

# Operating Manual of Dispersion Technology Instruments

DT-1202: particle size and zeta potential

DT-100 : particle size

DT-300 : zeta potential

DT-310 : zeta potential with titration

DT-330 : zeta potential universal

DT-600 : rheology longitudinal

## Options available:

- conductivity aqueous
- conductivity non-aqueous
- pH/temperature measurement
- titrations
- porous body characterization
- rheology
- temperature heating control
- porous materials characterization

Dispersion Technology Inc, 364 Adams Street, Bedford Hills, NY 10507  
914-241-4777, [service@dispersion.com](mailto:service@dispersion.com)

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## Hardware, general overview

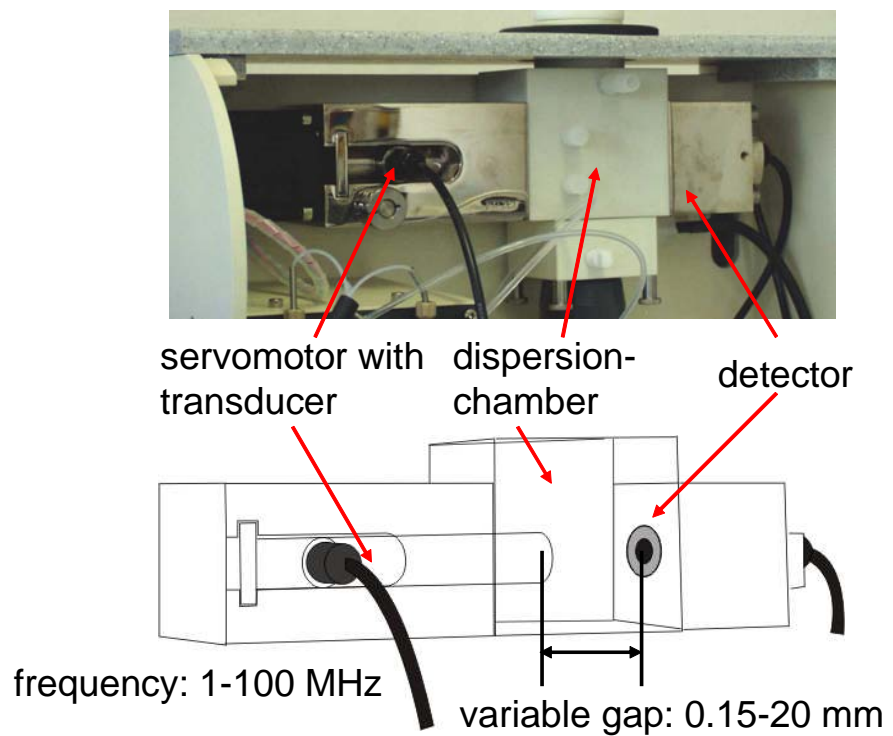
All Dispersion Technology instruments that are based on ultrasound use the same electronic block with built in computer with MS Windows HP operating systems, as shown on Figure below.



Difference between instrument models is related to a set of sensors used for different models. For instance Figure below shows model DT-310 that includes Zeta potential probe and 2 burettes titration. Model DT-300 is the same, but with no titration.



Models DT-1200, DT-100 and DT-600 include a special sensor block shown on the first Figure. This block contains Acoustic sensor presented on the next Figure:



There are several more optional sensors: pH probe, temperature probe, aqueous conductivity probe, non-aqueous conductivity probe and heater for temperature control.  
All these probes can be connected to the panel with outlets that are unique for each probe:



Photographs of individual probes are below:



pH and temperature probes



heater





aqueous conductivity probe



non-aqueous conductivity probe

There is a section “Sample handling systems” that describes variety of ways these probes can be used for characterizing liquids and dispersions.

## Getting started – turning on power and starting software

For convenience, you can leave the system always turned on, with the DTI software running, so that it will always be available for measurements as needed.

Turn on the Electronics box by depressing the power button on the front panel.

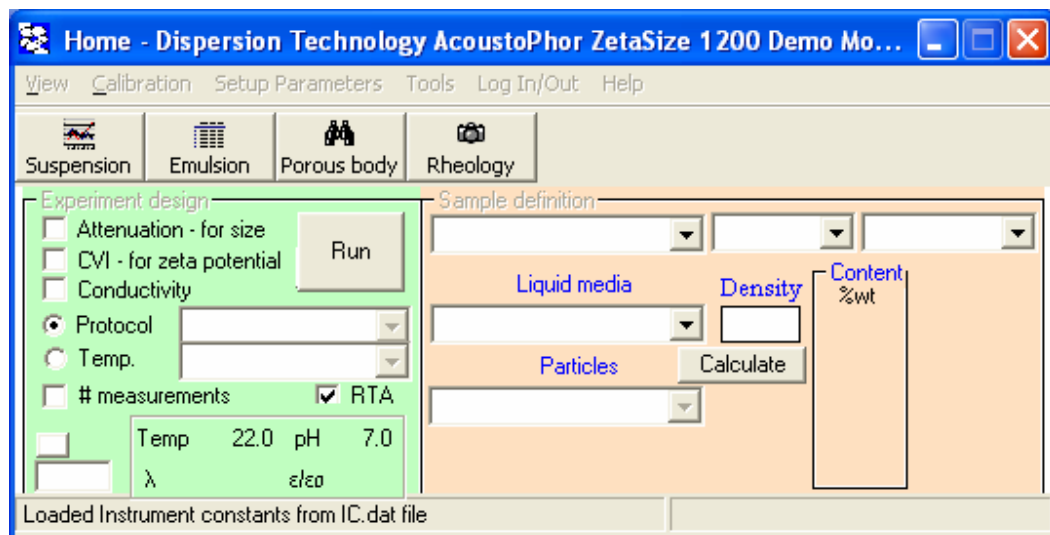
You will see a black DOS window that report tests of the communication links between computer and the electronic block.

The Windows operating system should be open within a minute or so. You will see DTI software icon on the left top corner.

If the DTI is already powered up, check to see if the DTI program is running. If it is running, either the “Home” window will be visible, or it will be minimized and shown only as an icon on the status bar at the bottom of the screen. If the program is running but minimized, click on DTI icon on the status bar at the bottom of the screen.

If the DTI program is not running, double click on the DTI icon. You will see combination of 3 windows appearing on the screen.

Top one is DTI software Home Page.



The other two are message asking you to wait till installation finished and Instrument Status for. There is description of this form in Calibrations. .

## Start Up - Dispersion Technology Instrument

Welcome! ..... Please wait a moment while your instrument is tested.....

This technology for characterizing particle size, zeta potential and rheology of concentrated colloids, dispersions, and emulsions is brought to you by Dispersion Technology, the leader in Acoustic and ElectroAcoustic Spectroscopy.

