Matec Applied Sciences

Zeta-APS Operation Manual

Version 2.0

Particle Size and Zeta Potential Analysis….at High Percent Solids.

Measure Particle Size Distribution, Zeta potential, Percent Solids, Sound Attenuation and Speed Spectra, pH, Conductivity, and Temperature simultaneously and without the need for sample Dilution.

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Zeta-APS Frequently Asked Questions

1. ***How does the Zeta-APS measure particle size distributions?***
   The Zeta-APS accurately measures acoustic attenuation (dB/cm) vs. frequency of sound (1 to 100 MHz) of colloidal dispersions. These measurements are commonly referred to as Acoustic Attenuation Spectroscopy. The Zeta-APS also simultaneously measures speed of sound vs. frequency, percent solids, pH, conductivity, and temperature.
   The attenuation level, as well as, the shape of the acoustic attenuation curve shape is related to the particle size distribution (PSD). PSD's are calculated from the acoustic attenuation data using software developed and patented by Lucent Technologies. This Lucent Technologies software is based on the Epstein and Carhart (later refined by Allegra and Hawley) theory of acoustic attenuation. See the References section for suitable literature.

2. ***How does the Zeta-APS measure Zeta Potential (ZP)?***
   The Zeta-APS uses an electroacoustic technique called Electrokinetic Sonic Amplitude (ESA, invented by Matec Applied Sciences) to measure Zeta potential of particles suspended in liquids. The Zeta-APS' Zeta sensor applies short high-frequency (AC) pulses to the sample located within the electrode region of the Zeta sensor. These pulses last about 30 micro-seconds in the frequency range 0.5-3.5 MHz, and with amplitudes 100-600 volts. The particles "jiggle" back and forth due to their surface electric charge which produces an output sound wave of the same frequency as the applied sound wave –provided there is a particle/solvent density difference of at least 2%. The sample can be mixed and/or pumped during the measurement without interfering with the ESA measurement.

3. ***How is a sample run? What is the sample-analysis procedure?***
   Turn on the Zeta-APS unit using the power switch on the front panel of the Zeta-APS control unit (main unit). If you wish to measure both size and Zeta, two green power lights should be lit around the power switch. The bottom LED is for the Zeta power supply which can be turned on/off on the rear of the unit.
   Launch the Zeta-APS software by double-clicking on the Zeta-APS software shortcut located on the PC Windows desktop.
   Allow the Zeta-APS electronics to warm up for a minimum of 30 minutes.
   Pour your sample into the sample cell. Make sure that the sample meets the sample requirements (see sample description below).
   Verify that the sample fully covers the transducer(s).
   Note: Minor contaminant matter present in the sample cell prior to pouring the sample will not affect the resulting Zeta-APS data. This is due to the fact that Zeta-APS measurements are made on concentrated samples. As a result, there is no need to clean the sample cell very thoroughly.
   Turn on, and adjust the speed of the sample mixer by flipping the small metal switch located on the left side of the sample cell and by rotating the black knob on the front panel. Suitable mixing speeds are such that the particles are maintained

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in suspension, the sample is homogeneous, and air is not entrapped into the sample.

Note: avoid inserting your fingers into the sample cell. The mixer propeller may impact your fingers. Also, the reflector moves back and forth during sample analysis. Your hand may be pressed against the sample cell walls by the reflector. Using a soft object such as a disposable plastic transfer pipette or a soft brush, remove any air bubbles, if present, from the transducer and stainless steel reflector surfaces facing each other. Note: the reflector surface facing away from the transducers is irrelevant. Avoid scratching the transducer glass rods and the reflector as scratches may reduce Zeta-APS analysis accuracy. You can also rotate the Zeta sensor to dislodge air bubbles form the electrode area.
You can run the Zeta-APS unit in one of three modes: Particle Size, Zeta Potential, or Combined Size and Zeta.
Click the Zeta-APS software "Meas Single Point" toolbar button. Alternatively, you can select "Measurement/Single-Point" from the file menu. This option allows you to perform a PSD and/or Zeta measurement.
For APS sizing, select the appropriate matrix file for your samples. The matrix file contains the acoustic-attenuation properties of samples composed of a given particle and solvent materials (see matrix section in the matrix-file section of the manual). Examples of suitable matrices are "silica particles in water", "Alumina in water", and "carbon black in dodecane". Matrices are built separately by clicking "Build Matrix".
Select your sample solvent. Examples are water, dodecane, and Isopar. *Note: Non-aqueous solvent spectrum files must be created prior to sample analysis. This is achieved by placing the non-aqueous solvent in the sample cell, then choosing Calibration/Solvent-Spectrum. The solvent attenuation spectrum is subtracted from that of the sample during the PSD computations.*
Enter the PSD data file name. Data files can be long, and contain spaces (do not use periods). You can either type it in the entry box or you can click browse, select a similar file name, then modify it, and click Save.
The PSD data file will be automatically saved at the end of the sample run. *Note: two files will be saved; a filename.ASP file will contain the PSD data; a filename.ASV will contain the acoustic attenuation or raw data.*
Enter any pertinent comments.
Click "Sample Ready. Analyze!" in order to start the measurement.
The acoustic attenuation data will be collected and the resulting PSD displayed at the end of the run. The PSD data file will be saved automatically. You can click "Report" in order to view a detailed PSD report.
For Zeta measurements, click "Reuse Stored Setup" if you already have created a Setup; otherwise, click "Generate New Setup". A Setup contains information on particle and solvent properties plus sample-analysis data/procedures.

4. ***What types of samples can be analyzed on the Zeta-APS?***
Examples of suitable samples are inks, ceramics, CMP slurries, minerals, metal oxides, coatings, organic and inorganic pigments, bio-colloids, polymer latex,

pharmaceuticals, W/O and O/W emulsions, and others.
Because sound travels through all material media, the Zeta-APS can perform sizing measurements on a wide variety of aqueous and non-aqueous colloidal dispersion samples in the particle size range 10 nm to 100 microns. The suitable sample percent solids range is 0.1 to 50% by volume. Zeta potential measurements cannot be made on samples with less than 2% particle/solvent density difference.
Even though the Zeta-APS is capable of analyzing samples without the need for sample dilution, some samples are more easily analyzed at percent solids levels below 10% volume. One reason is that sample handling plus sample cell cleaning are easier. Another reason is that some samples attenuate sound very strongly; such samples require that one use lower particle concentrations.
The Zeta-APS successfully analyzes the PSD's of neutrally buoyant, high-density, and low density particles. There is no minimum particle/solvent density requirement (Zeta requires 2% minimum density difference).
The Zeta-APS can successfully perform sizing measurements on samples regardless of their Zeta potential or particle electric surface charge level, including samples at or near their Iso-Electric Point (IEP). Zeta potential measurements cannot be made on samples with less than 2% particle/solvent density difference.

Samples can be poured into the sample cell. The on-board mixer keeps the sample thoroughly mixed, with the particles in suspension. An additional magnetic stirrer can be used for added mixing. Samples can also be pumped through the sample cell as well by using a peristaltic or other suitable pump.

5. ***Do samples have to be highly electro-statically charged for Zeta-APS sizing measurements? Does the sample's Zeta potential have to be high?***
   No. The Zeta-APS can successfully measure particle size regardless of the sample's particle surface charge or Zeta potential.

6. ***How long does a Zeta-APS analysis take?***
   A Zeta-APS measurement takes about 10 minutes, depending on the sample's PSD width. Some Zeta-APS complicated-PSD computations take a few minutes longer.

7. ***Can I install the Zeta-APS software on an unrelated PC, such as the one in my office?***
   Yes. Zeta-APS users may install the Zeta-APS software on PC's other than the Zeta-APS's. See the Software Installation appendix.

8. ***What sort of sample preparation is required? Should samples be sonicated?***
   Sample preparation is related to the purpose of the sample analysis. Users should sonicate their samples prior to analysis if they wish to measure the particle size of the primary particles, as opposed to any aggregates. Sonication should not be performed if one wishes to measure particle aggregation levels. Do not sonicate

samples in the sample cell as it may damage the ultrasonic transducers.

9. ***Should sample measurements be made at a fixed temperature?***
   Particle size, Zeta potential, and especially percent solids data tend to be more accurate and reproducible if performed at a fixed temperature such as 25 or 30 degrees C. Accuracy is increased if sample analysis is performed at the same temperature as the sample-property matrix (see software section, matrix). Sample temperature affect the attenuation level, as well as, the ESA measurement. Attenuation increases as the sample temperature decreases. As a result, the percent solids data is mainly affected. In order to maximize PSD-data accuracy, it is highly recommended that the sample temperature not vary by more than about half of a degree C during the Zeta-APS measurement. The Zeta-APS measures the sample temperature several times during sample analysis. The mean temperature value is used in the PSD computations.

   The onboard Zeta-APS sample heater provides sample temperature-control capability. The sample mixer should be used in order to prevent temperature overshoot, as well as, ensure temperature homogeneity in the sample cell. The heater set temperature can be set by clicking *System/Hardware Setup/.* We do not recommend set temperatures higher than 35C since they may adversely affect the transducers. A suitable set temperature is one or two degrees above ambient.

   The sample temperature is displayed on the Temperature Controller unit located on the Zeta-APS front panel.

10. ***Does the Zeta-APS need to be calibrated with particle size calibration standards?***
    No. The Zeta-APS measurement uses fundamental acoustic attenuation theory in order to compute particle size distributions.
    There is an important system calibration (using DI water) that must be performed periodically in order to ensure maximum data accuracy.
    Zeta potential is calibrated using either Ludox TM 2.5% volume or a high molecular weight salt.

11. ***Can I overlay Zeta-APS data plots?***
    Yes. Click the Overlay toolbar button. Add file names to be overlaid. The plot colors, curve type and many other features can be changed by right clicking on the data list on the right side of the graph.

12. ***Can the Zeta-APS measure sound speed? Is there a quick way?***
    Yes. The Zeta-APS measures sound speed automatically when analyzing a sample. Alternatively, you can quickly measure sound speed by clicking System/Configure-System/New-Hardware-Setup/Meas-Xducer-to-Home.

13. ***What do I do if my sample attenuates too much sound?***
    The Zeta-APS software initially measures the attenuation level of your sample for

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each transducer (high- and low-frequency). If the attenuation level is too high for the high frequency transducer, the software automatically switches to the low-frequency transducer only. A message indicating so is displayed. This is done in order to maximize the accuracy of the measurement.

Some samples may be too attenuating. If so, the software will prompt you to dilute the sample in order to make a successful measurement.

One less-convenient alternative to diluting the sample is to use shorter measurement paths. These result in lower total sound attenuation. You can either modify your current path file or create a new path file.

A convenient option to create a new path file is obtained by clicking System/Configure-System/New-Hardware-Setup/Create New Path File. Follow the instructions on the screen in order to create a new path file. Basically, you just have to choose a new path filename and select path locations that are suitable. Select path locations that do not coincide with secondary echoes. The shortest path distance allowable is 0.15 cm.

Alternatively, file C:\Z-APS\aps.ini\ contains the name of your path file. You can use Notepad to modify current aps.ini file. You must exit the Zeta-APS software before editing this file.

14. ***When should the Resonant Zeta Probe (RZP) be used?***

    With the Zeta-APS (and also the Zeta*Acoustic*), the RZP is recommended for non-polar organic solvents. The Non-Resonant probe can be used for aqueous, as well as, alcohol-based samples. Note: Matec's Zeta*Finder* instrument can only use the RZP probe. The Zeta*Finder* cannot use the Non-Resonant Zeta Probe.

15. ***What is Particle Frequency in the PSD graphs?***

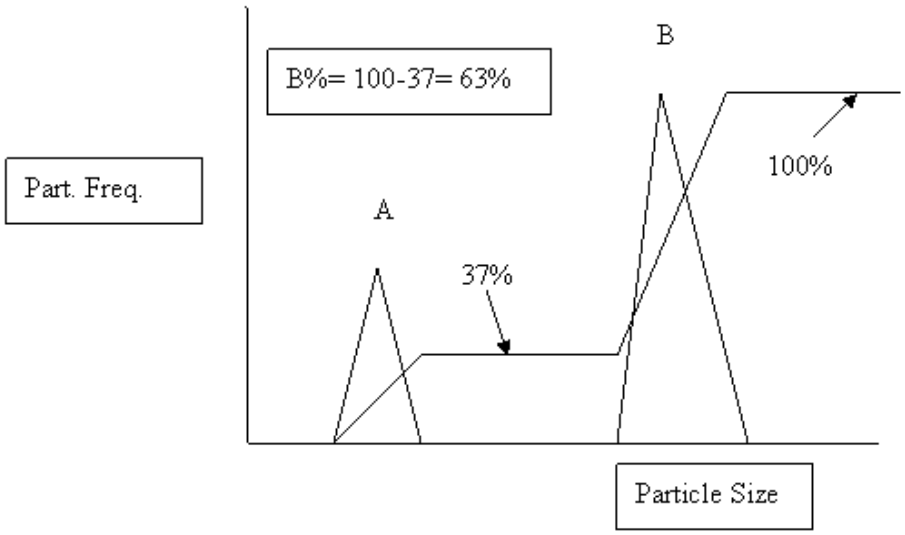
    "Particle Frequency" and "Volume Percentage" are different. The Zeta-APS can produce absolute and relative PSD's. We prefer to show relative PSD's.

    An absolute PSD shows the actual particle volume percentage. However, absolute PSD's are sometimes difficult to compare between different samples (overlay). The reason is that samples with different percent solids will show different PSD-peak heights.

    Relative PSD's as currently shown by the Zeta-APS are normalized so that the tallest peak or peaks have a height of 100 "frequency units". These are not percentage values because there can be two or more peaks with the same 100 value. Also, next to the 100 point, there are values of 98, 92, 86, and so on. If you add all these together, you would obtain much more than 100% for the sample.

The particle frequency can be used to compare two populations. For example, a 37% peak has half the percentage of a 74% peak.

If you need to know the percentage of a population, you should use the cumulative distribution (CD). You can subtract the CD values at both ends of each peak. This gives you the percentage of this peak. See example graph below.



16. ***How can the percentage of a particle-size population be calculated?***

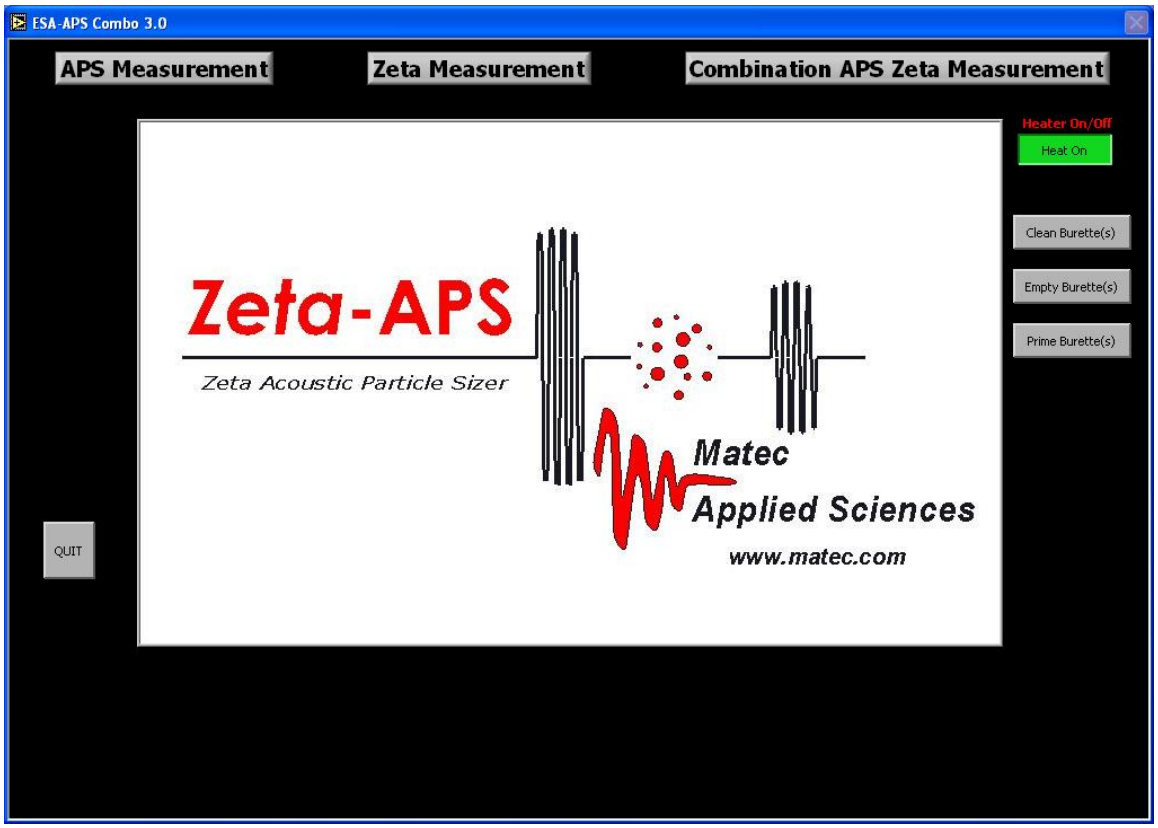
See "Particle Frequency" question above.

17. ***Can the particle size data be imported into MS-Excel files?***

Yes. Each sample produces three files. The sizing data is in file *.aspdat. You need to open this file from Excel and use Tab+space delimiter in order to use it. Copy rows 10-509 (if acoustic matrix has 500 classes) of columns A, and B to cell A5 of the Excel file (Matec gladly provides a master file preloaded with a graph). The mater file's graph tab (lower left) shows the PSD graph (updated automatically upon pasting the data).

Zeta-APS Sample Analysis

The Zeta-APS instrument can be operated in one of three modes: Particle Sizing, Zeta potential, and Size and Zeta combined ("Combo"). One of these modes can be chosen upon launching the Zeta-APS software as shown below.



Once you select a mode, you can easily select a different Sizing/Zeta mode by clicking on "Quit" (without having to exit completely the Zeta-APS software). The sample heater can be turned on/off by clicking "Heat On" which is useful is the heater will be dry for more than a few minutes. This lengthens the heater's lifespan.

pH, temperature and conductivity are automatically measured during sizing/Zeta measurements. "Burette" refers to the digital syringe pump that can be used to deliver acid/base/surfactants or other reagents into the sample at specified rates.

Any particle size/Zeta measurement is best performed while providing sample mixing, at a sample temperature within 0.5 degrees from the pre-selected set point. The on-board heater provides heating capability (not cooling).

Zeta-APS Quick Guide to Particle Size Analysis

This section describes the procedure for making particle size distribution (PSD) measurements. Later, Zeta potential (ZP) measurements are described. Combined PSD and ZP analysis procedures are presented afterward.

PSD analysis is quick and simple with the Zeta-APS. Prior to sample analysis, the Zeta-APS unit must be set up properly. All the cables between the sample cell, the control unit, and the PC must be connected to their respective ports. The temperature probe must be used when performing sample analysis. You may also use the optional pH, and conductivity probes. Please always follow safe instrument operation procedures.

1. Turn on the Zeta-APS unit using the power switch on the front panel of the Zeta-APS control unit (main unit).

2. Launch the Zeta-APS software by double-clicking on the Zeta-APS software shortcut located on the PC Windows desktop.

3. Allow the Zeta-APS electronics to warm up for a minimum of 30 minutes.

4. Prior to running samples, it may be necessary to perform an APS-sizing System Calibration (about once a month). Pour DI water into the sample cell to cover both xducers, then click "System/System-Calibration" to achieve this. You may also need to calibrate the pH and conductivity probes if pH and conductivity data are desired. Transducer realignment may be necessary if the PSD data is inaccurate. See Zeta-APS Calibration.

5. Pour your sample into the sample cell. There are two sample cups: "combo" (larger volume) and APS (smaller volume). If Zeta is not needed, you can use the APS cup for smaller sample volume.

   Make sure that the sample meets the sample requirements (see sample description below).
   Verify that the sample fully covers the xducers, about half a centimeter from the top of the cup.
   Note: Minor contaminant matter present in the sample cell prior to pouring the sample will not affect the resulting Zeta-APS data. This is due to the fact that Zeta-APS measurements are made on concentrated samples. As a result, there is no need to clean the sample cell very thoroughly.

