both indicators will remain 'off', until the next Zetatrac function (Background, Loading Screen, SetZero, etc.) is performed; at that time normal function should return (only one indicator on).



Accessing Bench Status Function From An Active Measurement Screen

Bench Status Results Screen

Checked / Retrieved Parameters:

The following items are checked or retrieved during a Bench Status update:

- Bench Type: As a result of its legacy to the Nanotrac analyzer, the Bench Type returned will be 'Nanotrac'; this is acceptable even though this is a Zetatrac analyzer;
- Bench Serial Number: Assigned by Microtrac during Zetatrac production;
- Loading Calibration Factor and Reflected Power Factor: Fixed factors assigned during Zetatrac calibration;
- Cell Temperature: Active measurement of the latest sample-cell temperature, with or without fluid in the cell.

Tested Electronics Parameters:

Various parameters pertaining to Zetatrac electronics and the interaction with the PC-based data acquisition board are tested, and return a status of OK or BAD as a result of the Bench Status update. These include from 'Detector DC Level' down through 'LP Filter DC Level H'.

If any of these items indicate a BAD status, again check the cable connection between the Zetatrac and the computer-installed data acquisition board. Then run another Bench Status check. If the items remain in a BAD condition, contact Microtrac® service for more information.

Tested Optical Parameters:

Reflected Power

Optical probe cleanliness, or other probe issues, with affect ‘reflected power’; this may lead to a Bench Status Reflected Power status of BAD.

Clean the Zetatrac probe face and fill the sample cell with clean fluid. Repeat the bench status. If the Reflected Power remains high, an electrical alignment or other action may be required. Call Microtrac® service for more information. Possible causes are:

- Probe disconnected
- Fiber connection at laser not seated all the way
- Bad or broken probe optical fibers
- Defective/misadjusted Laser Drive Circuit
- Defective Detector

Laser Drive Current

If the laser drive current is greater than the maximum allowable current for the installed Laser Diode then the status is BAD. If the status indicates BAD, contact Microtrac® Service for more information. Possible causes are:

- Ambient operating temperature too high
- Laser drive circuit misadjusted
- Defective laser driver device
- Defective laser

Laser Monitor Current

If this value varies +/- 15% from the value recorded on the last electrical alignment then its status is BAD. If the status indicates BAD, contact Microtrac® Service for more information. Possible causes are:

- Laser drive circuit misadjusted
- Defective laser driver device
- Defective laser

Laser Power

If this value exceeds 100% then its status is BAD. If the status indicates BAD, contact Microtrac® Service for more information. Possible causes are:

- Laser drive circuit misadjusted
- Defective laser driver device
- Defective laser

Troubleshooting: Do Reference Samples Test Correctly?

Microtrac makes available particle size reference kits to assist in performance evaluation. These kits contain samples of known properties. In cases where the performance of the Zetatrac™ is in question, a sample of reference material may be analyzed, and, if desired, the results sent to Microtrac for evaluation.

Instructions for proper use of the reference material and expected results are provided with the kit. Do not test with materials from an expired kit; expiration dates are printed on the outside of the kit.

Some reference samples are provided with the Zetatrac system. Contact your representative or Microtrac to determined which reference materials are available, and to purchase additional reference materials.

Troubleshooting: Are Warning or Error Messages Displayed During SetZero or Run?

During a measurement operation, for example RUN or SETZERO, the Zetatrac system continuously monitors various critical parameters such as laser power level and total reflected power. If any parameters fall outside acceptable limits, appropriate warning messages are displayed during the operation. The results of the measurement should be considered suspect until the exact nature of the problem is determined. Examples of warning messages are described in the following paragraphs.

Reflected Power (Low or High)

Reflected power is the total amount of light (reflected plus scattered) reaching the photo-detector. The main component is the light reflected from the probe/fluid interface, but it includes the light scattered from the particles, which is normally no more than a few percent of the reflected light level.

With clear water in the cell, the reflected power is nominally 300, but can vary considerably (100 to 450 are acceptable levels for water) due to minute variations in the probe/fluid interface caused by such things as a buildup of thin layers of contamination on the probe face. Note: these values for reflected power are inherent to the Zetatrac system and software; these values may not be displayed to the user.

With fluids other than water, the expected reflected power is computed from the refractive index of the fluid and is typically lower than 300.

Variations in reflected power do not affect the accuracy of the particle size measurement. However, if the measured reflected power exceeds the limits for the fluid used, the REFLECTED POWER LOW (or HIGH) message is displayed after a run, indicating the need for corrective action. Possible causes of reflected power deviation and corrective actions are these:

- Dirty probe(s). Gently clean the cell and the optical probes.
- No fluid in cell. Supply clean fluid, the same as used for sample mixtures.
- Wrong refractive index value. Enter the correct value in the Flex 'Setup - Options - Analysis - Fluid Information' screen.
- Excessive particle concentration. Use properly diluted sample.