### Instrument Status - during Self Test

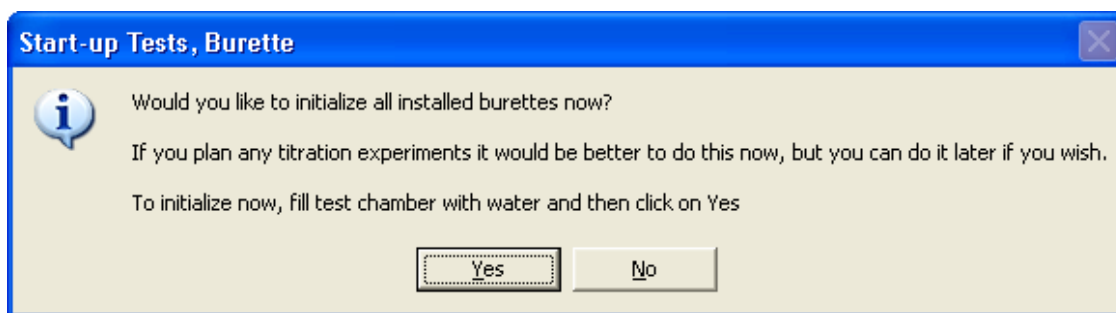
Signal Processor	installed
DAQ	installed
Motor Control	installed
Acoustic Sensor	installed
CVI Sensor	installed
Conductivity probe	not installed
NonAq Conductivity	not installed
Dielectric probe	not installed
Temperature probe	installed
pH probe	installed
Burette 1	installed
Burette 2	installed
Sample Heater	not installed
Sample Cooler	not installed
RS232 Comm	not installed
NonAq media	not installed
Digital Stirrer	installed

Miscellaneous

System SN	221
Burette Com Port	2
RS232 Com Port	1

Each time the DTI program starts it performs tests to check the performance of each installed component. The Instrument Status form, shown at right, indicates the status of each component as the test proceeds.

If one or more burettes are installed a message box, such as shown below, will appear that asks if you want to initialize the burette now. Answer No, as we will do this later.



When the start-up tests are complete, the Home page will appear as shown below. “Ready for measurements!” will appear on the status bar at the bottom of the window.

## Basic Operating Instructions

Making a measurement and printing the results is simple. This section provides a quick overview of the sequence of steps necessary to do a single measurement. Succeeding sections describe these steps in more detail to answer any questions. This section assumes you are using the instrument in its basic configuration, i.e. all sensors combined into a single assembly mounted in the Measuring Unit.

There are 10 steps to making a single measurement,

1. Empty any sample from the chamber.
2. Fill the chamber with your new sample.
3. On the Home page, click on each type measurement that you want to perform.
4. Select the name of the media from list of materials.
5. Select whether your disperse phase is a solid or liquid.
6. Select the name of the disperse phase from list of materials.
7. Enter the weight fraction of your sample.
8. Press the Run button.
9. Wait till the measurement is complete, and the analysis of results appears.
10. On Analysis report, click on File- Print report.

Before making measurements you might consider logging in following instruction from Advanced Topics.

### *Getting familiar with the basic sensor configuration*

In the basic sensor configuration the acoustic, Cvi, Temperature, and Conductivity sensors are all installed together so as to form a common “sample chamber “. The chamber is mounted inside the Measurement Unit, which is shown below. A top and front cover protects the sensors from exposure to spilled sample or cleaning fluids.

Some users prefer to work with the covers removed. The front cover can be easily removed by pulling it forward. The top cover is removed by first removing the knurled ring around the filling port, loosening the screws on each side plate, and then lifting the top cover.



### ***Emptying and cleaning the chamber, basic sensor configuration***

The chamber should always be left filled with some appropriate clean “storage” fluid, e.g. water. This will keep any residual contaminants from drying out on the surfaces, in which case they would be more difficult to remove.

Your first step in measuring a new sample is to empty any storage fluid or any previous sample.

If the pH probe is inserted in the cap, remove it and insert it in the storage fixture for now.

Remove the cap at the top of the chamber and put it aside.

Place a beaker below the chamber set the rotary valve to open all three ports so that the sample is emptied. The handle points to the open ports.

Rotate the handle of the valve to close the exit port but allow circulation of the sample.

Fill the chamber with some appropriate cleaning fluid depending on the last sample that was measured (e.g. plain water, water with detergent, solvent, etc). For now, we will use just plain tap water.

If necessary, use the supplied nylon brush to clean the chamber. Note: It is not important that the chamber be spotlessly clean. Some discoloring of the walls of the chamber after using various pigments is not a problem. This is one of the advantages of working with concentrates; they are not easily contaminated in a significant way.

Rotate the valve to the empty position and let the sample run out. Then rotate the valve again to the circulate position.

Continue on to the next section. Do not stop here! We always want to leave the chamber filled with fluid when it is not being used. If the chamber is left empty, any residual contamination will dry on surface and may later be difficult to remove. Wet surfaces stay cleaner than dry surfaces.

### ***Filling the empty chamber with a new sample, basic configuration***

Empty the chamber of any fluid as described above. Rotate the valve to the circulate position. Turn off the stirrer.



The basic configuration requires about 120 ml of sample. Fill the chamber with sample as shown in figure at right. As the sample chamber is filled, the fluid will also rise in the external bypass tube that connects the bottom to the top portion of the sample chamber. The sample chamber should be filled till the fluid reaches the return port at the top of the chamber. It should not be overfilled since, as the acoustic sensor closes, the level will rise approximately 6 mm further.

### ***Stirring the sample, if equipped with digital stirrer***

If your unit is equipped with a digital stirrer the speed is controlled from the keyboard. The digital stirrer provides constant speed, independent of the viscosity of the sample.



The stirrer button and dial are located on the bottom left corner next to the box with number 7 in it. This number indicates speed of magnetic cross rotation.

The stirrer can be turned on by clicking on the stir button at which point it will turn red to indicate the stirrer is on. If you click the button again the stirrer will turn off and the red light will go out. You can adjust the speed by first displaying the desired speed (using the list box scroll keys) and then clicking on it so that it is displayed with blue background. The indicated value is the stir speed in revolutions per second. It is sometimes more convenient to change speed using the UP/Down keys on the keyboard which works immediately after you turn the stir motor on.

Sometimes it is convenient to have the stirrer turn on automatically whenever you start a new experiment. You can arrange this on the Home Page by clicking on Tools Options and then checking the box “AutoStir On at Start of Experiment” as shown in figure below.

In a similar way you can arrange to turn off the stirrer automatically at the end of an experiment by checking the box “AutoStir Off at End of Experiment”

### ***Preparing pH probe for first use***

If pH probe has not been previously used, remove it from box in which it was received. A sleeve will normally be installed over body of probe as shown at right. Holding probe vertically, check level of fluid in probe.

When the pH probe is not being used, it should be stored safely in the provided storage tube or in the pH stand

***Caution: The tip of the pH probe is delicate and easily broken.*** Take care in moving the pH probe from place to place.