6. Turn on, and adjust the speed of the sample mixer by flipping the small metal switch located on the left side of the sample cell and by rotating the black knob on the front panel. Suitable mixing speeds are such that the particles are maintained in suspension, the sample is homogeneous, and air is not entrapped into the sample.

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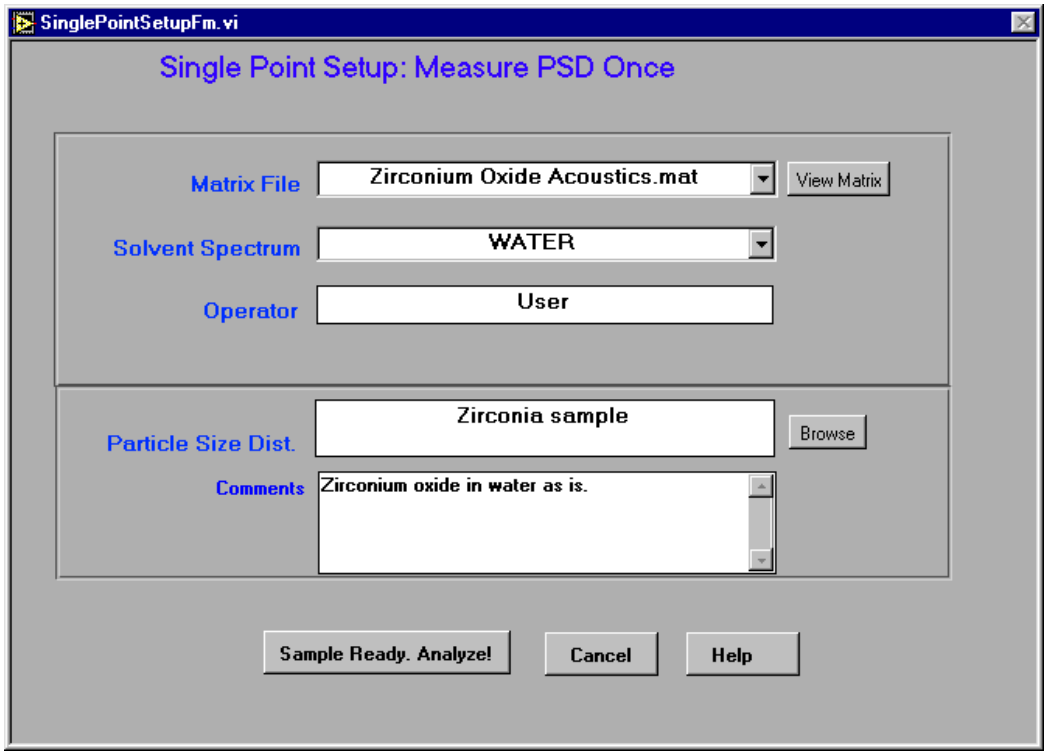
Note: avoid inserting your fingers into the sample cell. The mixer propeller may impact your fingers. Also, the reflector moves back and forth during sample analysis. Your hand may be pressed against the sample cell walls by the reflector.

7. Using a soft object such as a disposable plastic transfer pipette of a soft brush, remove any air bubbles, if present, from the transducer and stainless steel reflector surfaces facing each other. Note: the reflector surface facing away from the transducers is irrelevant. Avoid scratching the transducer glass rods and the reflector as scratches may reduce Zeta-APS analysis accuracy.

8. Click the Zeta-APS software "Measure Single Pt" toolbar button. Alternatively, you can select "Measurement/Single-Point" from the file menu. This option allows you to perform a PSD measurement. See figure below.



9. Select the appropriate matrix file for your samples. The matrix file contains the acoustic-attenuation properties of samples composed of a given particle and solvent materials (see matrix section in the matrix-file section of the manual). Examples of suitable matrices are "silica particles in water", "Alumina in water", and "carbon black in dodecane".

10. Select your sample solvent. Examples are water, dodecane, and Isopar. Non-aqueous solvent spectrum files must be created prior to sample analysis. This is achieved by running the non-aqueous solvent as a sample, then saving the resulting sample file. The solvent attenuation spectrum is subtracted from that of the sample during the PSD computations.

11. Enter the PSD data file name. Data files can be long, and contain spaces. You can either type it in the entry box or you can click browse, select a similar file name, then modify it, and click Save.
    The PSD data file will be automatically saved at the end of the sample run. Note: two files will be saved; a *filename*.ASP file will contain the PSD data; a *filename*.ASV will contain the acoustic attenuation or raw data.

12. Enter any pertinent comments.

13. Click "Sample Ready. Analyze!" in order to start the measurement.
    The acoustic attenuation data will be collected and the resulting PSD displayed at the end of the run.

    Note: the Zeta-APS software initially measures the attenuation level of your sample for each transducer (high- and low-frequency). If the attenuation level is too high for the high frequency transducer, the software automatically switches to the low-frequency transducer only. A message indicating so is displayed. This is done in order to maximize the accuracy of the measurement.

    Some samples may be too attenuating. If so, the software will prompt you to dilute the sample in order to make a successful measurement.

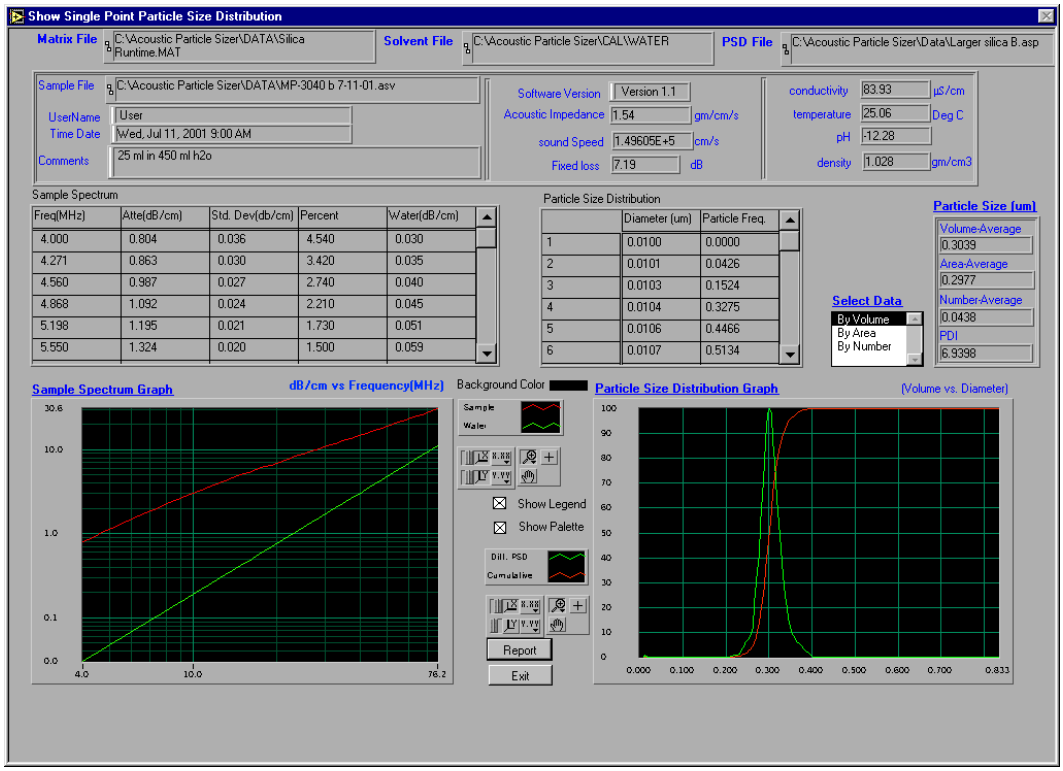
    One less-convenient alternative to diluting the sample is to use shorter measurement paths. These result in lower total sound attenuation. You can either modify your current path file or create a new path file.

    A convenient option to create a new path file is obtained by clicking System/Configure-System/New-Hardware-Setup/Create New Path File. Follow the instructions on the screen in order to create a new path file. Basically, you just have to choose a new path filename and select path locations that are suitable. Select path locations that do not coincide with secondary echoes. The shortest path distance allowable is 0.15 cm. The longest path distance depends on the type of motion stage on your Zeta-APS unit. Currently, there are two models with maximum distances of 5 and 2.5 cm.

    Alternatively, file C:\Acoustic Particle Sizer\aps.ini\ contains the name of your path file. You can use Notepad to modify current aps.ini file. You must exit the Zeta-APS software before editing this file.

    The PSD data file will be saved automatically. You can click "Report" in order to

view a detailed PSD report. See the figure below.

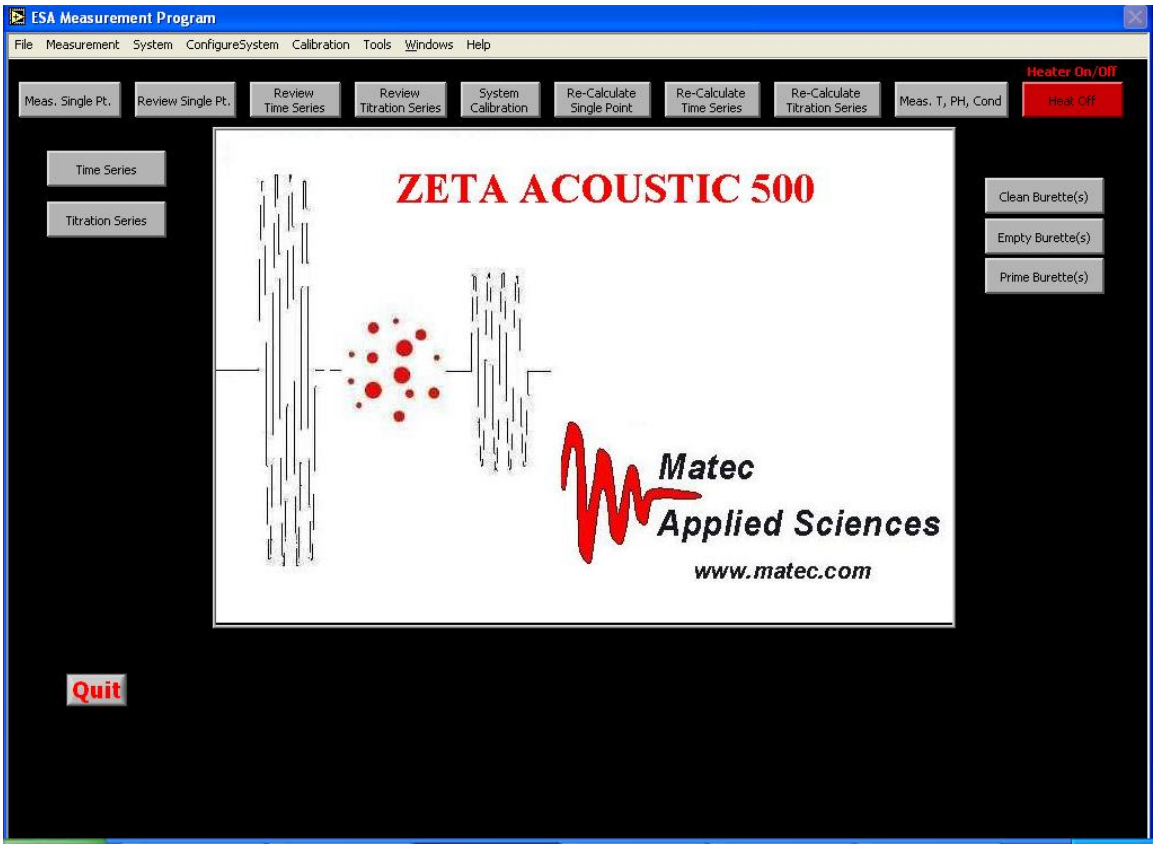


14. You can zoom-in on the graphs by clicking and dragging. Click on the tool palette in order to select zoom tool, axis format and precision, logarithmic or linear, plus other graph features. You can also change the plot features such as color, thickness, etc. by right clicking on the plot legend.

15. You can print a report by clicking “Report”, then clicking “Print” in the report window.

Zeta Potential Analysis

You can choose to make Zeta potential measurements without simultaneously measuring particle size by clicking "Zeta Measurement" upon launching the Zeta-APS software. You can also do so by clicking "Quit" from the APS or Combo modes. Please refer to *Appendix IV* for Zeta cabling connections and *Appendix V* for a short theoretical discussion on Zeta potential. The Zeta Home Window is as follows:



The sample heater can be turned on/off by clicking "Heat On" which is useful if the heater will be dry for more than a few minutes. This lengthens the heater's lifespan by preventing overheating. Quick measurements of pH, temperature, and conductivity can be made by clicking "Meas. T, pH, Cond".

Particle Size Correction

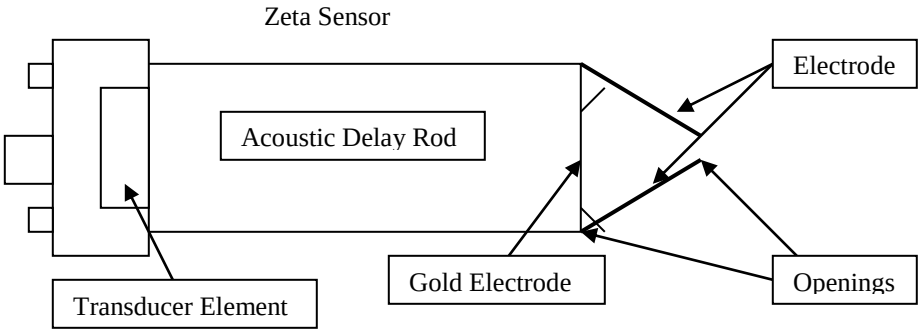
The calculation of Zeta potential requires a correction for particle size (inertia) effects. Particle inertia is more noticeable for particles over one micron in size. Increasing particle inertia (size) reduces the ESA amplitude (particles more slowly than expected) while increasing the phase angle of the particle's ESA response. The effect on the Zeta data is that, without a particle size correction, Zeta would be underestimated for larger particles.

The Zeta-APS offers three options for particle size correction as follows: (i) user-entered particle size (area-weighted mean particle size recommended), (ii) Automatic Acoustic correction, and (iii) APS-measurement particle size. In the Zeta-only mode, only options (i) and (ii) are available. The three options can be used in Combo mode.

The Automatic Acoustic Correction is obtained by making limited Acoustic-attenuation measurements. If so wished, the user can use the same Zeta data file to re-calculate the Zeta potential under these different particle-size options and Save As a different file name.

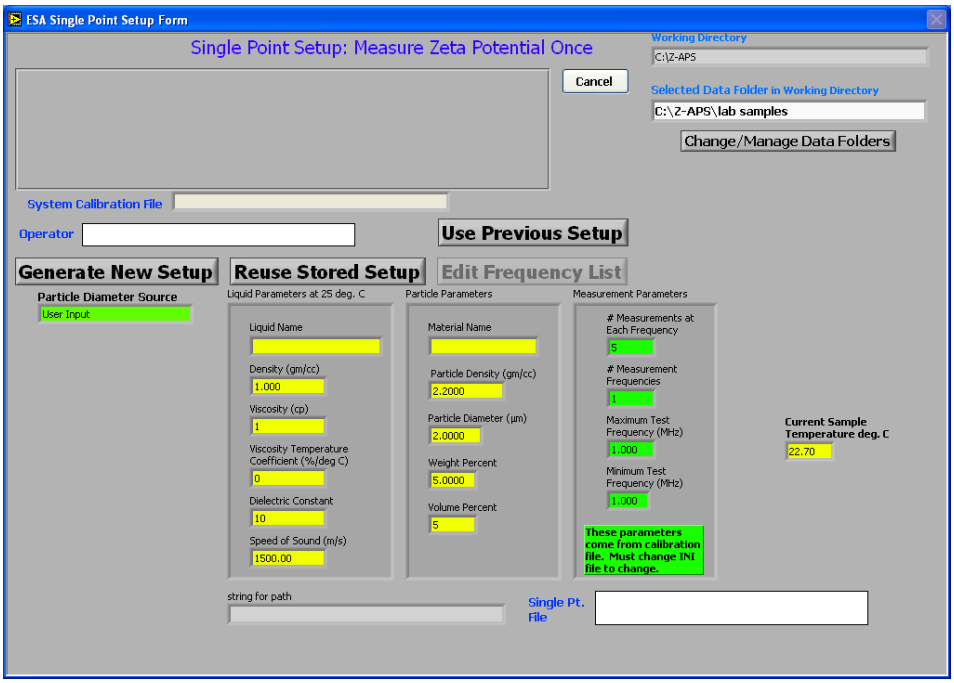
Zeta Potential Single Point

Single point is used to make a single Zeta potential measurement of a given sample. Make sure the sample covers the openings at the tip of the probe. It is advisable to rotate the probe to dislodge any air bubbles trapped at the tip of the probe. See figure below.

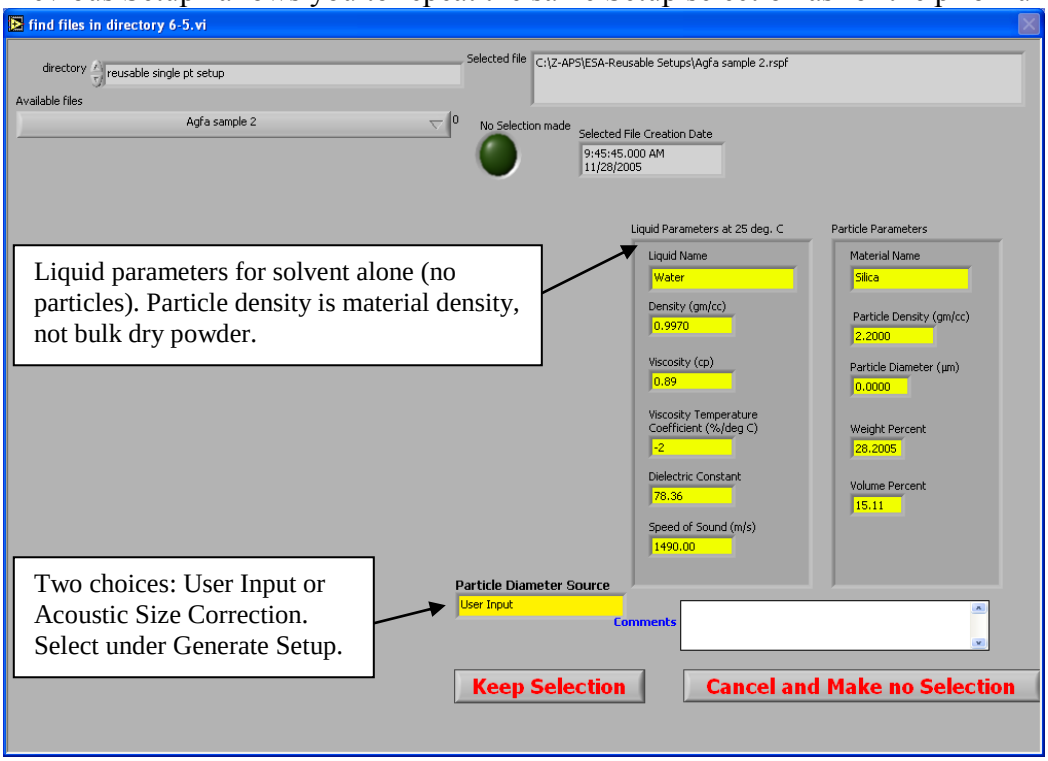


You can make measurements in the regular Zeta-APS sample cup or in a separate container (beaker) for smaller sample volumes. If using a separate container, provide sufficient sample mixing via a magnetic stirrer or other suitable means. You should also insert the temperature sensor and the heater unit in the sample in order to measure at or near the temperature set point.

Simultaneously, pH, temperature and conductivity are measured. Upon clicking Single Point, the sample's current temperature is compared to the set point value. Upon reaching it, the following window is displayed.