After cleaning the cell and probe or eliminating other potential causes, perform a SETZERO and a run. If the REFLECTED POWER LOW (or HIGH) warning still appears at the end of the run, other possible causes are:

- Unauthorized adjustment of the LASER PWR potentiometer
- Instrument malfunction

In this case, the Zetatrac will require service or repair. Contact Microtrac Technical Support for further information.

Laser Power (Low or High)

Possible causes of large changes in laser power values are:

- Laser degradation
- Fault in laser drive circuitry
- Unauthorized and potentially damaging adjustment of the LASER PWR potentiometer.

In this case, the Zetatrac will require service or repair. Contact Microtrac Technical Support for further information.

Excess Reflected Power

Possible causes of excess reflected power deviation and corrective actions are these:

- Dirty probe(s). Gently clean the cell and the optical probes.
- No fluid in cell. Supply clean fluid, the same as used for sample mixtures.

If this message persists, contact Microtrac Technical Support for further information.

Excessive Loading

During a RUN, reflected power level should not be more that 10% higher than the level measured during SETZERO. Light scattered from the particles is normally no more than a few percent of the total light arriving at the detector. As particle concentration increases, scattered light increases and can become a significant portion of the total light.

Increased scattered light causes certain second order effects, such as self-beating signals and multiple scattering signals, to become apparent. Such second order effects introduce errors in the detected frequency distribution and subsequently in the reported particle distribution. The typical effect is to give a distribution that is broader than normal and skewed toward smaller particle sizes.

The onset of second order effects is possible when a RUN reflected power level is 10% above that reported during a SETZERO. In this case, the particle distribution is still computed and presented, and the EXCESSIVE LOADING message is displayed during the run. This is a warning that the reported distribution may contain errors and the sample should be diluted and run again.

If this message then persists, contact Microtrac Technical Support for further information.

Invalid Temperature

This message indicates that the sample cell temperature, as determined by the sensing thermistor, is outside the 10^{o}C to 82^{o}C design limits of the instrument. If the user is neither cooling their sample below the low limit, nor heating their sample above the high limit, then this error message is likely due to a hardware problem that the user cannot fix. Additional diagnostic can be performed by running the Bench Status check, previously described. Note that 'Cell Temperature' is indicated. If Bench Status is performed with no liquid in the cell, then 'Cell Temperature' should give an approximate indication of surrounding ambient temperature; for example a typical temperature-controlled laboratory may show a readout of '22.0°C' or thereabouts. If this error persists, and the Bench Status indicates abnormal ambient temperature, then contact Microtrac Technical Support for repair or service information.

Unstable Temperature

The sample temperature must be known for an accurate computation of the particle size distribution, the sample temperature must be known, since the viscosity of the dispersing fluid is a function of temperature. The temperature is measured at the start and end of a run. The average value is used to compute the fluid viscosity. Temperature variations during the run add uncertainty to the viscosity and to the computed particle distribution. The *UNSTABLE TEMPERATURE* warning message is displayed at the end of a run if the computed uncertainty exceeds +/-0.3 standard (fourth-root) channel widths.

This error could be due to temperature fluctuations of the surrounding environments, particularly during especially long run times. If possible, move the Zetatrac to a more temperature-stable area, or provide protection or shrouding to prevent excessive temperature flow across the unit. Additionally, try running with the minimum recommended run-time. If the error persists, contact Microtrac Technical Support for further information.

5.5 Requesting Service

The Zetatrac contains no user serviceable parts. Zetatrac service or repair should be coordinated by contacting Microtrac Technical Support:

Microtrac Technical Support
12501-A 62nd Street North
Largo, FL 33773
(727) 507-9770

ATTENTION

The shipping cartons in which the Zetatrac was shipped have been optimized for this instrument. Microtrac recommends that all cartons and shipping materials be retained, in the event that the Zetatrac has to be returned to Microtrac for repair or service.

5.6 Requesting Parts and Accessories

As of release of this document, the available accessories for the Zetatrac include:

Microtrac P/N	Description
900383-001	Standard reference material; sizing standard; polystyrene spheres
400196-100	Standard reference material; zetapotential standard
400176-002	Disposable pipette, fine tip
400193-001	Microbrushes
900797-001	Accessory kit (contains all of the above)
900825-001	Chamois swabs

Parts and accessories can be ordered by contacting:

Microtrac Order Entry
148 Keystone Drive
Montgomeryville, PA 18936
TEL (215) 619-9920
FAX (215) 619-9932

www.microtrac.com