Before using the pH probe it is necessary to slide open the fill port at the top of the pH probe, as shown in figure to right. (*This allows a small amount of saturated filling solution to pass through the small reference junction. If this port is left closed, the pH probe may develop a somewhat sluggish response.*)



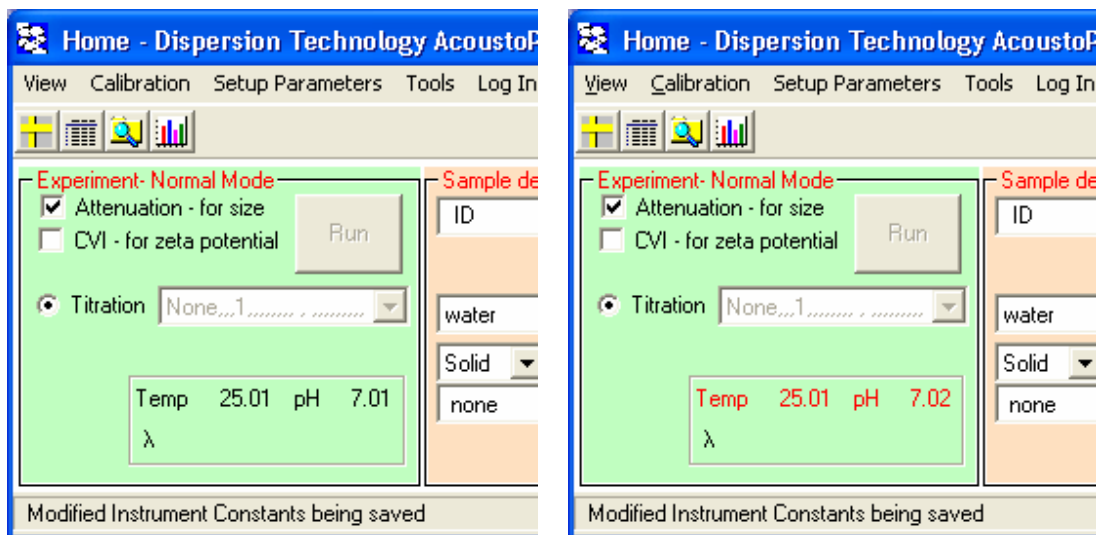
Install the cap on top of the Chamber. It is held in place with an O ring around the side. Twist it slightly while pushing downward so that it is seated on the top flange of the chamber.

***It is important that the pH probe is not inserted too far, or the probe will be broken during attenuation measurements as the gap is closed!*** A 12 mm long white spacer, also shown in the figure to the right, should be installed on the body of the pH probe that keeps the tip of the probe extended at least 40 mm from the bottom of the cap, ***but no more than 45 mm.*** \*

Carefully insert the pH probe into the Cap as shown in the figure.

### ***Displaying temperature and pH, automatic and manual entry***

A platinum resistance temperature probe is installed in the bottom sensor block and protrudes about 10 mm into the sample so as to precisely monitor the sample temperature. The present value of the sample temperature is displayed on the Home page. Check that the displayed temperature is approximately that of the room temperature.



Freshly distilled water has a pH of 7.0. In coming into equilibrium with the CO<sub>2</sub> in the atmosphere, the pH gradually decreases to about 5.5. Check to see that the pH reading of your distilled water is reading in the range of 5.0.

There may be times when you want to enter the sample temperature manually, instead of measuring it. Double click on the label “Temp, C” on the Home page. Note that this label becomes red in color, indicating that it is no longer being measured. Now type in any temperature value, e.g. “30.6”. This new value will be accepted and, in fact, will be recorded in the database as if it were the actual temperature at which succeeding measurements are made. Double-click again on the temperature label, and note that the label returns to the original color and that the temperature displayed is again the actual measured value. It is important to understand that the temperature that is entered does not control the temperature; it only sets the temperature that will be recorded to the database for succeeding measurements.

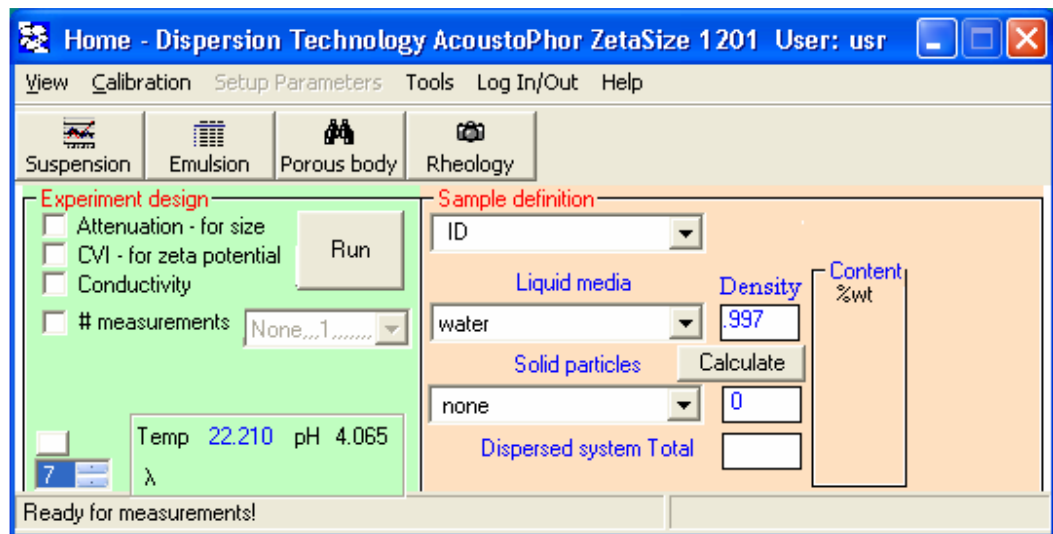
The pH value can also be entered manually in the same way as temperature. Double-click on the label “pH”. The label text turns to a red color. You can now edit the pH value. This value will be recorded in the database for all succeeding measurements until the box is double-clicked again. Double-click again on the pH value to return to monitoring pH.



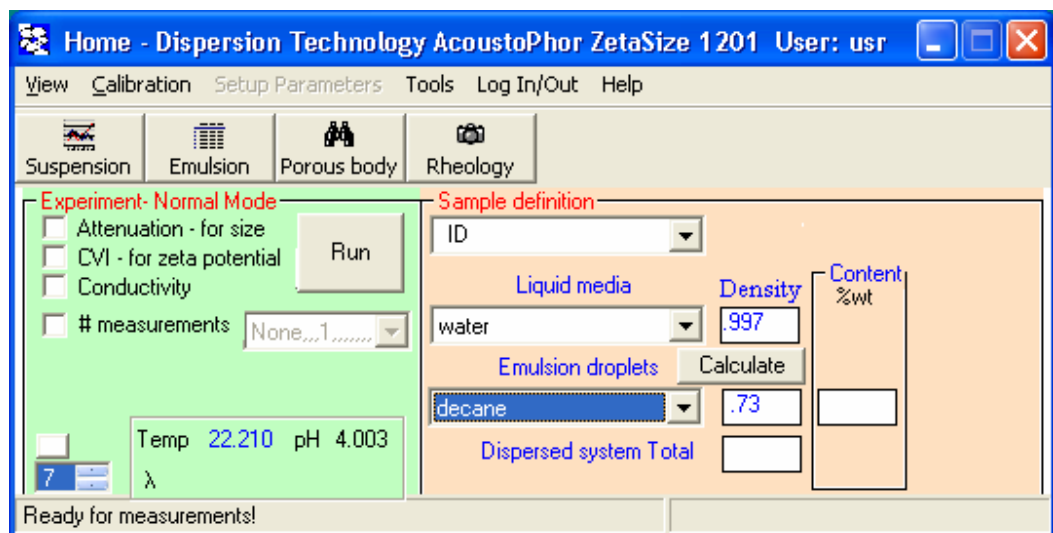
## ***Sample types: Suspension, Emulsion, Porous body, Liquid Rheology***

DTI instruments are capable of measuring wide variety of natural samples with practically no sample preparation. DTI software Home page has 4 different setups for 4 different types of samples specified in the title of this section.