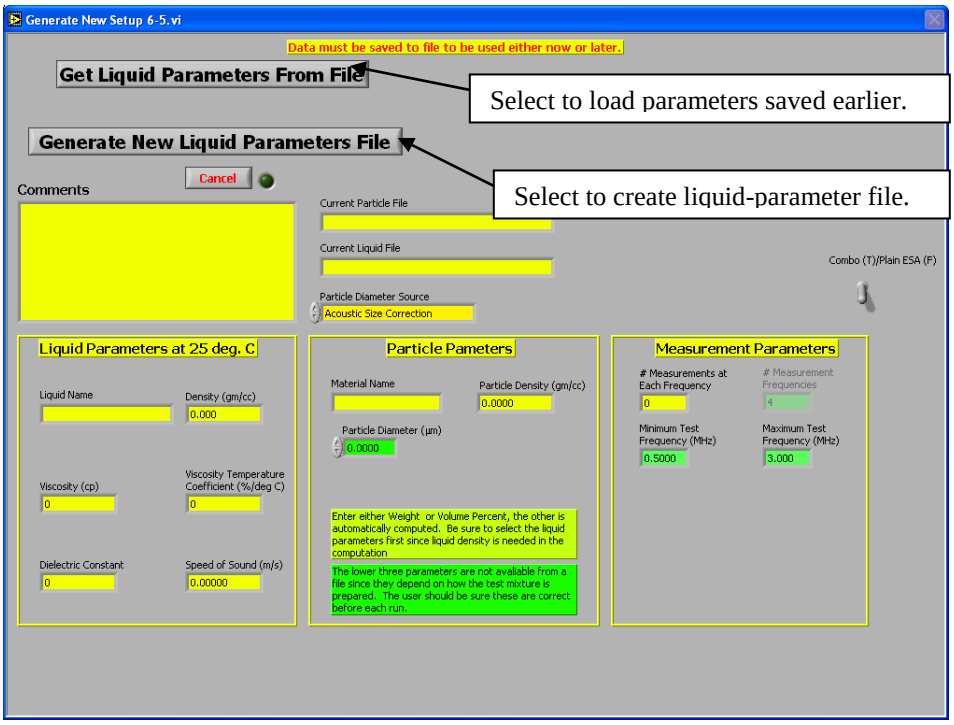


Select "Reuse Stored Setup" if you have previously created an Analysis Setup for the sample to be analyzed. The Reuse Stored Setup window is shown below. "Use Previous Setup" allows you to repeat the same Setup selection as for the prior run.



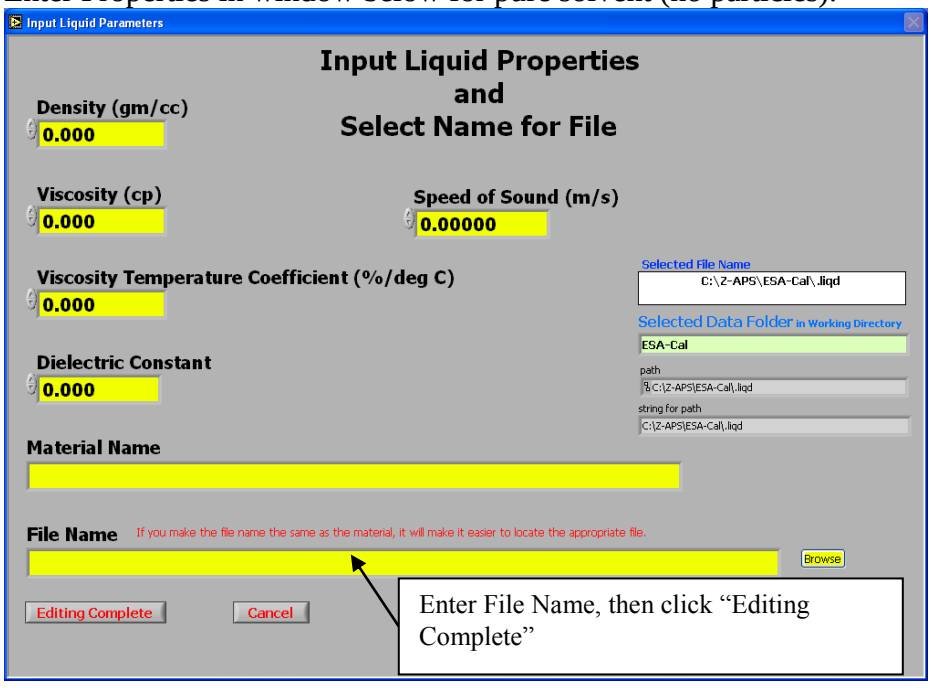
Click under "Available Files" to select a Setup file, then click "Keep Selection". The "Particle Diameter Source" selection is made when creating the Setup under

"Generate New Setup". The Solvent and Particle properties are for each individual phase. Use particle material density, not dry (bulk) powder.



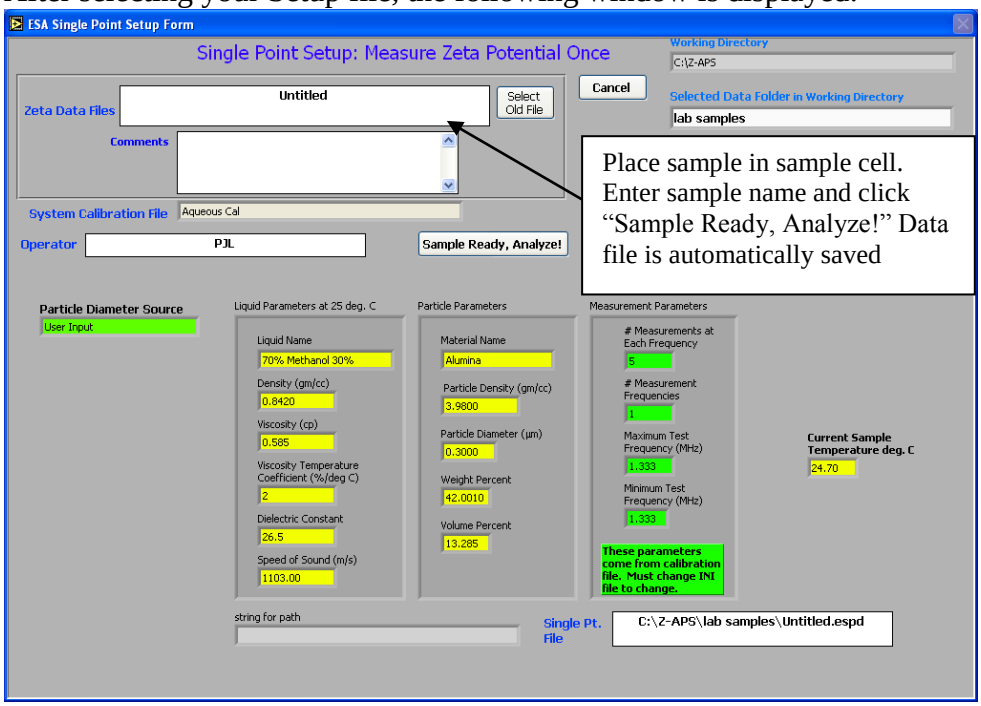
Creating Liquids (solvent)-Property File:

1. Click "Generate New Liquid Parameter File".
2. Enter Properties in window below for pure solvent (no particles).

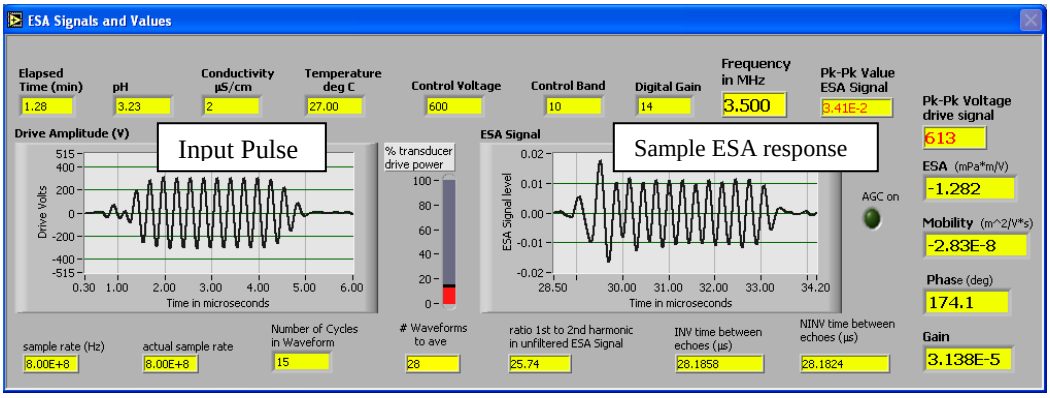


3. Click “Editing Complete” to save data.

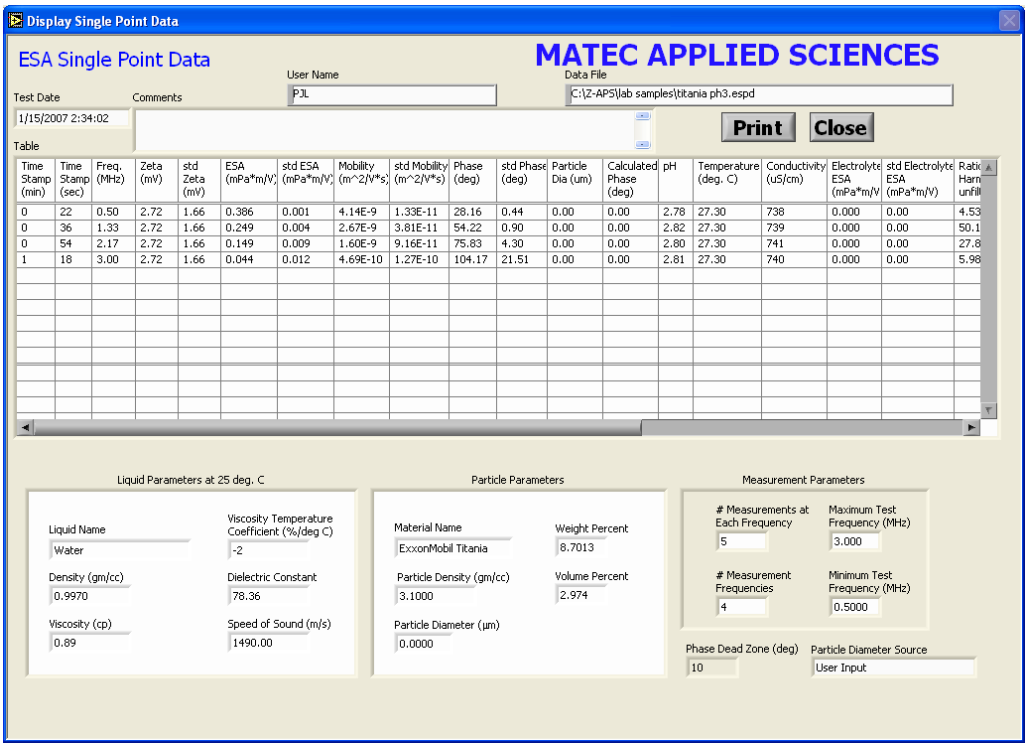
After selecting your Setup file, the following window is displayed:



The measurement proceeds automatically. The window below shows the ESA waveforms: input, and sample (colloid) response.



The window below shows an example of data output:



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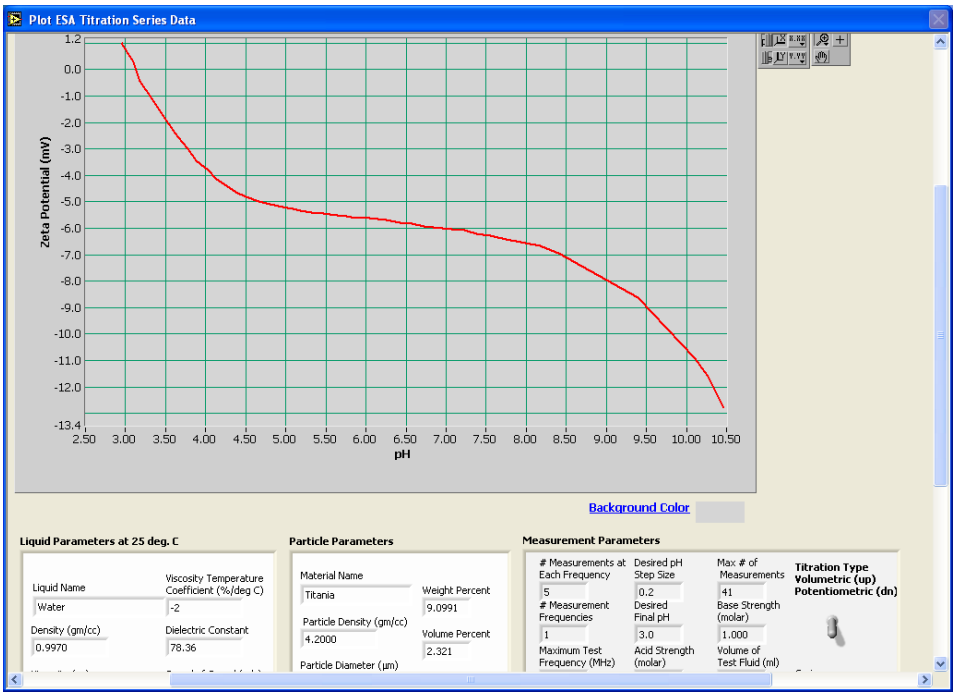
Time Series and Titrations

Time series allows you to make repeat measurements of the same sample. Simply specify the number of runs and the time between runs. Graphical data is produced showing the different parameters as a function of time. This feature is useful for monitoring a sample's shelf stability. Suitable sample mixing must be provided in order to avoid significantly particle settling over time which can noticeably affect the data.

Potentiometric titrations allow you to add acid or base to a sample in specified pH steps. This measurement allows easy determination of a sample's Iso-Electric Point (IEP). Simply specify the target pH, the pH step (usually 0.2 units), and the equilibration time between additions (usually 30-90 seconds). Graphical data is produced for easy evaluation of pH effects.

Volumetric titrations are similar to potentiometric ones except that a volume of reagent (surfactant, electrolyte, etc) is specified. The figure below shows a potentiometric-titration graph.



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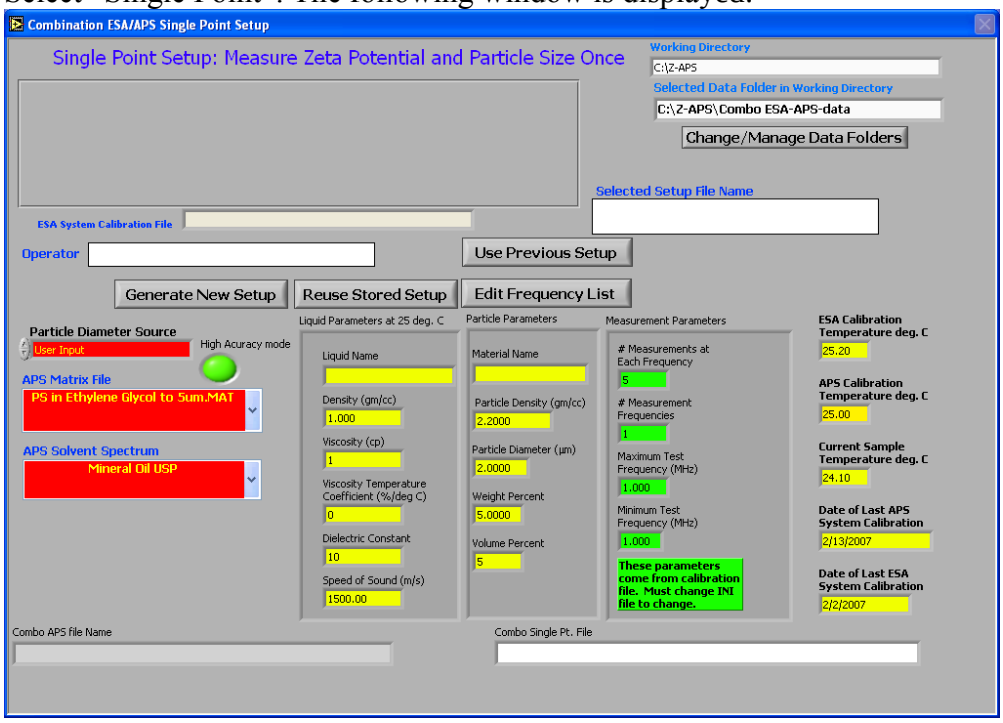
Combined Particle Sizing and Zeta potential Measurements

This section describes how to make simultaneous measurements of Particle Size Distribution (PSD), Zeta potential (ZP), percent solids, pH, temperature, conductivity, and sound speed and attenuation spectra. The reader will be referred frequently to the Sizing and Zeta sections above since there are various commonalities. See Zeta Time Series and Titrations for these measurements in combo mode.

Single Point Measurement

1. Place the sample in the sample cell. Ensure that the sample covers both transducers (about 0.5 cm from the top of the cell), and that the Zeta sensor's tip openings are fully covered.
2. Launch the Zeta-APS software and select "Combined Size and Zeta" mode.
3. Select "Single Point". The following window is displayed:



4. For particle sizing, select a suitable APS acoustic matrix, and solvent spectrum (see APS section).
5. For Zeta potential, select Reuse Stored Setup to select an existing Zeta method; alternatively, click Generate New Setup to create a new one. Select Use Previous Setup to repeat your selection for a prior sample.
6. Particle Diameter Source is used in the Zeta computations for particle-inertia corrections (see Zeta section). The choices are User Input, Acoustic (from Zeta measurement), and APS (from APS measurement).
7. Data output is similar to the Zeta output.

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Software

The Zeta-APS software is based on the National Instruments Labview® Graphic User Interface software system. The Zeta-APS also makes use of Microsoft C®-based application libraries (dll's). You can make any software selection by pointing and clicking via PC mouse.

Installation

Please refer to the Software Installation Appendix of the manual.

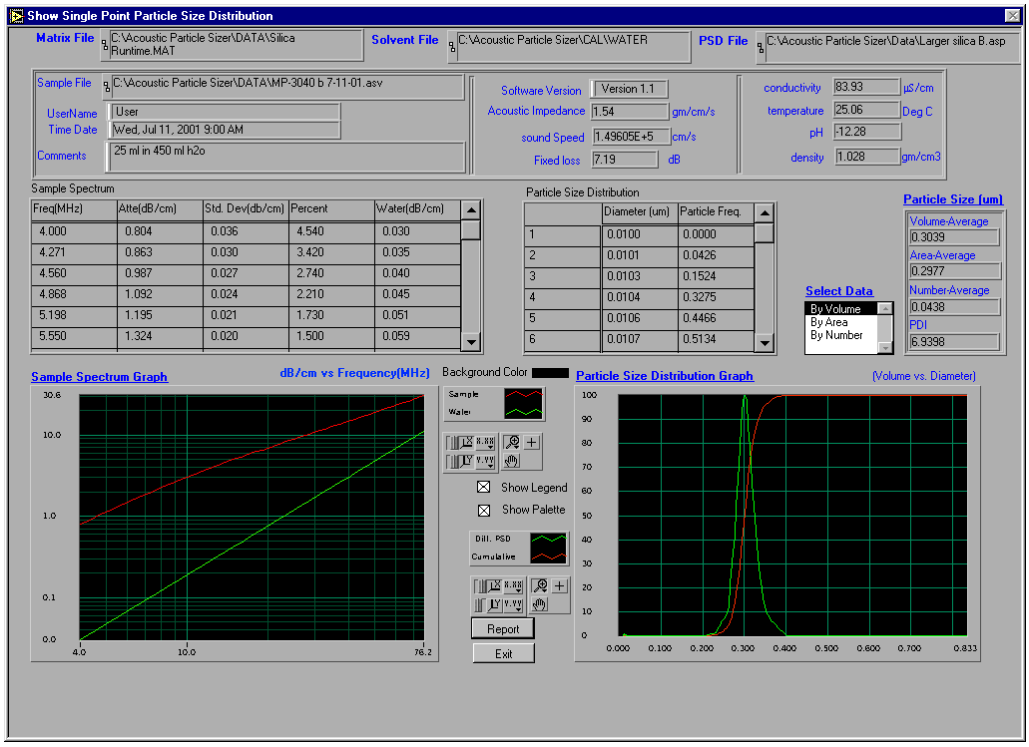
Operation

1. To launch the Zeta-APS software, double click on the Zeta-APS icon located on the PC desktop. Alternatively, you can launch Windows Explorer, and then double click on file "APS.exe" found in the "C:\Acoustic Particle Sizer" folder.

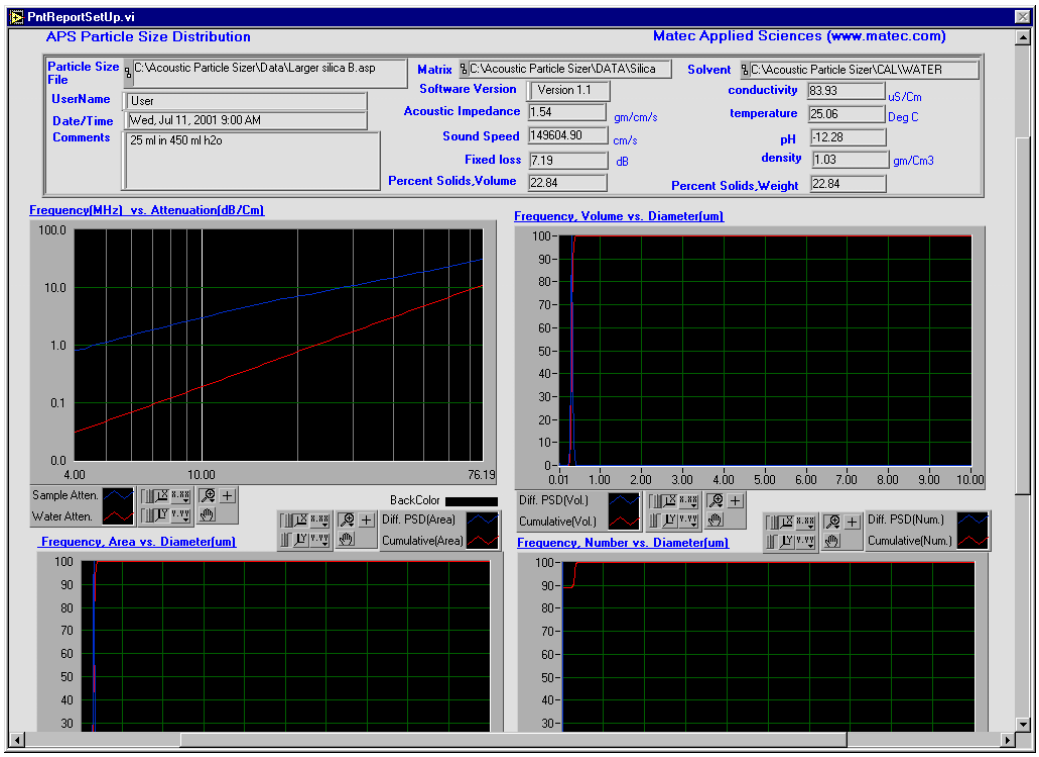
2. Enter a password at the Login window if the password feature is enabled; otherwise, ignore the password entry. You may enter a user name in either case. Click OK.

3. Your next step depends on what you would like to do, e.g., run a sample, view stored data, re-calculate saved data, overlay data plots, create a new matrix, perform calibrations, or configure the Zeta-APS system. The File Menu and Toolbar appear below:

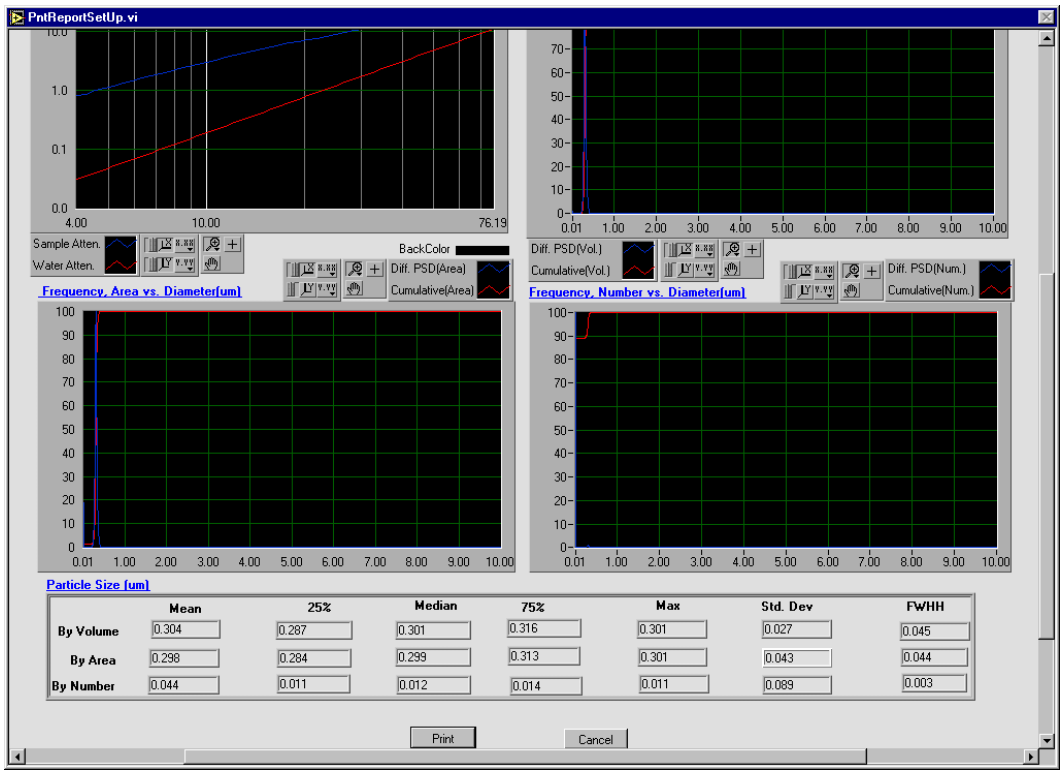
4. File/Open
   Select a file to open. Currently, only Single Point files can be opened. Single Point files contain PSD data. Upon opening a file, the following window is displayed:



The mean particle size figures are shown on the right side of the window. The left graph shows the sample acoustic attenuation spectrum (in red), plus the water theoretical spectrum (in green). Use the palette in order to edit the graphs, e.g., scale adjustments, zoom-in or out, and others. Right click on the plot legend in order to edit the curve type, color, width, etc. Click Report in order to view the PSD report as shown below.



scroll down to see the rest of the window:



This window displays several PSD figures such as volume-, area-, and number-average particle size values. The 25, 50 (median), and 75 percentiles are shown as

well. The Max figure represents the mode (peak maximum height) location. The standard deviation is for the whole PSD. FWHH is the PSD full width at half height.

Click Print in order to print the report to your Windows default printer. Click Cancel to return to the prior PSD window.

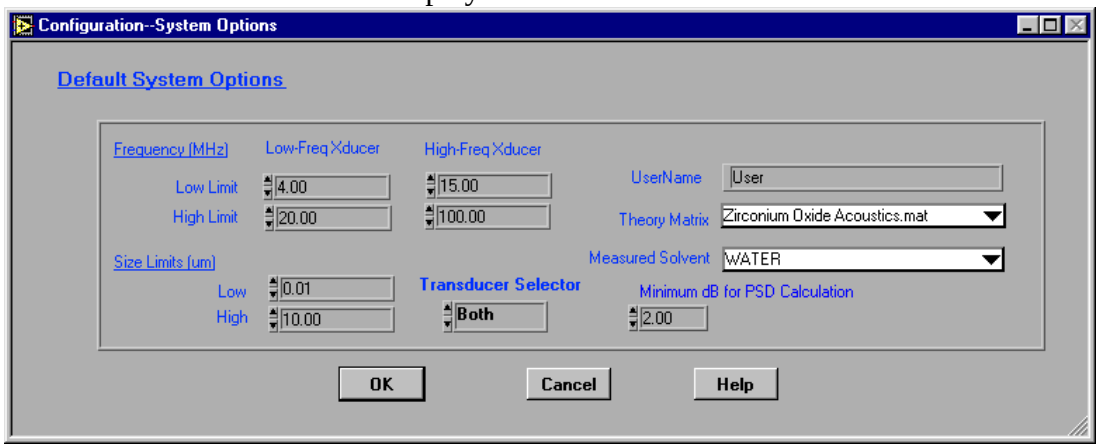
5. File/Page Setup.
   Set your printer preferences.

6. File/Exit
   Exit the Zeta-APS software.

7. Measurement/Single Point.
   This option allows you to make a Single Point PSD measurement. See Zeta-APS Quick Guide to Sample Analysis.
   Time Series, Concentration Series, and Potentiometric Titration will be functional at a later software upgrade.

8. Measurement/Meas T, pH, Cond
   Measures sample temperature, pH, and conductivity.

9. System/Configure System/System Options.
   A window as shown below is displayed:



The entries shown above refer to the default choices and are recommended.

It is recommended that you select a default theory matrix that you most commonly will employ.

The particle size limits (um) are used when creating the transducer calibration curves. In order to actually change the particle size limits used in the PSD computations, you must create a matrix with the desired particle size range, see Matrix section.

Use WATER as the measured solvent for most water-based solvents.

10. System/Configure System/New Hardware Setup/Meas Xducer to Home
    This feature allows the Zeta-APS to measure the distance between the xducers and the reflector. It also measures the sound speed of the sample/solvent. The sound speed is displayed for four seconds. This option is useful when building matrices of non-aqueous solvents. See Matrix building.

11. System/HF Transducer Alignment.

    This option allows you to align the high-frequency transducer. You can also use it to move the reflector to any allowed position. You need to align all newly-installed transducers (when replacing a transducer). You may need to re-align the two transducers approximately every six months.

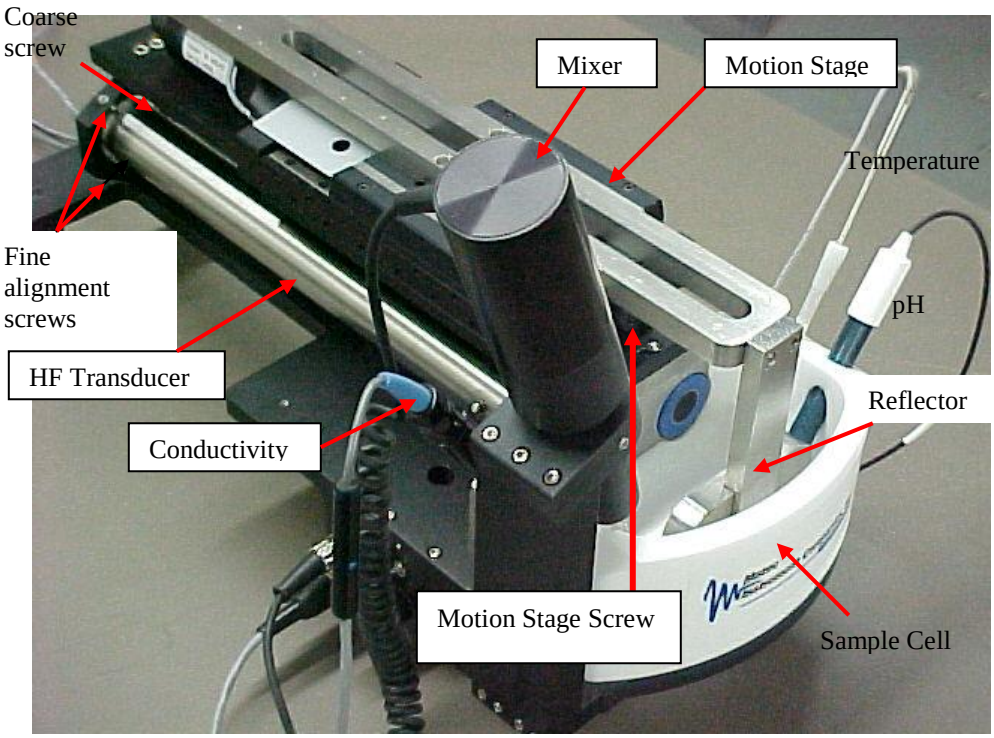
    Remove the sample cell cover and turn the two fine-thread setscrews as shown in the figure below. The displayed attenuation figure between the first (delay rod) and second echoes should be minimized as the screws are turned.

    Note: There is a third coarse screw facing the opposite transducer. This screw should only need to be turned when installing a new transducer. Re-alignment normally only requires the two fine-thread screws to be adjusted.

    The idea is to minimize the attenuation level between the first delay rod and the fluid echoes. Adjust the time delay and window with on both small windows in order to view the first delay rod echo on the left lower window, and the first fluid echo on the lower right window.

    Observe the Attenuation (dB) display. Adjust the setscrews in order to minimize it. Start at 50 MHz, then switch to 90 MHz frequency in order to fine-tune the alignment level. Adjust the power and gain settings so you see about full -scale waveform amplitude for the delay rod waveform.

    As a final check, set the frequency to 35 MHz. You should obtain about 9 to 11 dB/cm attenuation.

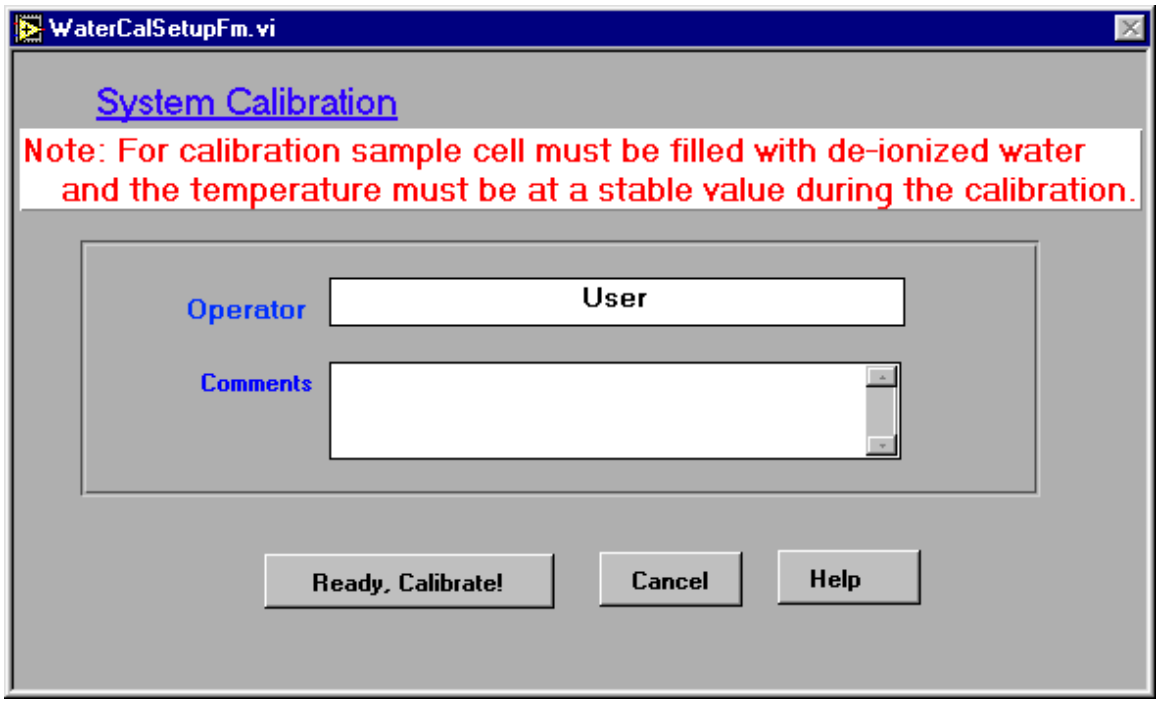


12. System/ LF Transducer Alignment
    This option allows you to align the low-frequency transducer (usually used in the frequency range 2-20 MHz). The alignment procedure is similar as the high-frequency transducer.

    Perform an initial coarse alignment at a frequency of 10 MHz. Complete a fine alignment at 35 MHz. The attenuation level at 35 MHz should be similar to that from the 35 MHz, about 9 to 11 dB/cm.

13. System Calibration
    You should perform System Calibration periodically, preferably at least once a week. System Calibration allows the Zeta-APS software to perform near field, as well as, diffraction corrections.

    Fill the sample cell with DI water (regular cool tap water is also acceptable). Allow the water temperature to become stable. Select System/System Calibration. The following window will be displayed:



Enter your comments. Click Ready, Calibrate!

14. System/Transducer Calibration
    This option is normally used when a new transducer is installed. Matec will usually supply you with a new transducer calibration file should you need to replace your transducer.

    The transducer calibration option is only available if the Zeta-APS software is set to Diagnostics mode in the C:\Acoustic Particle Sizer\APS.ini file.

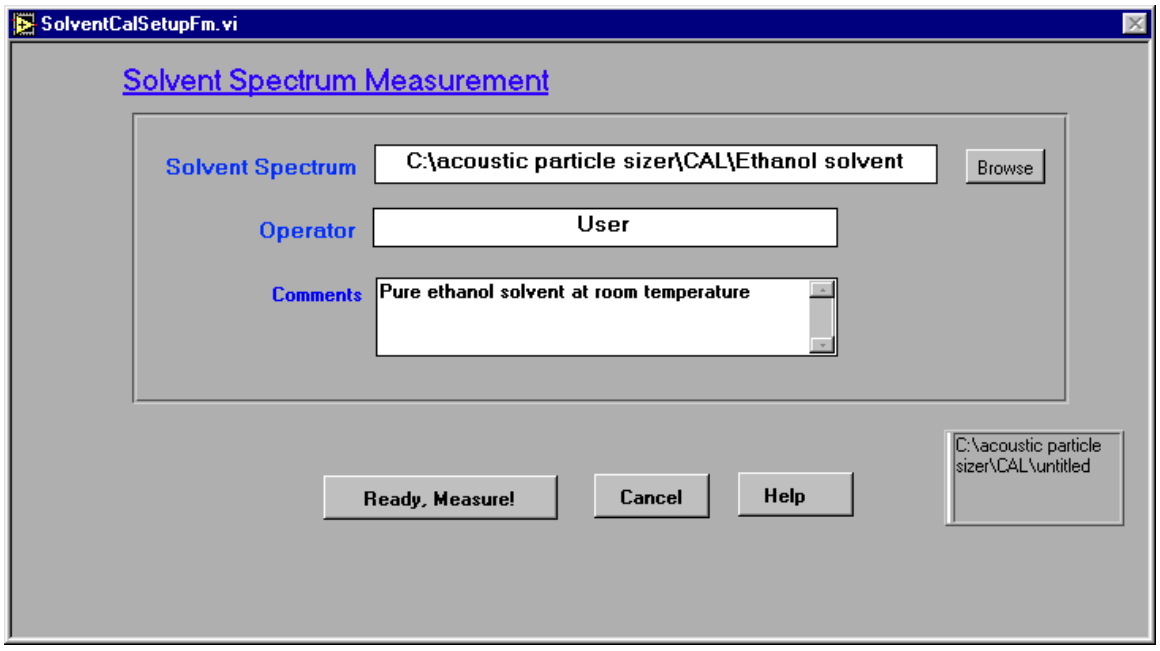
15. Calibration/Solvent Spectrum
    Samples that use solvents other than water require the operator to create a Solvent Spectrum file. Examples of non-aqueous solvents include Isopropyl alcohol, Isopar, Hexane, and other organic solvents. Select an appropriate solvent when running samples.

    Fill the sample cell with the non-aqueous solvent (without particles) as if it were a regular sample. Wait for the temperature to become stable.

    Enter a solvent spectrum file name, or click Browse to select an existing one. There is no need to enter file extension characters (.cal).

    Enter any comments if so desired.

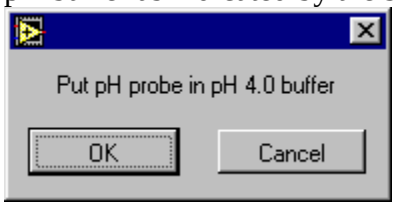
    Click Ready, Measure!



The Zeta-APS proceeds to collect a solvent spectrum for this solvent. This process takes about 10 minutes. The resulting solvent cal file is automatically saved. These solvent Cal files do not have to be periodically re-created. They represent the actual material attenuation behavior of the solvent. They can be used on different Zeta-APS units regardless of which Zeta-APS unit was used to create them.