In default it becomes loaded for Suspension samples, as shown below. Material of particles is marked as “Solid particles” and combo box becomes filled with only solids from the Material database. .



If you click on the button Emulsion, material of particles changes to the “emulsion droplet” and combo box becomes filled with only liquids existing in Material database.

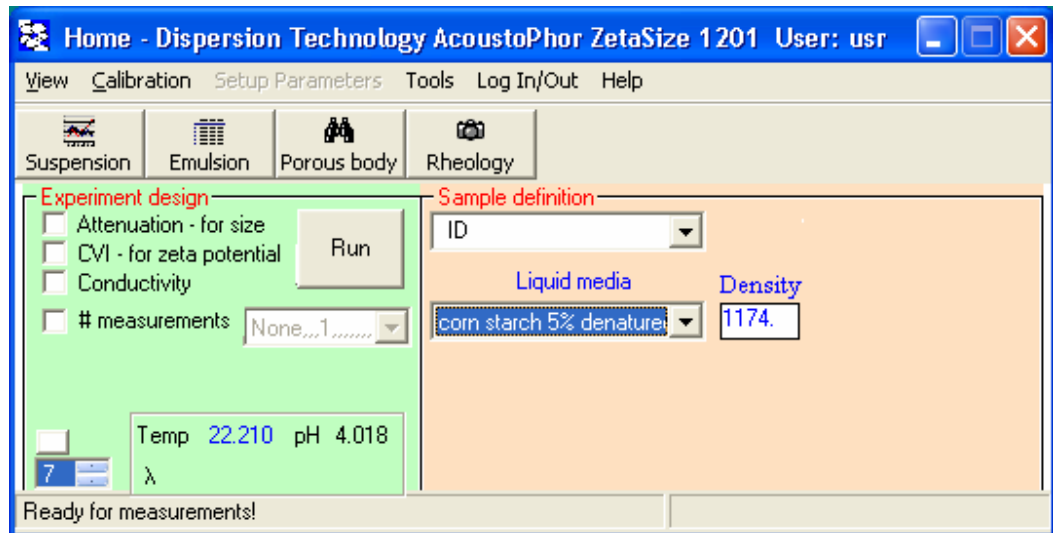


If you click on button Rheology, line for particles disappears. Liquid is treated as homogeneous for purpose of rheological study.

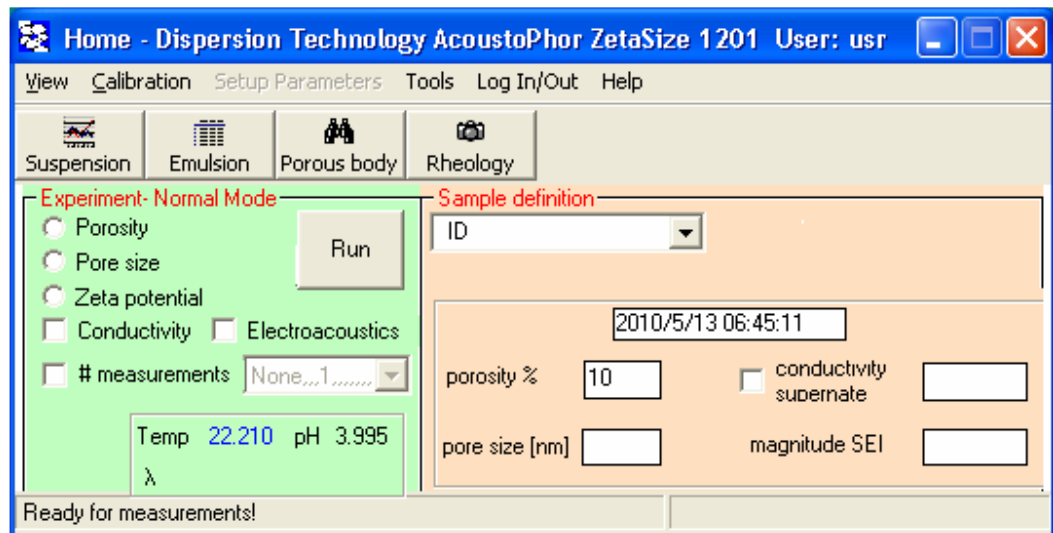
It is important to stress here that line between “homogeneous” and “heterogeneous” liquid is conventional. For instance, MILK can be treated as homogeneous liquid with certain visco-elastic properties, or, alternatively, as emulsion of fat droplets in water.

It means that ANY dispersion or emulsion can be still treated as homogeneous liquid and characterized in rheological terms.

Basically, DTI instrument with Acoustic sensor can be described as particle sizer and rheometer in the same instrument.



If the click on the button Porous body, the following form appears.

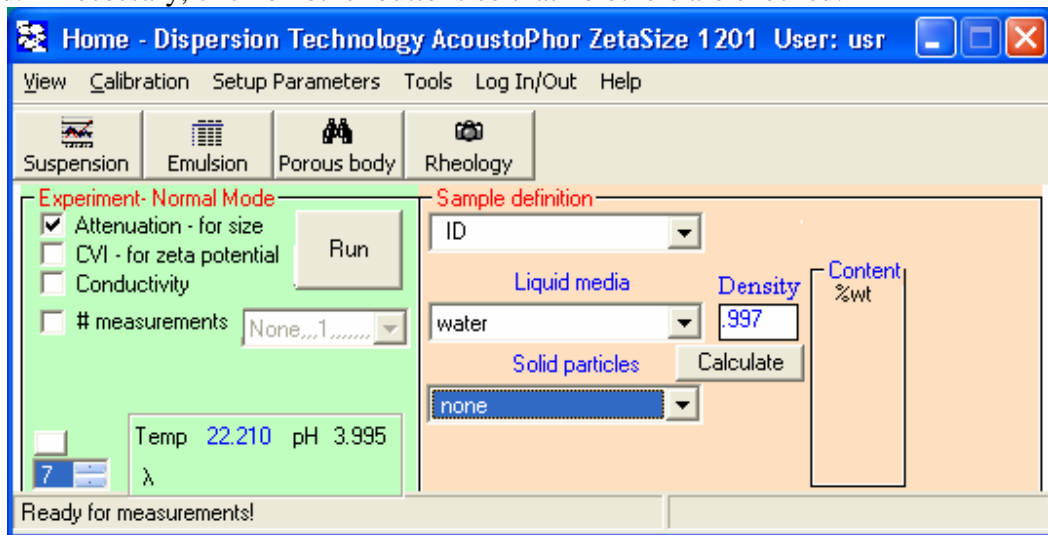


This is interface for measuring properties of porous bodies. There is a special section on this subject.