Select this solvent file when running samples that contain this solvent. See "Zeta-APS Quick Guide to Sample Analysis".

16. Calibrate pH sensor
    Select this option in order to calibrate your pH probe. You need to use 3 pH buffers as follows: 4, 7, and 10. Immerse the pH and temperature probes into each pH buffer as indicated by the software. See figure below.

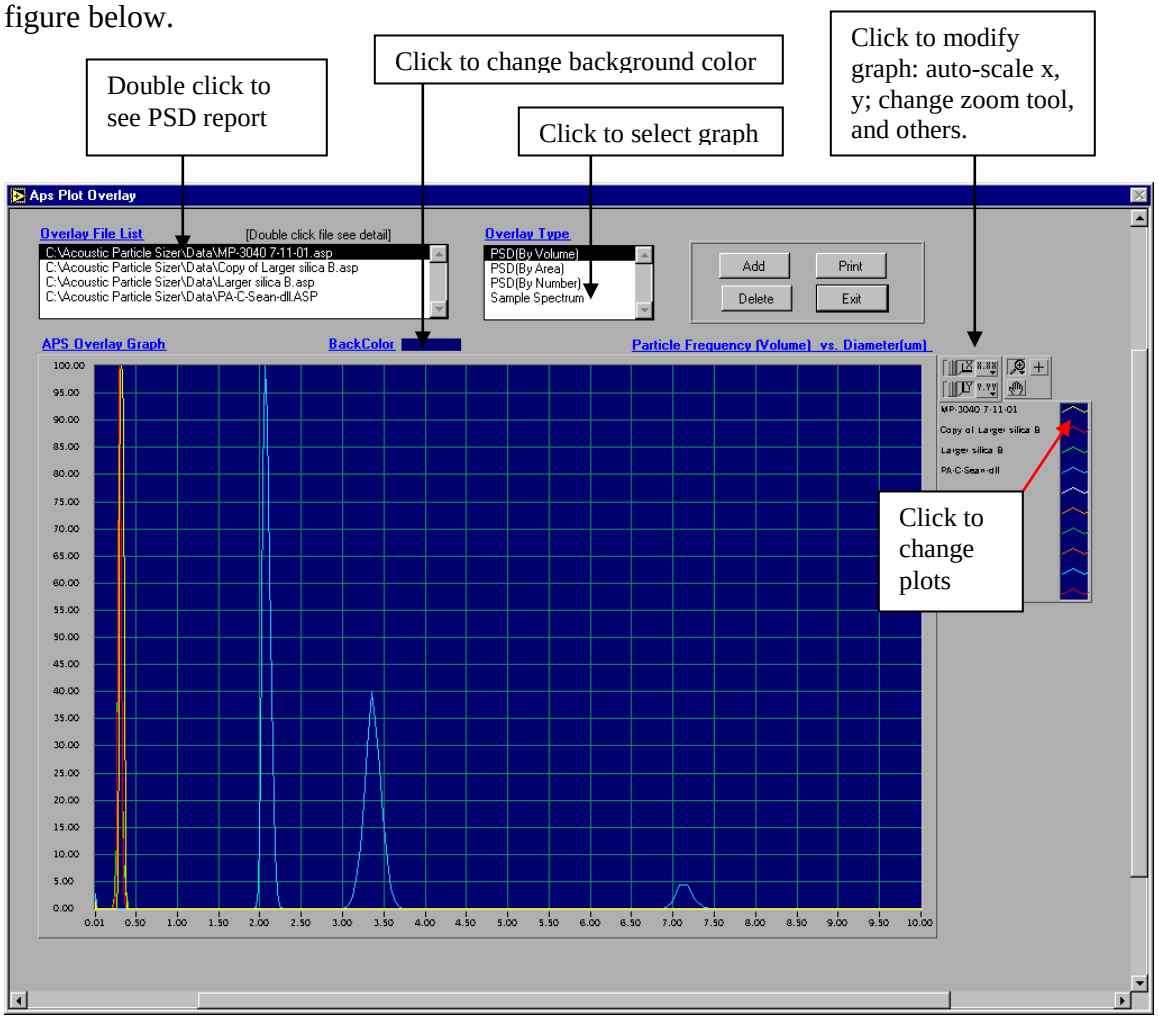


17. Calibration/Conductivity Sensor
    Fill the sample cell with a conductivity calibration standard, typically a 0.01 M KOH solution that produces 1,410-uS/cm conductivity. Follow the screen instructions in order to calibrate the conductivity sensor and circuitry.

18. Calibration/Temperature Sensor
    This option is only needed for units that do not have a sample heater, temperature-

control unit. Ignore this section if your unit has a sample heater. Select this option in order to calibrate the temperature probe and circuitry. Prepare a cold (recommended 15 degrees C) and a warm (recommended 45 degrees C) water samples. You will need to calibrate with both solutions. Follow the screen instructions in order to complete the calibration.

19. Tools/Overlay Plots
    Select this option in order to overlay plots. You can select Weight-, Area-, and Number-weighted PSD plots. You can also view acoustic attenuation spectra. See figure below.



    Click Add in order to add more PSD plots to the overlay graph. You can change the background color by clicking on Back Color. You can change curve features by clicking on each curve on the right side of the window.

20. Tools/Build Matrix
    APS PSD computations require the use of acoustic attenuation matrices. Upon selecting this option, you will be provided the following window:

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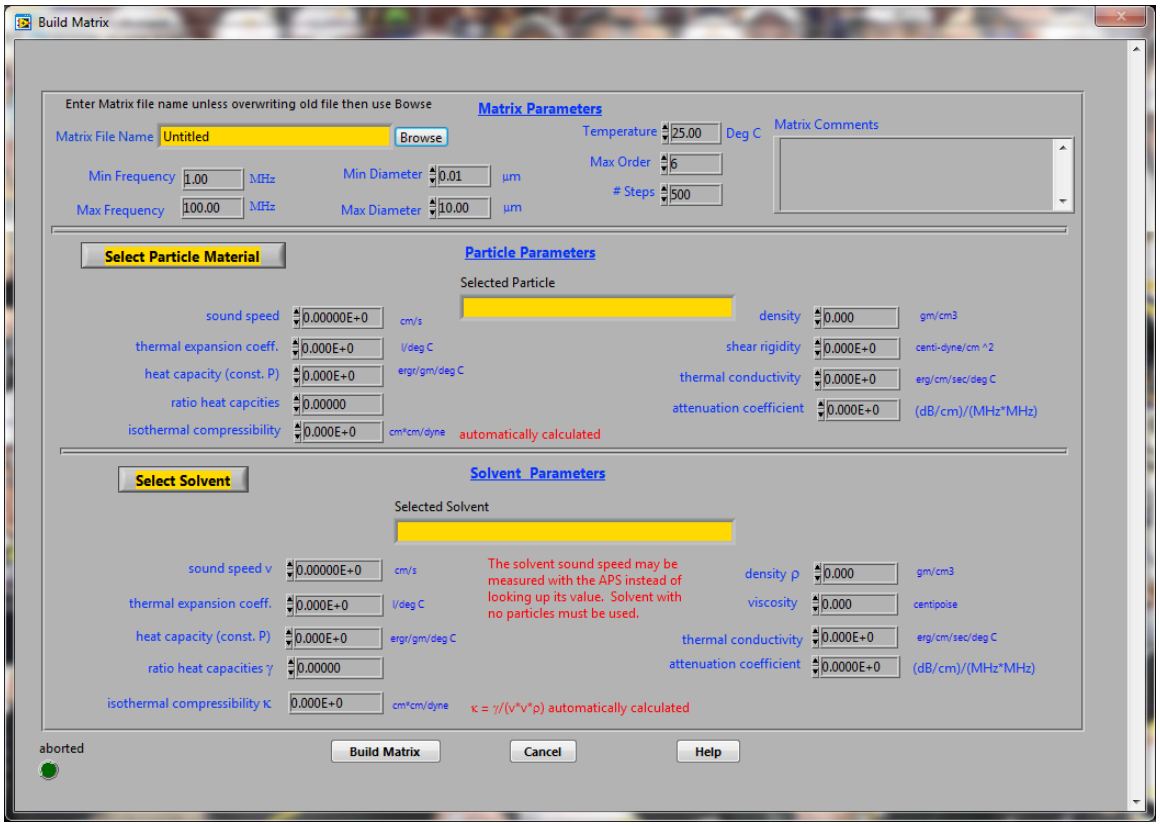

It is recommended that you use the Matrix Parameters shown above.

Enter a matrix file under Matrix File Name.

Select pre-stored Particle Material and Solvent parameters. You can modified any value after selection (before clicking Build Matrix). Enter cgs units for all parameters.

The APS-100 software automatically calculates the Solvent Iso-thermal Compressibility upon clicking Build Matrix. It is recommended that you measure the solvent sound speed using the APS-100 unit prior to building the matrix.

The particle Iso-Thermal Compressibility must either be found in suitable tables (recommended) or calculated as follows:

$\kappa = \gamma/(\rho * v^2)$

where κ is the Iso-Thermal Compressibility, γ is the ratio of heat capacities C_p/C_v, ρ is the density, and v is the sound speed. Use particle parameters for the particles, and solvent (without particles) parameters for the solvent.

The particle density is material density, not the bulk density from a dry powder.

Click Build Matrix in order to have a matrix built. These computations last several minutes. The new matrix is saved automatically. You can use any matrix when analyzing samples by clicking the drop-down arrow next to the Matrix entry (see Particle Size Analysis).

21. Tools/Build Th. Ac. Spectrum
    This option will be available in future software upgrades. It will allow you to build theoretical acoustic spectra that can be compared to experimental data.

22. Tools/Calculate Particle Size Distribution
    You can re-calculate PSD data from previously saved data. You can change parameters such as matrix type, and compare the differences in PSD data by later selecting Overlay Plots.

23. Tools/Show Clipboard
    This shows you the contents of the Windows clipboard memory.

Appendix I

Software Installation

The Zeta-APS software can be supplied in two ways as follows (i) on a CD ROM; (ii) as an upgrade on Matec Applied Sciences ftp web site, see below.

Installation Software Supplied on CD.

Note: Do not connect your PC (USB) to the Zeta-APS unit until instructed to do so below.

The Zeta-APS can be operated using either a laptop or desktop PC running Windows 7.

A DVD drive on your Zeta-APS PC is required to install the software.

1. Several CD/DVD disks are provided with your Zeta-APS unit.
2. Load the PicoScope disk and install the PicoScope software. Click OK/Next on all prompts in order to install this driver. Use all default settings. Do not reboot yet.
3. Load the Zeta-APS Software Installation disk into your Zeta-APS DVD drive.
4. Copy all files and folders to C:\
5. Note: "yy" on the next instruction refers to the Zeta-APS software version.
6. Launch file C:\Compiled Zeta-APS yy\Driver Installer\Volume\setup.exe.
7. Click OK/Next on all prompts.
8. Launch file C:\Compiled Zeta-APS yy\Application Installer\Volume\setup.exe.
9. On the same disk, open folder \Prolific Hub Windows7 Driver\ and launch file PL2303_Prolific_DriverInstaller_v130.exe\. Complete this driver installation.
10. Load the Measurement Computing (MC) disk the disk containing software labeled as Instacal. Install all the software on this disk.
11. Turn off your PC (do not reboot/restart yet)
12. Connect the two USB cables between the Zeta-APS rear panel and the PC. Connect the power cable. Ensure that the power 110/220V is correct.
13. Turn on the Zeta-APS unit. Two power lights should be lit; if only one is lit, check power switch on the Zeta-APS' rear panel.
14. Turn on the PC. The SerialGear Hub should be automatically detected. Before clicking OK, perform the step below.
15. You need to install several drivers including the SerialGear USB Hub driver.
16. Insert the SerialGear CD in order to install its driver.
17. Launch the Instacal software using Start/Programs/Measurement Computing/Instacal. The two MC boards should be identified. Click OK.
18. The Zeta-APS software can now be launched using the Zeta-APS desktop shortcut. If successful, start using your Zeta-APS instrument. Otherwise continue below.

19. If the initialization step fails, it is likely that one or more COM ports are incorrectly assigned. Click on Control Panel/System/Hardware/Device Manager/ and observe the COM port numbers.
20. Notepad open file C:\Z-APS\Common ESA-APS.ini\. You will likely need to change the COM number assignments for the Novatech board, and the temperature controller. See below.
21. Install the software for Compiled Novatech Test Program/Installer/Volume/Setup.exe. Run this program several times (click the white arrow to re-run after fail) until it detects the COM port number for the Novatech board. Enter this number in file C:\Z-APS\Common ESA-APS.ini\.
22. Repeat this procedure to find the Heater COM port by installing software Setup Temperature Control 5-2 Compiled/Installer/Volume/Setup.exe/. Re-run (white arrow) this program until you find the Heater COM port successfully.
23. Enter this number in file C:\Z-APS\Common ESA-APS.ini\. save and exit this file.
24. The Motion Stage COM port number can be found by trial and error upon launching the Zeta-APS software (Initialization Error does not appear).
25. Test the instrument using Ludox-TM 2.5% vol standard. Re-calibrate the Zeta measurement using System Cal.

Software Upgrades Downloaded from ftp.matecma.com.

Follow the next steps:

1. In Windows Explorer (not Internet Explorer), enter the following in the Address line ftp://ftp.matecma.com/MAS/ (do not use www) and hit Enter; download the latest Zeta-APS software upgrade version (zip file). Extract the compressed folder to C:\. If a password is required, enter the following: masahiro
2. A new installation folder will be created with a name related to the software upgrade number.
3. Uninstall your old Zeta-APS software from Control Panel-Add/Remove Programs.
4. Launch file setup.exe located in the following subfolder of the installation folder: \Installer\Volume\setup.exe.
5. Click OK/Next on all prompts.
6. A new Zeta-APS desktop shortcut has automatically been created.
26. Launch the software by double-clicking on the Zeta-APS desktop shortcut and verify the version number by clicking Help/About.

Appendix II

Acoustic-Transducer Alignment

NOTE: READ THIS PAGE IN ITS ENTIRETY BEFORE ALIGNING THE TRANSDUCERS.

This appendix describes the procedures required to perform alignment of the Zeta-APS' high (HF) and low (LF) frequency acoustic transducers relative to the reflector.

Both Zeta-APS acoustic transducers must be properly aligned in order to maximize PSD data accuracy. This procedure should be performed after the Zeta-APS sample cell is shipped, the motion stage and/or a transducer is replaced.

Unless the Zeta-APS unit has been shipped or subjected to extreme movement, you will only need to perform "fine" alignment as described below.

Note: Transducer performance may vary. You may need to set the Gain somewhat lower or higher than suggested in order to observe a "Peak Value Full Waveform" (first echo) at near full scale.

Also, refer to the "Alignment Hints and Tips Section" at the end of this section for more alignment help.

There are three alignment screws on each alignment ring (one alignment ring per transducer); two of the alignment screws are smaller with fine threads; the third alignment screw is larger with coarser threads. The HF larger screw is somewhat hidden from view.

The idea for this alignment is to first loosen (one to two turns) the Coarse screw so the spring inside the coarse screw still pushes against the fine screws while allowing them to rotate in either direction so that the attenuation level in water is minimized.

Fill the sample cell with DI water, about 1 cm from the top of the cell. Rotate first one of the fine screws while watching the attenuation readout on the alignment window of the software (details below). Try to minimize the attenuation level. Switch to the other fine screw and repeat. Go back to the first fine alignment screw and try again to minimize the attenuation level. Keep going back and forth between the two fine screws until the attenuation is about 9 dB for the HF transducer at 33 MHz, and about 7-8 db for the LF xducer at 15 MHz. Once completed, *gently* tighten the large coarse screw to prevent unintended transducer motion.

1. **Low-Frequency (LF) Transducer Alignment Procedure:**

A. Verify that the sample-cell valve is closed. Fill the sample cell with DI water.
B. Turn on the Zeta-APS unit and motion-stage power supply (if present).
C. Launch the Zeta-APS software.
D. Click *System/LF-Transducer Alignment.* Wait for the reflector to stop moving.
E. Loosen the large coarse screw about 1-2 turns to allow the fine screws to rotate in either direction.
F. For coarse alignment, set the alignment parameters as shown below:
    a. Frequency: 10 MHz
    b. % transducer drive power: 50
    c. Digital Gain: Adjust as needed so that "First Echo" and "Second Echo" waveforms (the two smaller graphs in alignment window above) are within scale (not clipped or "flat" top and bottom).
    d. 2nd echo Time Delay (µs): 6.00
    e. 2nd echo Window (µs): 4.00

G. Turn the two fine-alignment screws (see stage photo in this manual) in order to minimize the "Attenuation (dB)" value as shown on figure 1.

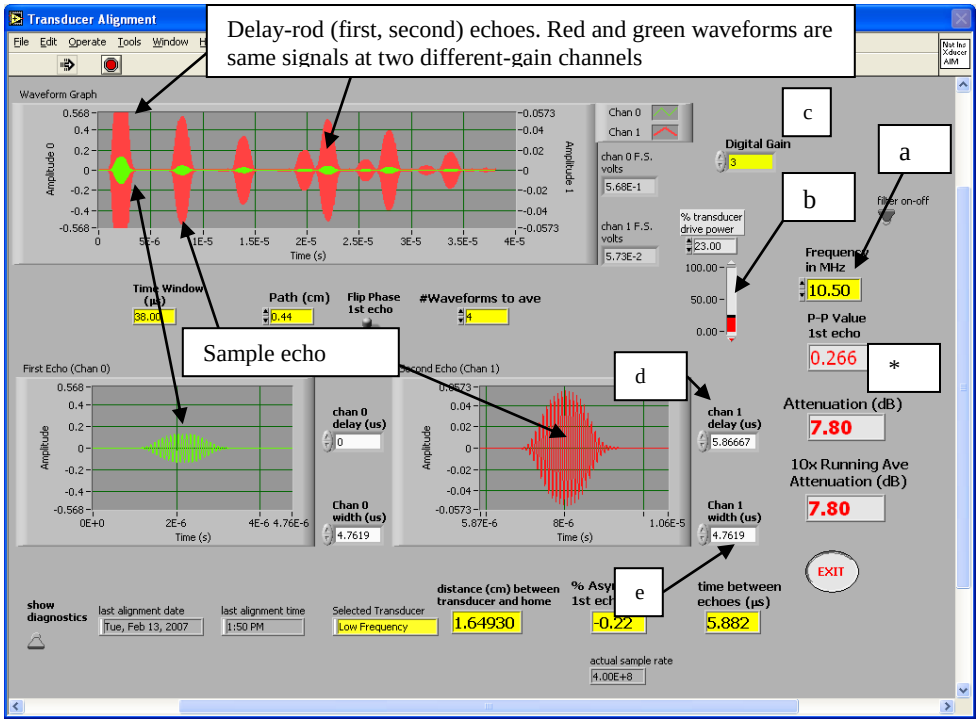

Fig. 1. LF transducer coarse alignment settings at 10 MHz.

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H. To perform LF fine alignment, set the alignment parameters as shown below:
   a. Frequency: 35 MHz (set to 32 MHz if no signal at 35 MHz; set to 18 MHz if 35 and 32 MHz produce no signal).
   b. % transducer drive power: 100
   c. Digital Gain: Adjust as needed so that "First Echo" and "Second Echo" waveforms (the two smaller graphs in alignment window above) are within scale (not clipped or "flat" top and bottom).
   d. 2nd echo Time Delay (µs): 6.00
   e. 2nd echo Window (µs): 4.00

I. Turn the two fine-alignment screws (see stage photo in the Zeta-APS manual) in order to minimize the Attenuation (dB) value as shown on figure 2.

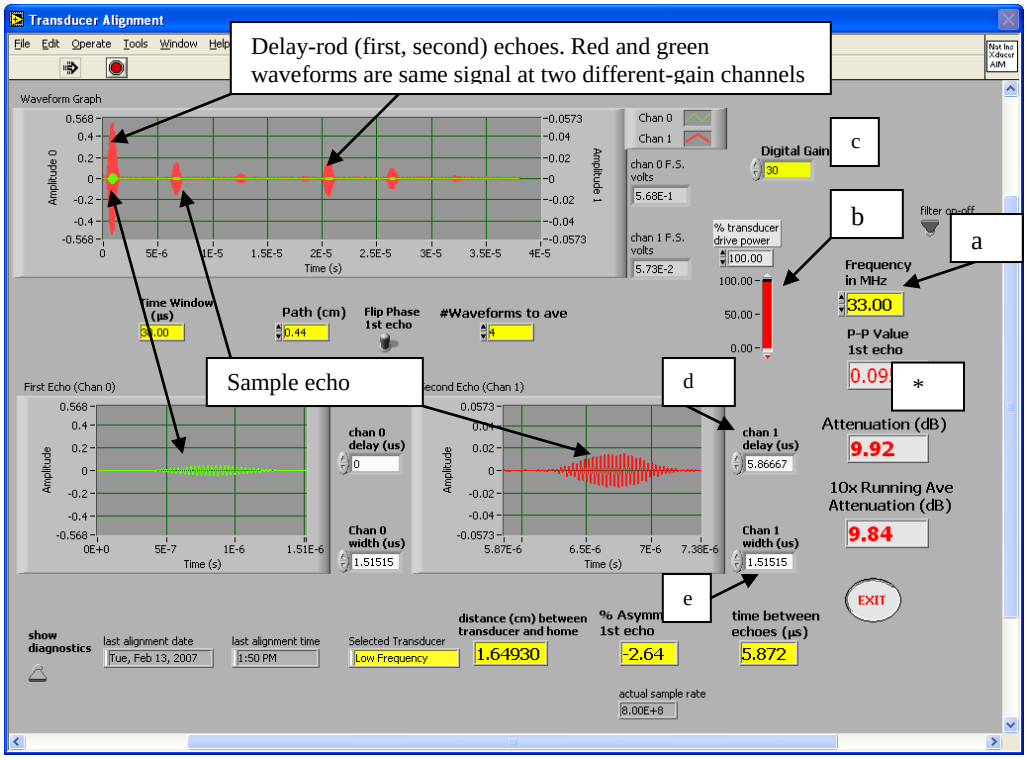

Fig. 2. Fine alignment settings for LF transducer.

J. Click Exit. Answer "Yes" to the ensuing alignment question.

2. **High-Frequency (HF) Transducer Alignment:**

A. Verify that the sample-cell valve is closed. Fill the sample cell with DI water.
B. Turn on the Zeta-APS unit and motion-stage power supply (if present).
C. Launch the Zeta-APS software.
D. Click *System/HF-Transducer Alignment.* Wait for the reflector to stop moving.
E. Loosen the large coarse screw about 1-2 turns to allow the fine screws to rotate in either direction.
F. For coarse alignment, set the alignment parameters as shown below:
    a. Frequency: 50 MHz
    b. % transducer drive power: 50
    a. Digital Gain: Adjust as needed so that "First Echo" and "Second Echo" waveforms (the two smaller graphs in alignment window above) are within scale (not clipped or "flat" top and bottom).
    c. 2nd echo Time Delay (µs): 6.00
    d. 2nd echo Window (µs): 1.00

G. Turn the two fine-alignment screws (see stage photo in the Zeta-APS manual) in order to minimize the "Attenuation (dB)" value as shown on figure 3.

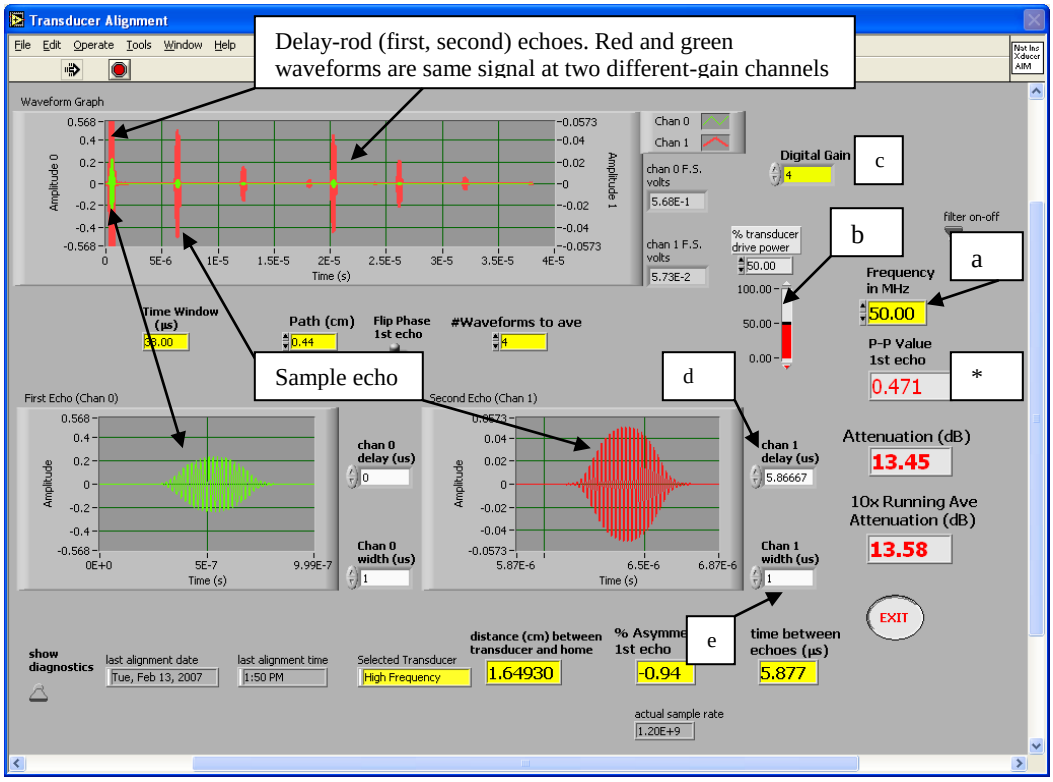

Fig. 3. Coarse alignment settings for HF transducer.

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H. To perform HF fine alignment, set the alignment parameters as shown below:
   a. Frequency: 75 MHz
   b. % transducer drive power: 100
   b. Digital Gain: Adjust as needed so that "First Echo" and "Second Echo" waveforms (the two smaller graphs in alignment window above) are within scale (not clipped or "flat" top and bottom).
   c. 2nd echo Time Delay (µs): 6.00
   d. 2nd echo Window (µs): 1.00

I. Turn the two fine-alignment screws (see stage photo in the Zeta-APS manual) in order to minimize the Attenuation (dB) value as shown on figure 4.

J. Click Exit. Answer "Yes" to the ensuing alignment question.

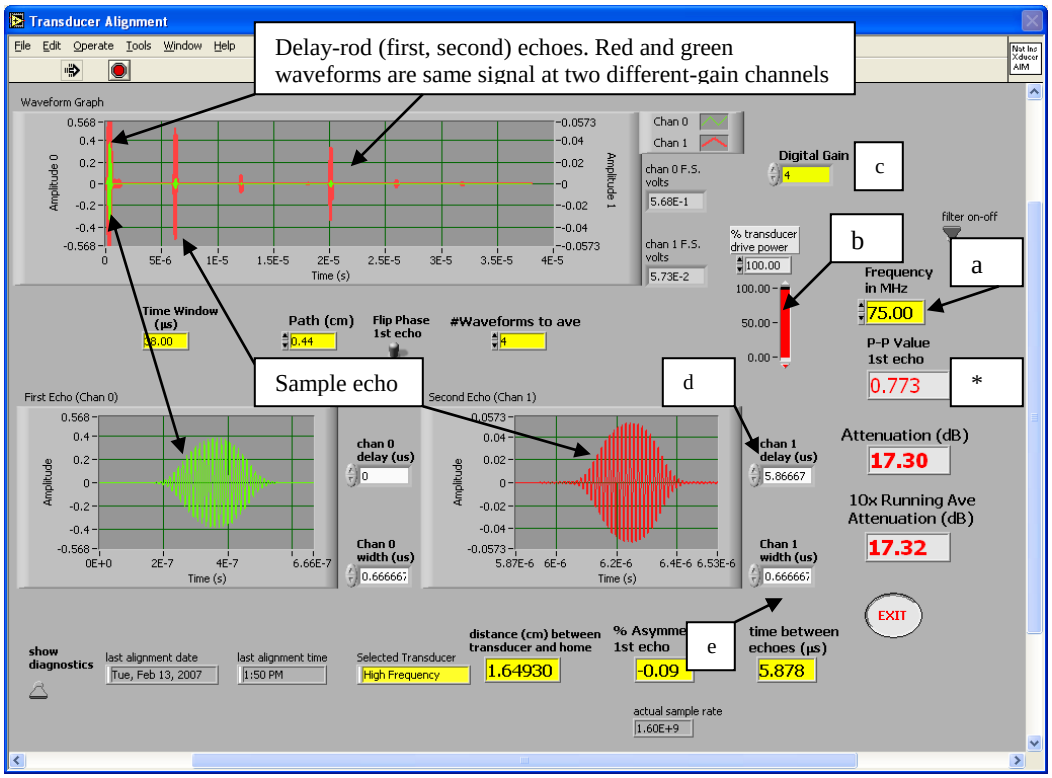

Fig. 4. Fine alignment settings for HF transducer.

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1. If you cannot find a suitable alignment setting, you can loosen all three alignment screws (AL), side fine, top fine, coarse screw.

2. Set the Frequency to 50 MHz for the HF xducer or 11 MHz for the LF xducer.

3. Set the % power to about 60%.

4. Set the Gain so that the "Peak Value Full Waveform" is near full scale. For better signal-to-noise ratio, it is better if Gain is set to 3 or higher.

5. Grab by hand the end of the xducer to be aligned (xducer) and gently move it around until you see sizable sample echoes on the alignment screen.

6. If possible, hold the xducer in this position while you turn two (the coarse one and the side fine screw) of the AL's so the xducer stays in about this position. You can leave the top screw loose for now.

7. Now, you can turn the side fine, and the coarse screw to try to minimize the attenuation reading.

8. Frequently, the optimal position will be reached while the top screw is still loose. Now tighten the top fine screw in order to optimize the alignment (minimum attenuation).

9. Sometimes when you tighten this top screw, the attenuation goes up instead of down! (confusing since you do not want to leave this screw loose!!). No problem. If so, you can tighten the top screw until it just touches the xducer. Then, you can tighten the coarse screw; say one turn (you will notice that alignment stays the same). How can this be? There is an internal spring inside the coarse screw. This spring allows you to move the xducer toward the coarse screw without having to loosen the coarse screw. If you fully tighten the coarse screw (fully compress the spring inside the coarse screw), you will not have alignment room toward the coarse screw (the xducer would not be able to move toward the coarse screw if its spring is fully compressed), so you want to leave a little internal-spring play on the coarse screw.

10. You should fine tune the alignment at 75 MHz (HF xducer) or 33 MHz (LF xducer), % power= 100, and Gain set to achieve full-scale peak value (without exceeding the scale –“flat” top- see above). To fine align, you should only need to turn the two fine screws (assuming you left some spring play on the coarse screw).

11. Double-check at 33 MHz for both xducers, % power= 40 (HF) or 100 (LF), Gain set accordingly. You should obtain about 9 dB.

12. To be safe once you have aligned, you can also reset the "Distance to Home" by filling the sample cell with DI water, and clicking System/New Hardware Setup/Measure Xducer to Home/.

13. Perform a Water System Cal, and Water Single Point. Verify that the measured and theoretical curves are close to each other.

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Appendix III

APS Transducer-Replacement Procedure

This appendix describes the procedure to replace one or two Zeta-APS transducers. Note 1: Avoid scratching the transducer polished quartz face. Note 2: Avoid "crashing" the replacement transducer into the reflector as you push the replacement transducer though the metal plate opening. You can move the reflector away from the metal plate by increasing the "path (cm)" value to 2 cm in the transducer-alignment option in the software. See Appendix II.

In summary, the replacement procedure consists of removing the faulty transducer, installing the new transducer and either installing or creating the transducer calibration file. Transducer installation is facilitated by using a screw driver to push the new transducer into its sample-cell opening. Use the other transducer alignment ring to provide leverage for pushing the replacement transducer. Apply lubricant to the opening O-ring before mounting the replacement O-ring.

1. Empty the sample cell.
2. In order to prevent crashing the replacement transducer into the reflector, move the reflector 2 cm away from the transducer by using the Zeta-APS Windows software. Select "System/LF transducer Alignment". Enter 2 cm in the "path (cm)" option. See Appendix II for more on the alignment option.
3. It is advisable to keep a finger or other cushioning material between the reflector and transducer in case that the replacement transducer slips into the reflector.
4. Disconnect the black cable from the old transducer micro-dot connector (see sample cell figure in the Zeta-APS manual).
5. Remove the old transducer. Removal may be helped by rotating the transducer to allow it to dislodge from the opening O-ring.
6. Place lubricant on the O-ring.
7. Carefully slide the new transducer through its alignment ring.
8. HF transducer installation: Use a screw driver or similar device to help push the new HF transducer into place. The screwdriver can be leveraged against the neighboring LF alignment ring for easier installation. See figure 1.
9. LF transducer installation: Use a screw driver or similar device to help push the new LF transducer into place. The screwdriver can be leveraged against the neighboring HF alignment ring for easier installation. See figure 2.
10. Connect the transducer black cable to the transducer.
11. Fill the sample cell with DI water.
12. Exit the Zeta-APS software.
13. In order to ensure that both transducers are equidistant from the reflector, carefully push both transducers into the sample cell so that each transducer protrudes about 0.3-0.4 cm into the sample cell.

14. Turn off the power supply (black box) to the stage (if present). Turn off the Zeta-APS main electronics unit (large box).
15. Turn clockwise by hand the "Stage Motion Screw" (see sample cell photo in this manual) in order to move the reflector closer to the transducers.
16. Continue to turn the motion screw as the transducers are pushed by the reflector until the reflector contacts the metal plate.
17. Wait 15 minutes to allow the O-rings to relax.
18. Turn the motion screw counterclockwise by hand until the reflector is about 1-2 cm from the transducers.
19. If supplied with the replacement transducer, copy the new transducer calibration file to C:\Matec Cal Files\. Otherwise, skip to the transducer-calibration section below.

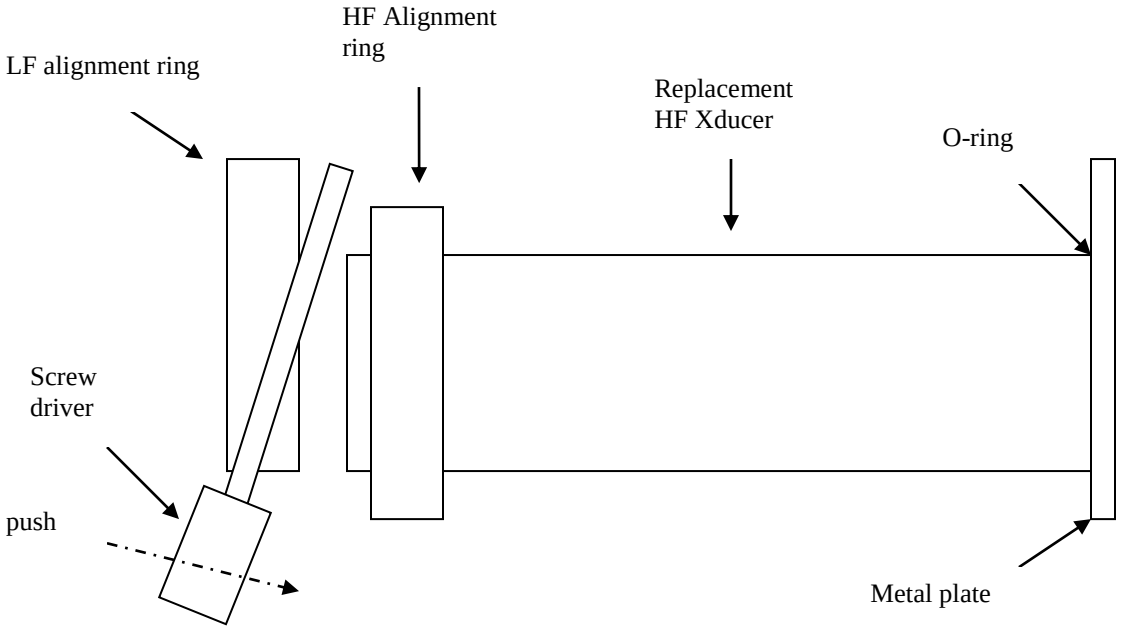


Fig.1. HF transducer installation.

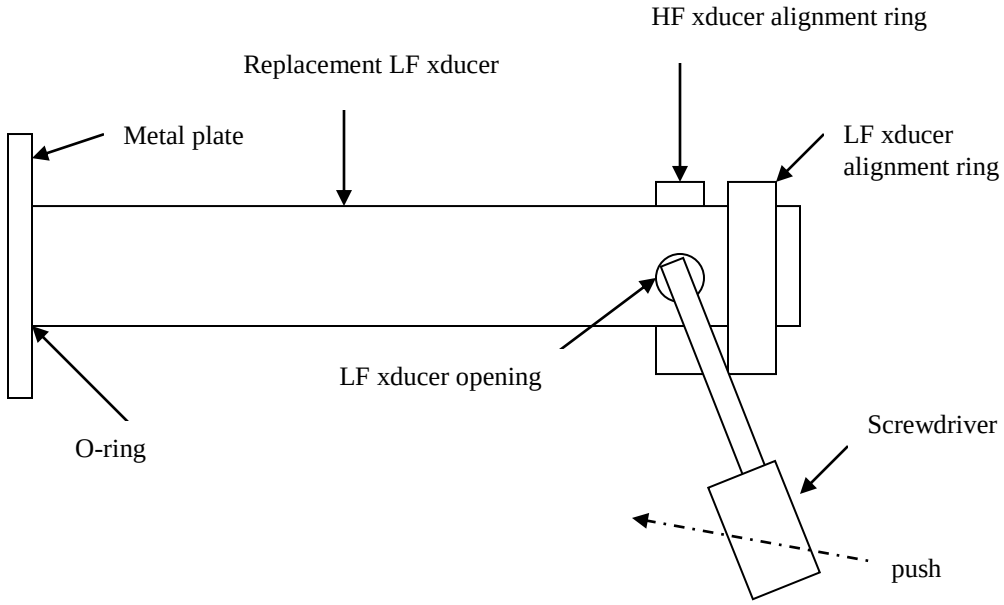


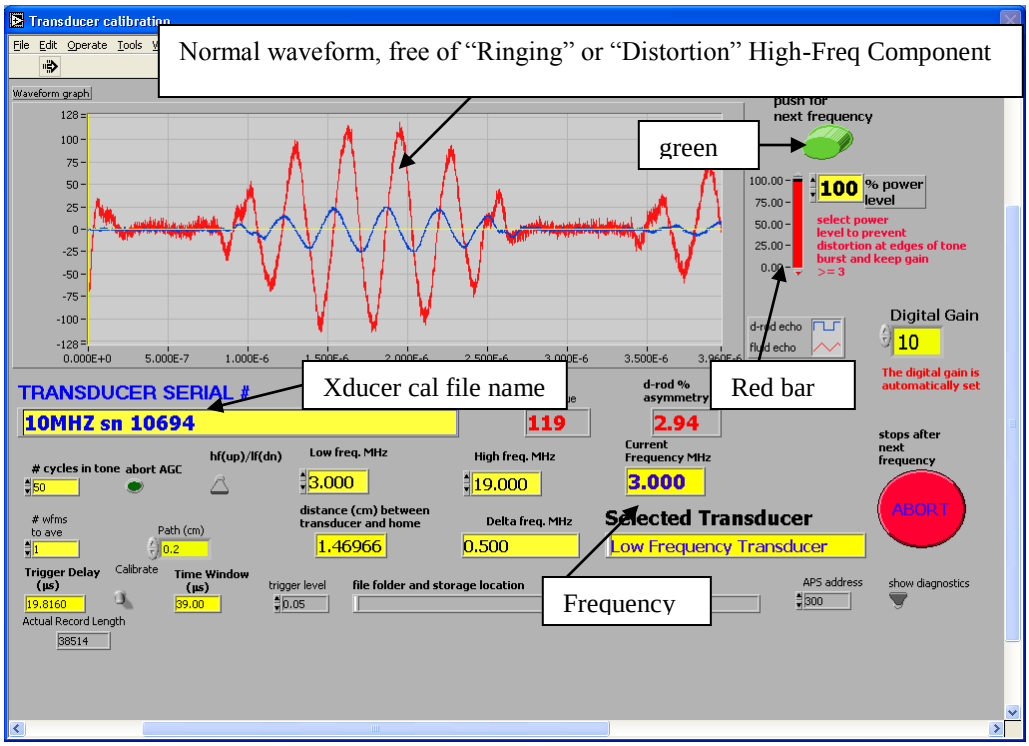
Fig. 2. LF xducer installation.