## Checking system by measuring intrinsic attenuation of water

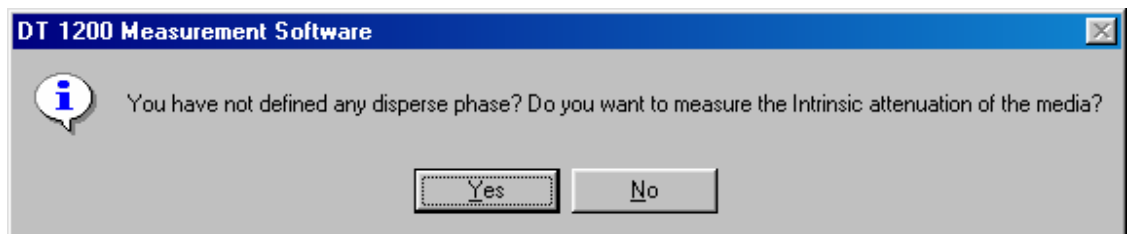
The DTI Acoustic sensor measures attenuation over a very wide dynamic range. Pure water has an attenuation which is less than any of the colloids that you might be interested in, so it is a convenient sample for testing the performance of the instrument at an extreme condition.

In the Experiment design frame, click the “**Attenuation – for size**” button so that it is checked. If necessary, click on other buttons so that no others are checked.



In the Sample definition frame, see that the Liquid media is **water** and the particles are defined as “**none**”. This is the default condition when the program starts, so you will not need to make any changes if you have been following these lessons in order.

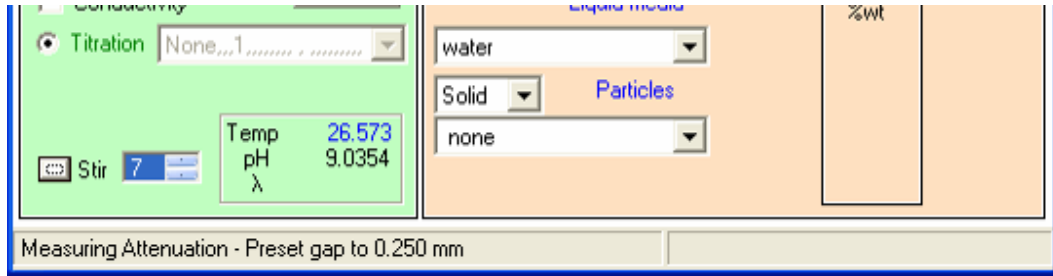
Press the **Run** button. The following message will appear since we have not yet defined any disperse phase.



We do in fact want to measure the intrinsic attenuation of water, so click **Yes**.

The Run button will initially change to “**Starting**” and then split into two separate buttons labeled “**Stop**” and “**Pause**”. We will discuss the functions of these buttons later when doing more complex titration experiments.

The status bar at the bottom of the home page, as shown below, is divided into left and right parts.



The left part of the Status Bar gives information as to what the system is doing right now. If you are doing an attenuation measurement, it will tell you that it is measuring the attenuation and will specify which gap is presently being used to collect data.

The right hand portion of the Status Bar indicates the amount of time required to complete the current experiment. If we are just making a single measurement, the required time is very short and this time estimate is not very helpful, but when doing a long titration experiment it will tell you how much longer it will take to complete the experiment.

When the measurement of your water sample is complete, the left side of the Status Bar at the bottom of the home page will indicate the time that this simple experiment was completed.

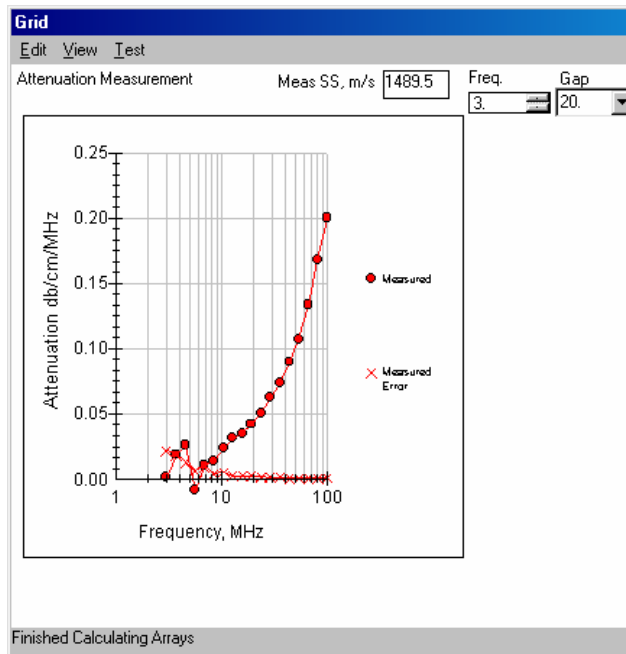
On the Home page:

Click **View > Grid Raw Data**. The Grid form will open.

On the Grid form:

Click **View > Attenuation**

The attenuation for the water sample you just measured will appear, similar to that shown below:.



The attenuation of water at 100 MHz varies from 0.018 to 0.022 db/cm/MHz depending on the room temperature. Two curves are shown in the figure. The top curve, described by the solid circles, shows the measured attenuation. The bottom curve, described by the x symbols, shows the

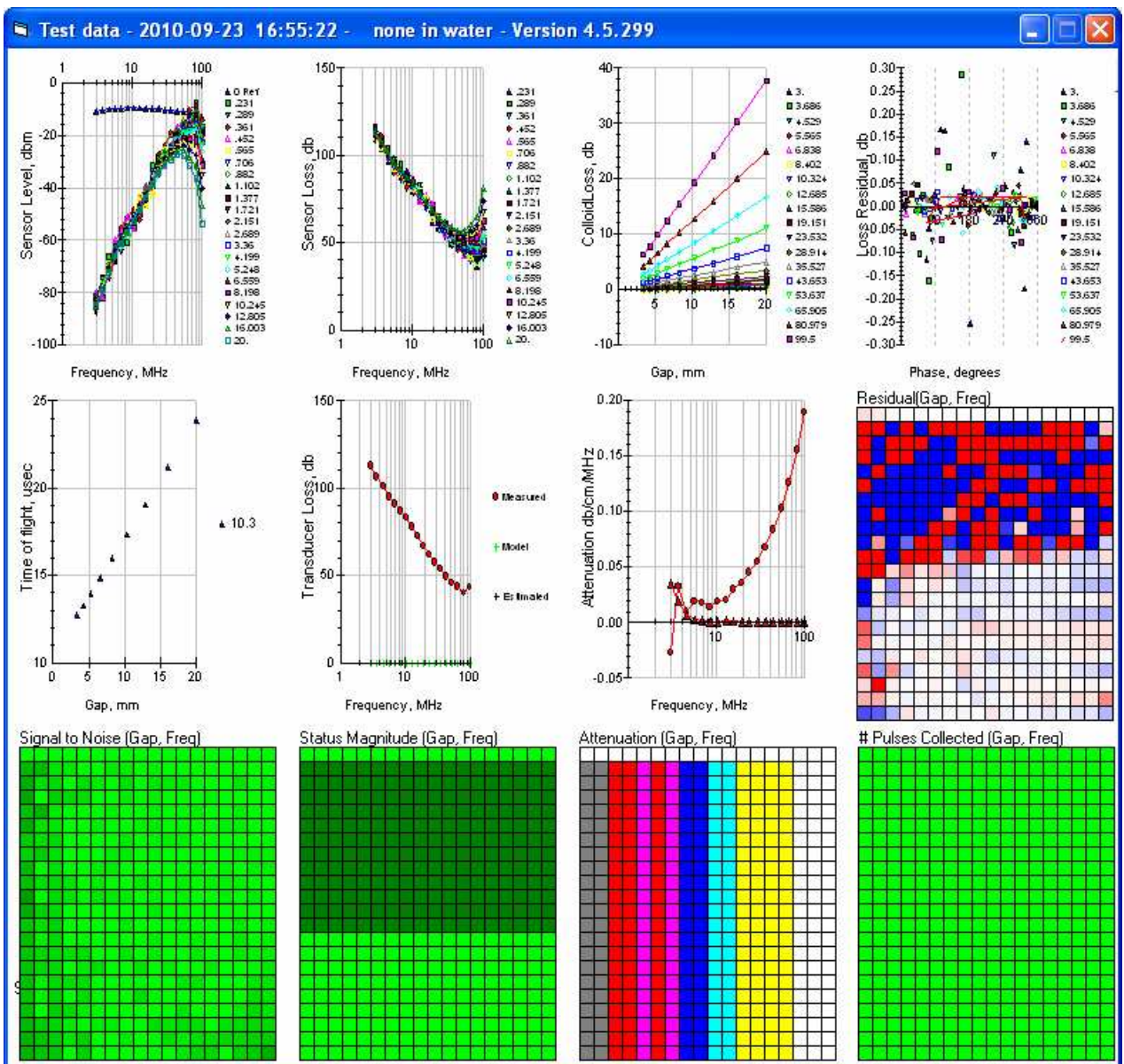
expected error for each measured attenuation. Typically the error increases as the frequency decreases, as shown in this example. The error will usually be less than 0.05 db/cm/MHz at 3 MHz.

This measurement reproduces remarkable law discovered by Stokes 150 years ago. For more details – the book, section 3.6, page 108.

The same measurement is used for troubleshooting DTI instruments. All required information is presented on the figure that is called “All graphs” on the Grid form. In order to see it:

On the Grid form:

Click **View > Attenuation**



If there is any suspicion about instrument function, you should measure attenuation of water, open this form, capture it to Clipboard by typing ALT and PrintSC together and then paste on MS Word file. Email it to DTI or DTI agents. This form is sufficient for determining reason of malfunction.

### ***Measured data and calculated parameters.***

There are two distinctively different ultrasound-based measuring methods. One is called acoustics and the other one is known as electroacoustics. They measure completely different parameters. Acoustics is simpler because a single field is involved—field of the mechanical stress. The measured parameters are usually *sound speed* and *attenuation coefficient*. The electroacoustic method is more complicated because it is based on the coupling of two fields—electrical and mechanical. The measured parameters are *magnitude* and *phase* of the electroacoustic signal.

Four raw parameters can be used as a fingerprint of a particular liquid system.

- *sound speed*
- *attenuation coefficient (usually at multiple frequencies)*
- *magnitude of the electroacoustic signal*
- *phase of the electroacoustic signal*

There is a set of complimentary raw data that can be collected with DTI sensors:

- *conductivity aqueous (at MHz range)*
- *conductivity non-aqueous*
- *pH*
- *temperature*

These raw data could be theoretically treated so that a multitude of other properties can be characterized. The theoretical treatment is the main reason for ultrasound's versatility for characterization purposes. It turns out that there are two very different sets of calculated parameters that can be extracted from the raw data, depending on degree of our *a priori* knowledge about the system.

If our prior knowledge is limited and we are forced to model a system as a homogeneous medium with unknown visco-elastic properties then the ultrasound-based method allows us to calculate following parameters:

- *viscosity longitudinal within 1—100 MHz frequency range*
- *viscous longitudinal modulus  $G''$*
- *bulk viscosity for Newtonian liquids*
- *elastic longitudinal modulus  $G'$*
- *compressibility*
- *Newtonian liquid test at MHz range*
- *iso-electric point – range of aggregative instability*
- *optimum dose of surfactant*
- *volume fraction of the dispersed phase from sound speed*
- *kinetics of dissolving, crystallization*
- *kinetics of sedimentation*
- *verification of large particle presence in opaque systems*

We would like to stress here that system might be very complex and intuitively heterogeneous but it should not prevent the application of homogeneous mode in its description. For instance, milk can be treated as a homogeneous liquid when we ignore the fact that it is actually a collection of fat droplets, proteins, and sugars in water. Basically, any liquid system can be modeled as homogeneous or heterogeneous. These models are creations in our minds for the adequate characterization of various physical and chemical properties.

In the case of ultrasound-based techniques, we can first model a system as homogeneous and calculate the set of parameters presented above. Then, as the next step, we can apply the

heterogeneous model and theoretically treat the same set of experimental raw data for extracting another set of parameters, given as following:

- *particle size distribution of solid particles with known density*
- *particle size distribution of soft particles (droplets) when thermal expansion coefficient is known*
- *volume fraction of solids at submicron range*
- *Hook parameter for particle bonds in structured systems*
- *micro-viscosity*
- *$\zeta$ -potential in particulates*
- *surface conductivity*
- *Debye length when conductivity is known*
- *ion size from compressibility in aqueous solution*
- *ion size from electroacoustics in aqueous and non-aqueous solutions*
- *electric charge of macromolecules*
- *porosity, pore size and  $\zeta$ -potential in porous bodies)*
- *properties of deposits and sediments*

Application of the heterogeneous model usually requires *a priori* information about the volume fraction of the dispersed phase. This parameter may be extracted from the raw data in some special cases, for instance when particles are rigid and their sizes range from 0.1 to 1 micron. Sometimes it can be calculated from the sound's speed as well. However, for the most reliable results, volume (weight) fraction of the dispersed phase must be treated as an independently known input parameter.

In terms of traditional measuring techniques, ultrasound can combine the descriptions of rheological, particle size distribution, and  $\zeta$ -potential into one unit instead of 3 independent instruments. This has substantial advantages over traditional methods.

It also makes possible characterization of particulates and porous bodies with the same instrument. This opens new ways for calibrating sensors.

From the software viewpoint measurement is setup by **Home page**.

All calculations are performed with **Analysis form**.

Calculations would require a proper set of input parameters. There is a special **Define Material form** for introducing a new material with appropriate set of input parameters. This form might force Analysis form towards calculation of some parameters that remained unknown after introduction of the new material has been finished. There are also some restriction on using freshly defined material if it has not been defined properly. It is all described in details in sections dedicated to the particular forms.

## **Quick start: particle sizing and zeta potential with 10 wt% colloidal silica sample.**

The next step in learning DTI instrument functions is measurement of 10 weight % dispersion of colloidal silica. This is DTI reference material, which is used for demonstrating particle sizing function and for calibrating zeta potential probe. There is newsletter #22 on DTI web site with results of round robin test of this material performed with 6 Universities in Germany.

This suspension is supplied in liquid form at a concentration of 50 wt% and must be diluted prior to measurement with 0.01 M KCl. The following procedure will prepare a sample of about 120 ml which is sufficient for making measurements using the basic sensor configuration.