20. Open file C:\Acoustic Particle Sizer\aps.ini
21. Enter the transducer calibration file name in the following lines (do not include the "xdr" file name extension). NOTE: Use the name of the supplied calibration file, not necessarily those shown below.
    HiFreq xducer Serial #=55MHZ sn 01234
    LoFreq xducer Serial #=10MHZ sn 07590
22. Save and Close the ini file.
23. Launch the Zeta-APS software.
24. Perform an alignment of both transducers as shown in Appendix II.
25. Update the distance from Home by clicking System/Configure System/New Hardware Setup/Meas Xducer to Home/
26. Perform a System Calibration.
27. Proceed to analyze samples as usual. Skip the rest of this appendix.

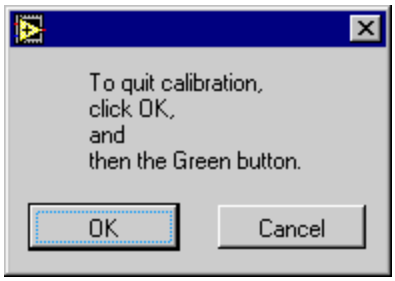
Transducer Calibration

Follow the steps below if you did not receive a transducer cal file with the replacement transducer. You can repeat the transducer cal if you make a mistake. If so, abort the calibration and repeat.

28. A new calibration file must be built if a replacement transducer is supplied without a calibration file.
29. Click System/Configure System/Set Calculation Limits/. Click on the "Diagnostics Switch" in order to enable the diagnostic mode. After you complete the transducer cal, set the "Diagnostics Switch" back to its original position in order to disable the diagnostics mode.
30. Click System/Transducer Calibration
31. The LF-calibration option is shown first as presented below:



32. If you are calibrating a LF transducer (new LF transducer just installed), skip this step. If you are calibrating a HF transducer only (replaced only the HF transducer), then exit the LF calibration by clicking "ABORT", then clicking the green button. The following message appears:



Click OK, then click the green button again. The HF calibration window is then displayed. Proceed with the HF calibration as shown below the LF steps.

33. **LF calibration proceeds as follows.**
34. Enter the LF "Xducer Cal File Name". NOTE: If you modify the displayed xducer cal file name, you must also enter the new name in file C:\Z-APS\aps.ini\ after you complete the calibration procedure. Usually, the file name consists of the frequency plus the serial number. An example is shown on the calibration window above. You can find the transducer serial number on the bottom line of the transducer engraved label near its micro-dot connector.
35. As the "% power level" is changed, the Zeta-APS software automatically adjusts the "digital gain" value. Set the "% power level" in order to obtain a "digital gain" value of 3 or higher. For better signal-to-noise data, try keep the "digital gain" value as low as possible, but still at 3 or higher (see table below for typical power and gain values); wait at least 2 seconds, then click the green button. The frequency is increased as the green button is clicked. The software stores the values for "% power level", and "digital gain" to the transducer cal file. Repeat until calibration is complete.
    After calibration, the transducers should be aligned, the Home distance updated, and a System Cal performed (see the end of this appendix for details).

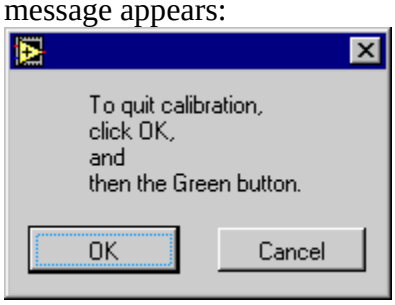
36. Acceptable values for "% power level" and Gain are shown on the table below:

LF Frequency, MHz	% Power	Gain	HF Frequency, MHz	% Power	Gain
3	65	7	16	35	18
3.5	70	6	17	38	16
4	74	5	18	40	15
4.5	80	3	19	40	15
5	80	3	20	42	13
5.5	74	3	21	46	12
6	67	3	22	50	11
6.5	59	3	23	55	9
7	51	3	24	60	8
7.5	46	3	25	65	7
8	41	3	26	70	6
8.5	35	3	27	75	5
9	31	3	28	80	4
9.5	26	3	29	80	3
10	23	3	30	80	3
10.5	20	3	31	80	3
11	16	3	32	75	3
11.5	16	3	33	69	3
12	16	3	34	66	3
12.5	21	3	35	62	3
13	32	3	36	60	3
13.5	45	3	37	57	3
14	61	3	38	54	3
14.5	88	3	39	52	3
15	100	4	40	50	3
15.5	100	6	41	48	3
16	100	8	42	46	3
16.5	100	10	43	44	3
17	100	12	44	43	3
17.5	100	14	45	41	3
18	100	16	46	39	3
18.5	100	18	47	38	3
			48	36	3
			49	35	3
			50	34	3
			51	33	3
			52	32	3
			53	31	3
			54	31	3
			55	30	3
			56	29	3
			57	29	3
			58	29	3
			59	29	3
			60	29	3

61	29	3
62	29	3
63	29	3
64	29	3
65	29	3
66	29	3
67	29	3
68	33	3
69	34	3
70	36	3
71	38	3
72	40	3
73	42	3
74	44	3
75	46	3
76	48	3
77	51	3
78	54	3
79	58	3
80	62	3
81	67	3
82	72	3
83	78	3
84	84	3
85	89	3
86	94	3
87	100	3
88	100	4
89	100	4
90	100	5
91	100	5
92	100	6
93	100	7
94	100	7
95	100	8
96	100	9
97	100	10
98	100	10
99	100	12
99.999	100	12

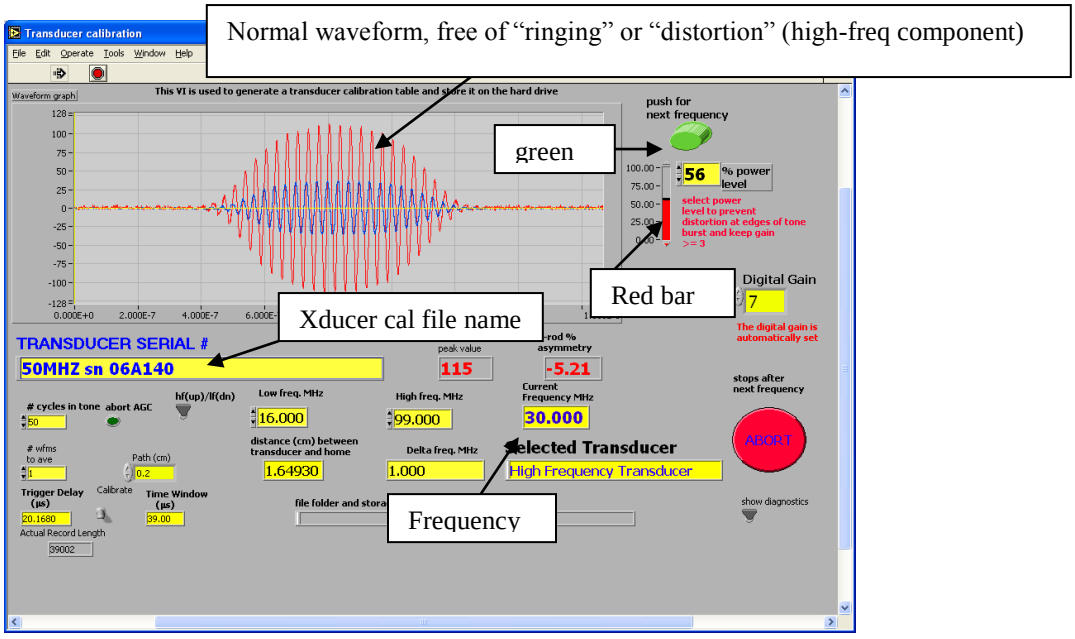
37. At each frequency, set the % power as shown on the table above. Once the LF top frequency has been reached, the software asks if you want to save the cal file. Click OK. The HF cal window is then shown as presented below.
38. If you need to calibrate the HF transducer (you replaced the HF transducer), skip this step. If you do not need to calibrate a HF transducer, then exit the HF calibration by clicking “ABORT”, then clicking the green button. The following

message appears:



39. Click OK, then the green button to exit the HF calibration. Skip to the section below titled "Before Running Samples".

40. **<u>HF transducer calibration procedure:</u>**



41. Enter the HF "Xducer Cal File Name". NOTE: If you modify the displayed xducer cal file name, you must also enter the new name in file C:\Z-APS\aps.ini\ after you complete the calibration procedure. Usually, the file name consists of the frequency plus the serial number. An example is shown on the calibration window above. You can find the engraved transducer serial number on the bottom line on the transducer near its micro-dot connector.
42. Set the "% power level" (see table above), wait at least 2 seconds, then click the green button. The frequency is increased as the green button is clicked. The software stores the values for "% power level", and "Gain" to the transducer cal file. Repeat until calibration is complete. The Gain value should be kept at 3 or higher.
43. At each frequency, set the % power as shown on the table above. Once the HF top frequency has been reached, the software asks if you want to save the cal file. Click OK.

BEFORE RUNNING SAMPLES

44. Exit the Zeta-APS software.
45. Open file C:\Acoustic Particle Sizer\aps.ini
46. Set the following line as shown:
    ShowDiag&Setup=FALSE
47. Enter the new transducer calibration file name in the following lines (do not include the "xdr" file name extension). NOTE: Use the name you used for Transducer Serial # (Xducer Cal File Name) during calibration, not necessarily those shown below.
    HiFreq transducer Serial #=55MHZ sn 01234
    LoFreq transducer Serial #=10MHZ sn 07590
48. Save and close the aps.ini file.
49. Launch the Zeta-APS software.
50. Perform an alignment of both transducers as shown in Appendix II.
51. Update the distance from Home by clicking System/Configure System/New Hardware Setup/Meas Xducer to Home/
52. Perform a System Calibration.
53. Proceed to analyze samples as usual.

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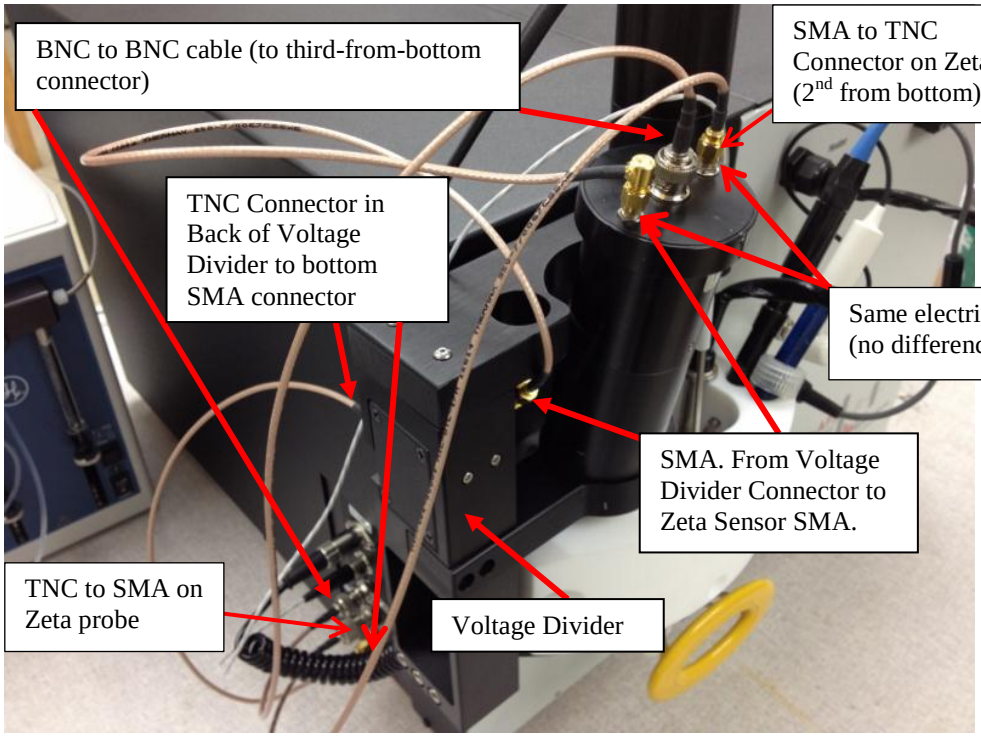
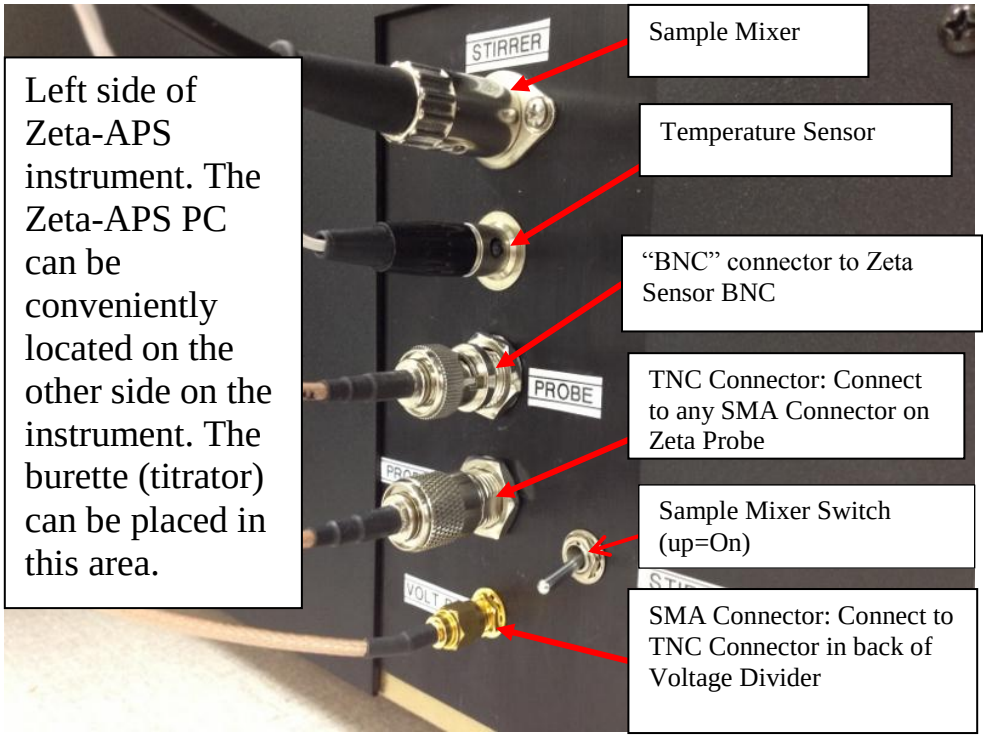
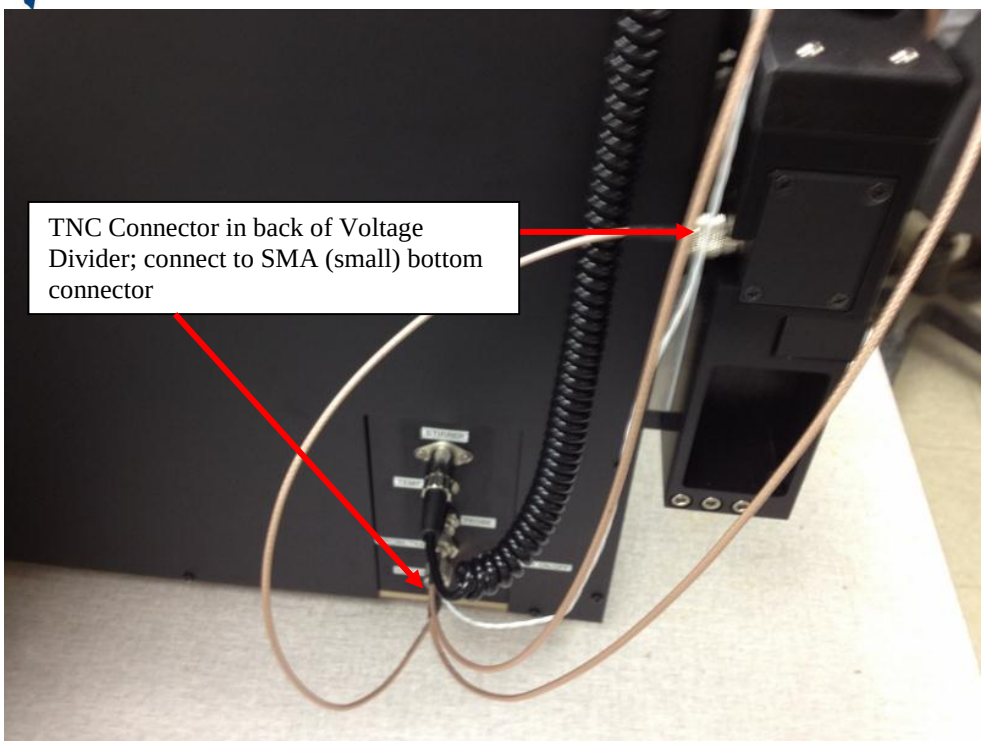
Appendix IV

Zeta-APS Zeta Connections






TNC Connector in back of Voltage Divider; connect to SMA (small) bottom connector

Appendix V

APS Motion-Stage Replacement Procedure

Note: There is no need to dismount the two transducers to complete this procedure.

1. Refer to Figures V.1, and V.2 below. Note: Your hardware configuration may be slightly different from the photos below.
2. Remove the Top Bar and Reflector (do not remove the reflectors from the top bar) by loosening the two Top Screws on the Top Bar. Be careful not to hit the transducers with the reflector.
3. Remove the Top Black Plate (TBP) by loosening the 2 Top Front Screws and 4 Top Rear Screws (connected to the black alignment rings).
4. Remove the TBP from the sample cell. The Motion Stage is still mounted on the TBP. The Motion Stage is held to the TBP by four metric screws located underneath the stage.
5. Loosen these four screws and remove the old Motion Stage.
6. Mount the new stage onto the TBP.
7. Install the four *bottom* screws loosely. Do not mount the TBP onto the Sample Cell yet.
8. Install the Top Bar and Reflector back onto the Motion Stage.
9. The next steps allow you to align the motion stage relative to the transducers. Use Fig. V.2. below as reference. Note: FRONT refers to the area near the reflectors; REAR refers to the area far from the reflectors; BOTTOM refers to the bottom of the TBP.
10. Turn the Motion Stage Screw (black round, approximately 1" diameter) clockwise until the reflectors touch the TBP. This will allow the stage and reflectors to be parallel to and Transducers.
11. Tighten the two bottom rear screws (you must not tighten the front bottom screws yet because the reflectors would be pressed hard against the TBP). Do not tighten the two front bottom screws yet.
12. Turn the black Motion Stage Screw (knob) counterclockwise so that the reflectors move away from the TBP and do not touch it.
13. Tighten the two bottom front screws so that the motion stage is firmly mounted on the TBP.
14. Place the TBP and motion stage back onto the sample cell.
15. Tighten the Front Top Screws and Rear Top Screws.
16. Re-align both transducers as described above.