Prepare a liter of 0.01 M KCl by adding .745 g of Potassium Chloride to a 1 liter volumetric flask and filling flask to 1 liter using distilled water.

Place a 250 beaker on a balance and tare it. Weigh out 25 g of the 50% concentrate. Dilute this to a total weight 125 g using 0.01 M KCl as diluent.

This sample will be used both as a demonstration colloid for illustrating particle size and as a zeta potential standard. The particle size measurement does not require any calibration.

Once a sample has been prepared by dilution, it should be used that day and then discarded. Although the 50% concentrate is stable for a long time, the pH and zeta potential value of the diluted sample will change slowly with time.

If we only want to measure the attenuation and sound speed of the sample, we do not need to know anything about the properties of the sample. However, if we want to calculate the particle size distribution we need to know some physical properties. For sub-micron rigid particles such as silica we need to know only the density of both the particles and the media, and also the viscosity of the media.

Since we now want to measure the PSD of this colloidal silica, we need to specify the ingredients of our sample. This is done in the “Sample Definition” frame on the Home form as shown in figure below.

First we click button Suspension, if Home Page setup is not in Suspension mode.

Then we define the liquid media. Water is selected by default when the program starts and we will use this default value for the medium of our colloidal silica dispersion.. The required density and viscosity for the selected media is automatically computed from information in the Material Table in the database.

Next we need to define the particles that make up the “disperse phase” in our colloidal dispersion. By default, the particles are assumed to be solid particles. Since our silica particles are indeed solid, we keep this default condition. Next we need to specify the material that our particles are made of, in this case silica. To do this we use the pull-down box to display a list of pre-defined materials that are saved in the Material Table in the database. To find the desired material quickly, we can just type the letter “s” and the pull-down list will display those materials starting with that letter. From this list, we select the material “silica, Ludox”. The required density of these silica particles is calculated from data in the Material table.



Once we have defined the materials that compose our colloid, we need to then specify how much of each material is present in our sample. This is done inside the “Content” frame. In this



simple case we can simply type the weight % inside the appropriate box in the content frame as shown in figure above.

We have already defined the sample for our 10 wt% silica.

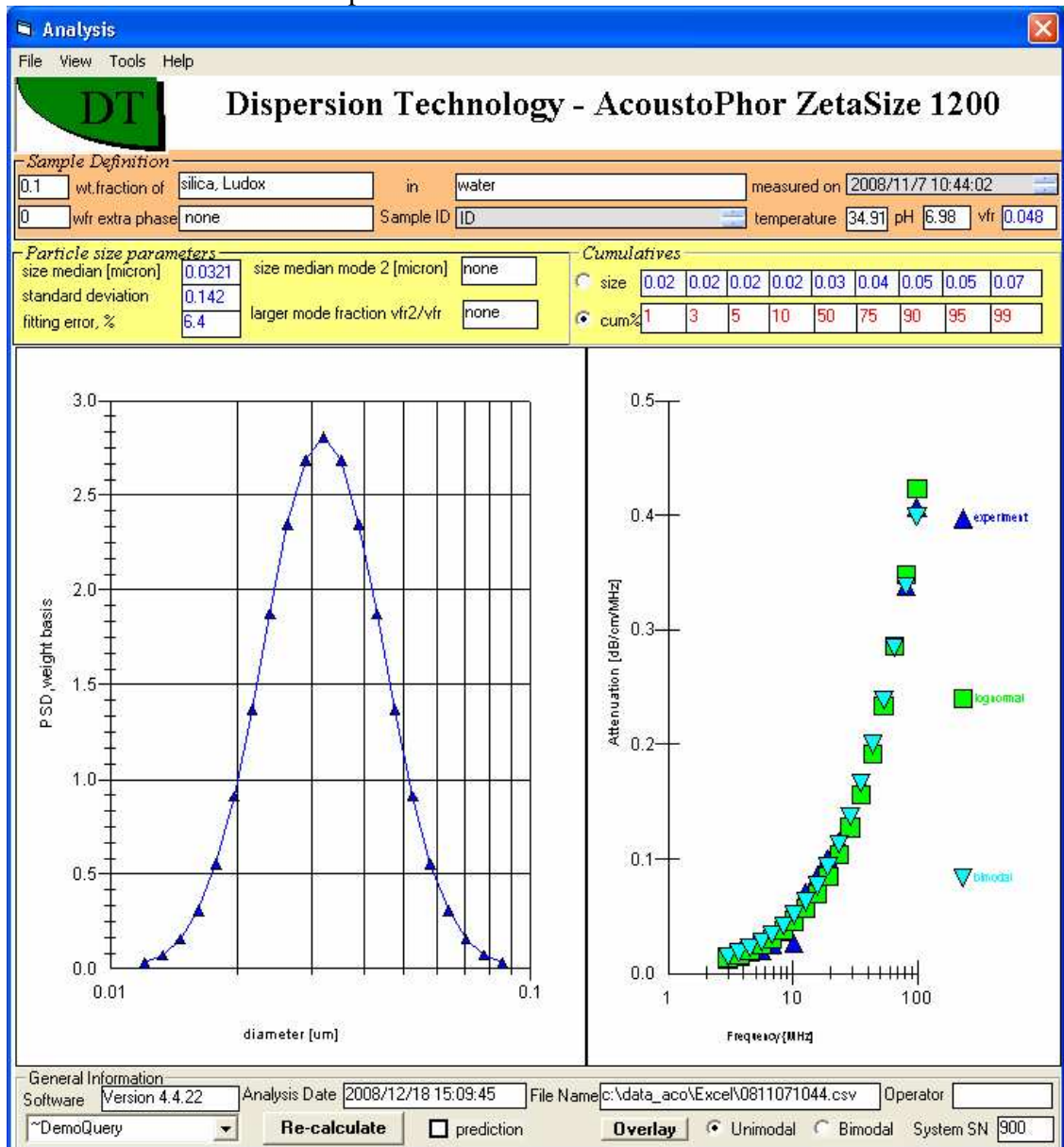
Click on the **Attenuation** box till it is checked. For now, make sure the **Conductivity** and **CVI** boxes are not checked. Click on either if necessary to uncheck it.

Click the **Run** button.

It will take several minutes to measure the attenuation spectrum.

During this time you will get updates in the left-hand side of the Status Bar as to the present gap position.

Eventually the Analysis form, similar to that shown below, will be displayed showing the particle size distribution for this sample.



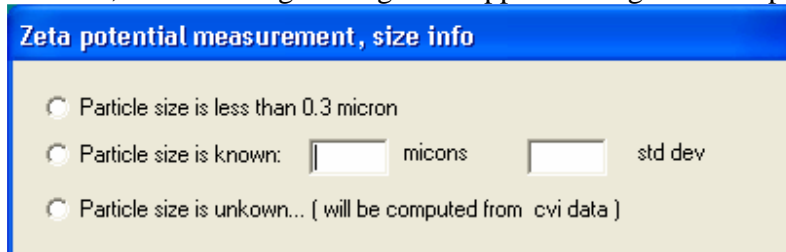
There is a special section describing various functions of Analysis form. At this point the median size of the silica particles is of the most importance – it is about 31 nm with precision of less than 1 nm.