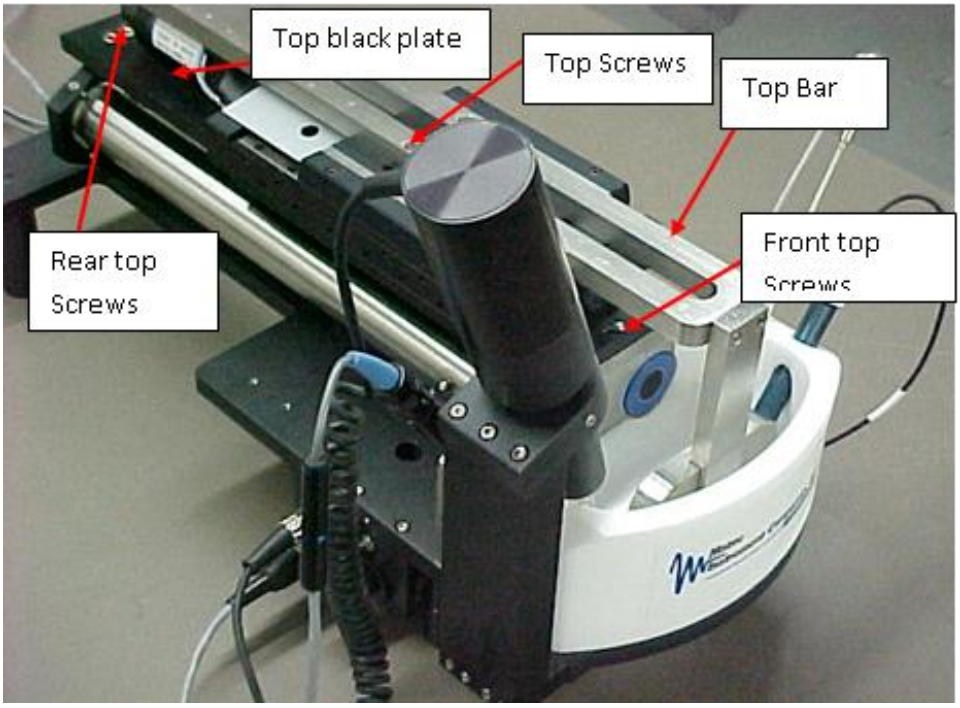

Top black plate
Top Screws
Top Bar
Rear top
Screws
Front top

Fig. V.1.

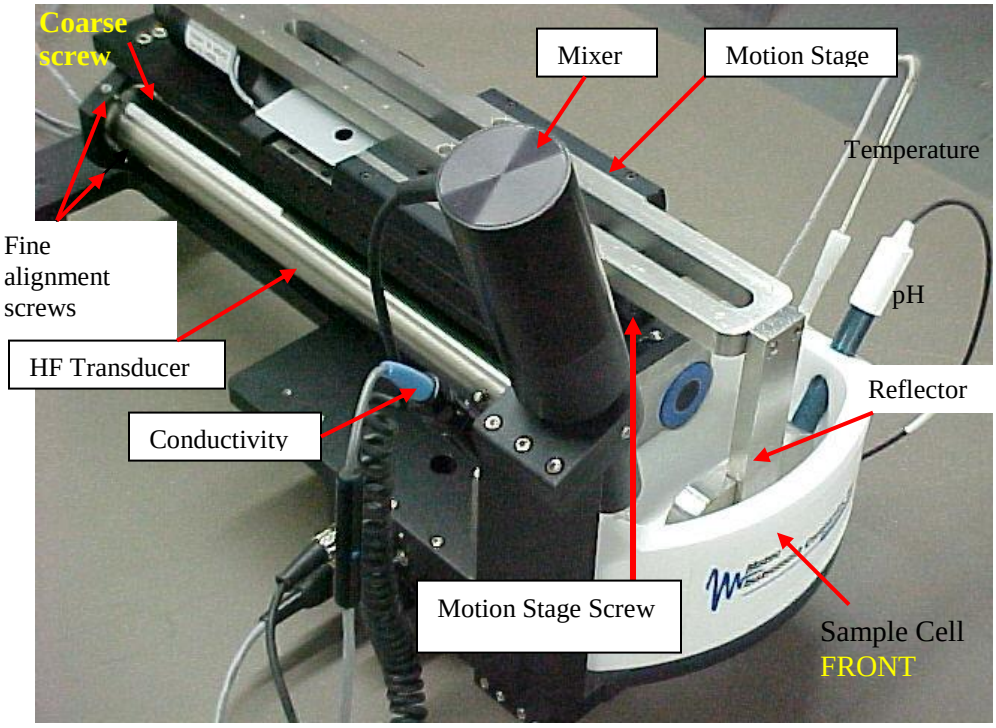

Coarse
screw
Mixer
Motion Stage
Temperature
Fine
alignment
screws
pH
HF Transducer
Reflector
Conductivity
Motion Stage Screw
Sample Cell
FRONT

Fig. V.2.

Appendix VI

Classical Electrokinetic Phenomena

The Zeta-APS System from Matec Applied Sciences takes a unique approach to measuring the **electrokinetic** properties of colloidal suspensions. This approach relies on the system's ability to take advantage of these electrokinetic properties to manipulate the suspended particles and measure and analyze the resulting motion.

When a solid particle or surface is brought into contact with a liquid phase, that solid develops a net surface charge. This surface charge may result from a variety of mechanisms, such as the dissociation of ionic surface groups, the absorption of charged material from solution, or unequal dissolution of ions from an ionic crystal lattice.

Surrounding the surface of the particle or solid surface is an area of oppositely charged "counter-ions" in the liquid phase that balances the net surface charge of the solid. The charged surface and these surrounding counter-ions form an electric double layer. This particle surface charge and the electrostatic repulsion that exists between similarly charged particles is the primary stabilization mechanism for lyophobic colloids.

The separation of charge that exists where the particle and the liquid meet gives rise to several dynamic phenomena associated with colloidal systems called electrokinetic phenomena. There are four classical types:

1. **Electrophoresis**: The motion of charge particles in a liquid under the influence of an externally applied electric field.

2. **Electroosmosis**: The motion of liquid past a charged solid surface under the influence of an applied electric field.

3. **Streaming Potential**: The potential difference that results when a liquid is forced to flow past a stationary solid surface by the application of a pressure gradient.

4. **Sedimentation Potential**: The potential difference developed when particles settle in a liquid under the force of gravity.

The driving force behind electrokinetic phenomena is not truly the surface charge per se, but rather the net charge of the liquid that is hydrodynamically bound between the particle surface and the rest of the fluid. This area of interface is called the **slipping plane or shear plane** and the potential at this interface is the **Zeta potential**. Determining this Zeta potential is the key to characterizing the electrokinetic properties of many disperse systems. All four types of electrokinetic phenomena involve the relative motion between the liquid and the charged solid surface and the behavior of this motion is governed by the Zeta potential of the solid. Furthermore, electrophoresis and electroosmosis have in common that they involve a characteristic motion caused by the application of an external electric field. Streaming potential and sedimentation potential involve the potential that occurs when motion is induced in a system by an external force, either an applied pressure gradient or gravity.

Electroacoustic Phenomena

In addition to the four classical electrokinetic phenomena, the Zeta-APS System also takes advantage of two more fundamental electrokinetic properties of disperse systems called **electroacoustic** phenomena.

When an alternating electric field is applied to a colloidal dispersion, the Zeta potential of the particles will cause the particles to oscillate in the electric field. If there is a density difference between the particles and the liquid, this oscillation will result in the transfer of momentum to the liquid and the development of an acoustic wave. This behavior was discovered at Matec and has been termed the **Electrokinetic Sonic Amplitude** or ESA. **ESA** is the pressure amplitude generated by the colloid per unit electric field strength and has SI units of Pascals per volt per meter. ESA is analogous to electrophoretic mobility, which is particle velocity normalized by the the applied electric field. See Figure 1.1.

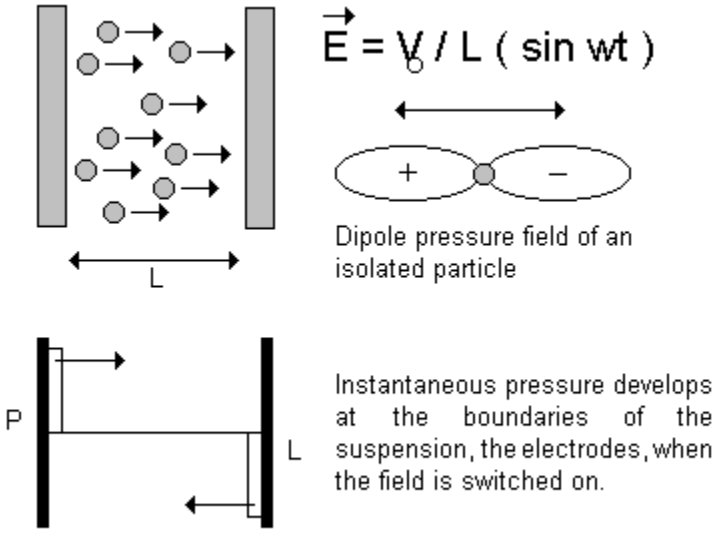


Figure 1.1 Diagrams describing the ESA Effect

Applying an alternating pressure field (acoustic wave) to a colloidal dispersion creates the inverse of the ESA effect. A density difference between the dispersed phase and the continuous phase will cause relative motion between the diffuse layer and the oppositely charged particles resulting in an induced electric dipole at the frequency of the applied pressure wave. This effect is termed **Ultrasonic Vibration Potential**, or **UVP**, and it was first predicted for electrolyte solutions by Debye in 1933. UVP is measured in units of volts per unit velocity amplitude of the applied acoustic wave, or volts per meter per second. In 1938, Rutgers and Hermans pointed out that the UVP effect would occur in dispersions of colloidal particles, and, in 1951, Enderby presented a detailed theory of the UVP effect in colloids, also referred to as the **Colloid Vibration Potential**, or **CVP**. Extensive studies of the UVP effect in electrolyte and polyelectrolyte solutions have since been carried out by Yeager et al. O'Brien developed a general theoretical treatment of electroacoustic effects in dilute colloidal systems and derived a reciprocal relation linking the ESA and CVP effects. Recently, electroacoustic phenomena have been reviewed by Babchin et al. Please refer to Figure 1.2 for an illustration of the CVP effect.

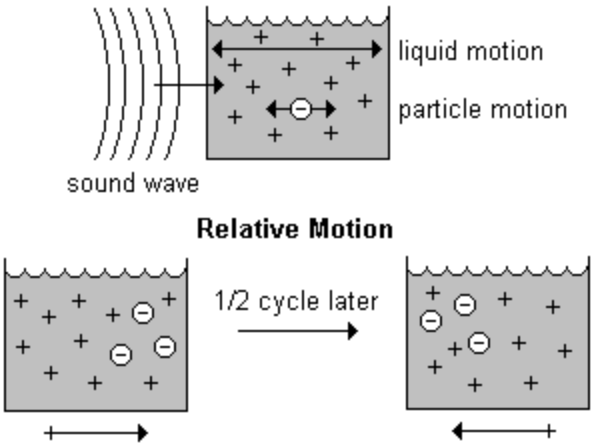


Figure 1.2 Diagrams describing the CVP Effect

It is clearly demonstrable that the electroacoustic effects, ESA and UVP, are true electrokinetic phenomena. They both involve the relative motion of a liquid with respect to a charged solid surface and the driving force for each is the solid's Zeta potential. One can relate ESA to electrophoresis and electroosmosis since it also involves the motion of the particles when the system is stimulated by and applied electric field. The UVP effect is more similar to the streaming potential and sedimentation potential in that it is the electrical potential that arises when a motion is induced in the system by an external force other than an electric field.

Electroacoustic measurements offer considerable advantages over previously employed techniques. Electroacoustic measurements can be made on dispersed systems over an extremely wide concentration range. The only upper limit on particle concentration is the viscosity of the sample; it must remain a fluid. The lower concentration limit is controlled by the ESA or UVP signal strength. The most common range of measurements can be made on systems with a wide range of particle size, from molecular size up to around 30 microns, at 1 to 30 volume %, far beyond the range possible with microelectrophoresis. It is not necessary for the particles to be microscopically visible and making measurements on opaque suspensions presents no difficulty.

An added advantage of the electroacoustic measurements is that they can be made on flowing or stirred systems. The stirring motion of the particles is far too slow to interfere with the high frequency electroacoustic measurement. Neither can motion induced by thermal convection interfere with the measurement, and any effect due to particle sedimentation can be avoided by keeping the suspension well agitated.

In addition, electroacoustic effects are virtually instantaneous, making the measurements very rapid. They are bulk measurements, made on concentrated suspensions, and thus they are not readily susceptible to errors caused by small amounts of contaminants.

Furthermore, the measurement process readily lends itself both to automated titration and the simultaneous determination of other suspension properties, such as pH and conductivity. The ability to perform titrations while taking the electroacoustic measurements is an enormous practical advantage. Users are not generally interested in a single Zeta potential at one condition but instead need to measure the Zeta potential as a function of some relevant experimental parameter like pH or dispersant concentration.

Appendix *VII*

Burette (Titrator) Setup

The Zeta-APS Burette can be used to perform automatic Potentiometric (pH), as well as, Volumetric titrations.

Connect the Burette Communication cable to the RS232-IN port on the Burette's Rear Panel (phone jack), and the 16-pin serial connector to the port on the Zeta-APS' rear panel. Turn on the Burette using the power switch.

For Potentiometric Titrations, fill the supplied titrant bottles with acid (left-side syringe), and base (right-side syringe). For Volumetric titrations, select from the software which syringe (which side) to use for your reagent.

It is important to remove all air from the inlet and output tubes, as well as the syringe(s) being used before starting a titration. Important: place the Dispensing Pipette(s) in a waste container (not your sample) before priming.

In order to perform priming, use either the Prime mechanical Switch on the Burette's front panel, or the Prime button on the software. The mechanical switch moves both plungers simultaneously while the software allows selecting which side to prime. See figure below for tubing arrangement.

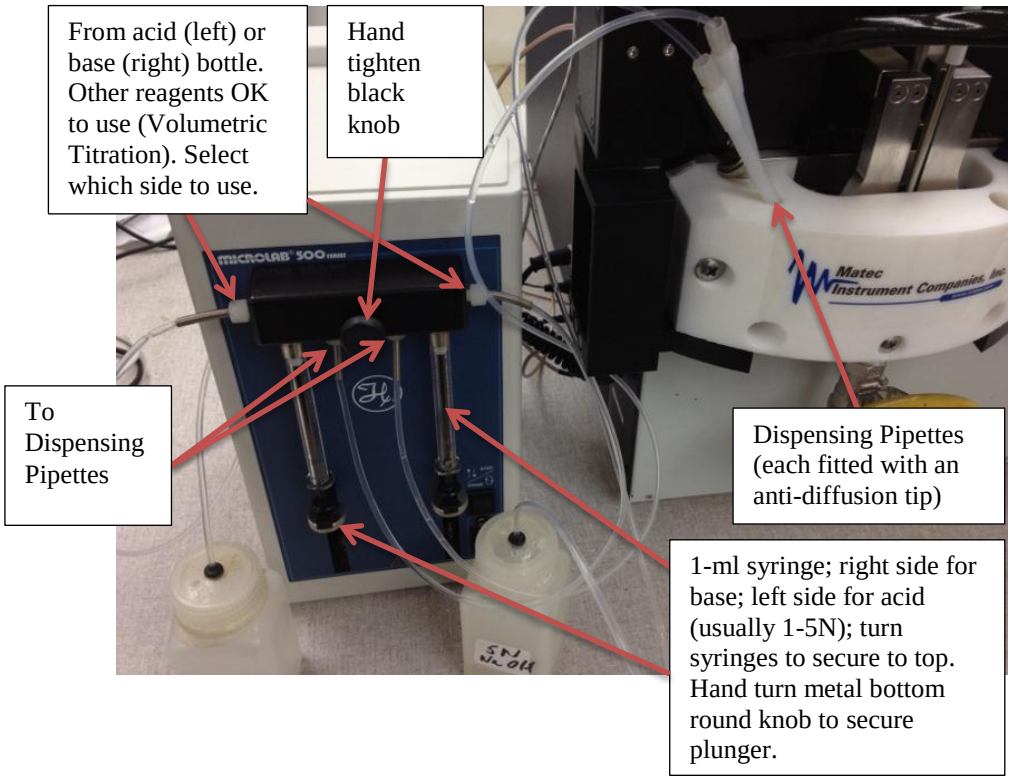
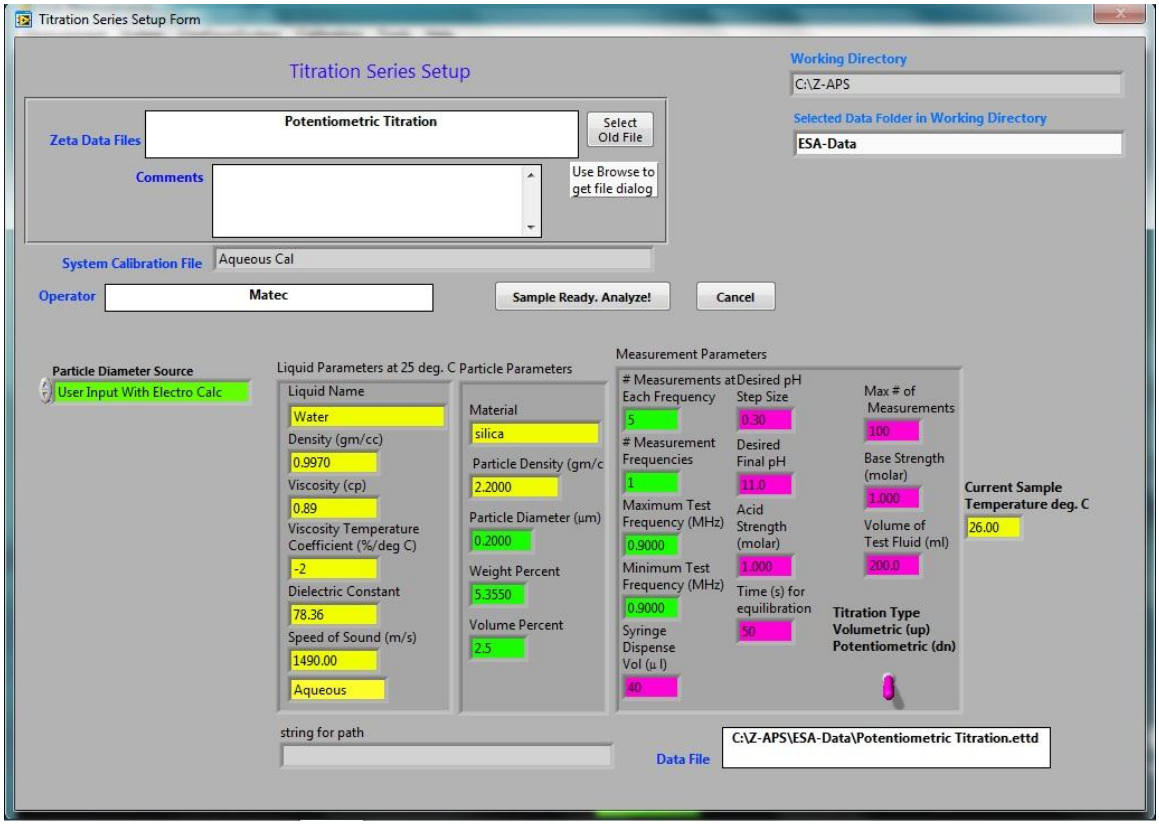


Fig. VII-1. Burette Tubing Arrangement.

The screen capture below shows some reasonable entries for a titration setup in the Zeta-APS (also ZA500, and Zeta*Finder*) software. Please refer to the Software section of this manual for more setup details.

Some important reasonable entries are as follows: (see screen capture below; Note: the window below is slightly different from the setup window; the idea is to show you reasonable values to use during setup).

pH Step size (delta)= 0.2 to 0.4
Acid or Base Concentration= 1-5 N (most commonly 1-3N)
Sample Percent Solids Level= 1-10% Vol (enter either Weight or Volume; the software calculates the other).
Titration pH Range= 2-12 (0-14 is possible but sample may change).
"Syringe Dispense Volume ul" actually refers to the first reagent addition during the titration (about 40 µl). Upon this first addition, the software calculates the slope of delta-pH vs. reagent volume in order to estimate further reagent volume additions.
Minimum and Maximum Test Frequency recommended value is 1.0 MHz (select under Configure System/ System Options; Note: Zeta must be pre-calibrated at the selected Frequency using Ludox 2.5% vol)



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