Another point of importance – quality of theoretical fit for experimentally measured attenuation spectra – graph on the right hand side of the Analysis form. This is measure of reliability of the calculated particle size distribution.

Let's now determine the zeta potential of this 10 wt% colloidal silica. We have already defined the sample. The next step is to define the experiment that we want to make by checking the appropriate boxes in the “Experiment” frame on the Home page. We want to measure zp, so we need to check the box labeled “CVI – for zeta potential” as shown in figure below.



To make the measurement, click on **Run**. If we are asking to measure only zeta potential, but not particle size, the following message will appear asking for some particle size information.



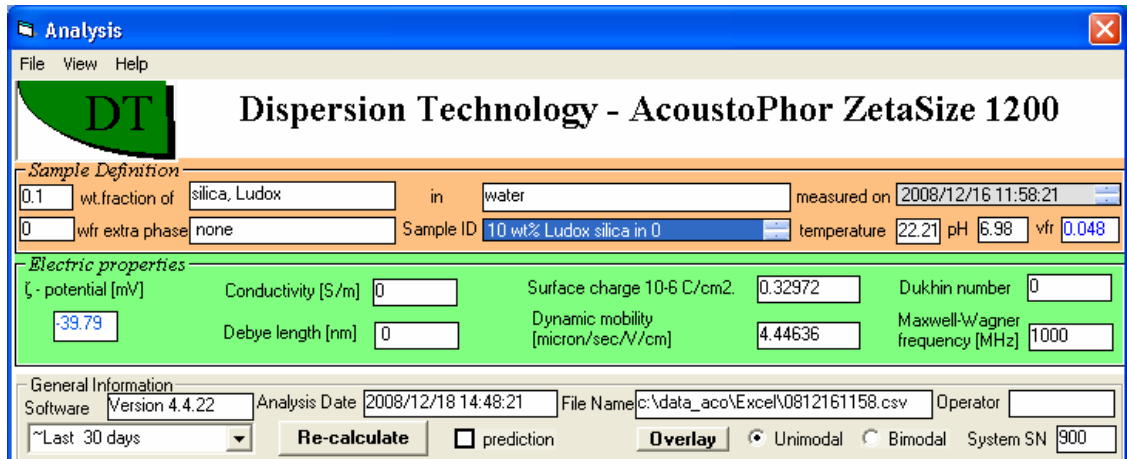
The calculation of zeta potential requires size information if the size is larger than about 0.3 microns. Below 0.3 microns, the calculation of zeta potential is independent of particle size. The colloidal silica we are using now has a size of about 0.03 microns, so you should select the first option, **Particle size is less than 0.3 um**.

If the particle size is larger than 0.3 microns and is known, then enter this known size in the box labeled “microns”. Unless the standard deviation is known, enter a value of 0.3 in the text box labeled “std dev”. Finally, select the second option box **Particle size is known**.

If the size is unknown, then check the third option box, **Particle size is unknown**.

After a few seconds, the Home page will appear with the left portion of the Status Bar indicates that the unit is now measuring the colloid vibration current.

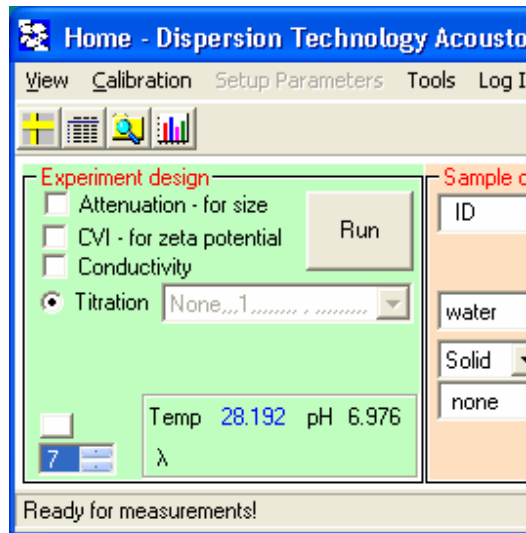
After a short time, the measurement is complete and the Analysis form appears with the calculated zeta potential, as shown below. The value should be approximately -38 mV. The calibration of zeta potential probe is discussed in the Calibration chapter.



## Quick start: conductivity non-aqueous

If the system includes a nonaqueous conductivity probe and if this probe is currently installed we can make measurements of the conductivity of a nonaqueous media in three ways.

First, if you just want to make a single measurement, without recording the data to the database, you need only click on the conductivity value on the Home page (space to right of  $\lambda$ ).



Second, if you want to make a continuous series of measurements, then on the Home page:

Click **Tools >> Monitor non-aqueous conductivity**

The conductivity measurements will continue until you uncheck this menu item by clicking on it again. The measured conductivity will appear to the right of the  $\lambda$  symbol.

Third, if you want to include a non-aqueous conductivity measurement as part of an experiment, then check the Conductivity box on the Experiment Design frame, along with any other choices such as “CVI – for zeta potential” or “Attenuation – for size”.

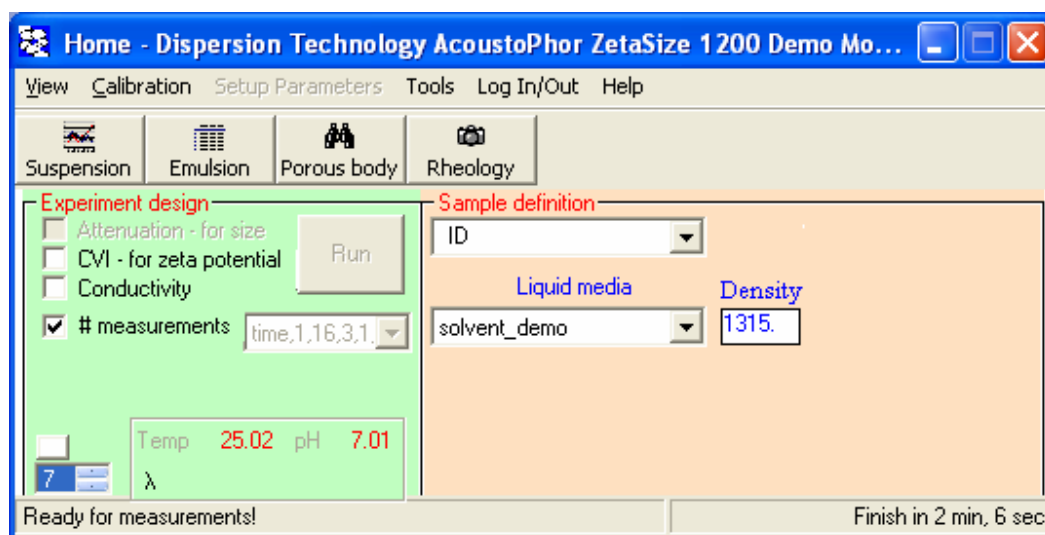
## Quick start: rheology longitudinal

Longitudinal rheology differs from the shear rheology due to the direction of the stress. There is some discussion of this issue in the paper: Dukhin, A.S. and Goetz, P.J. Bulk viscosity and compressibility measurement using acoustic spectroscopy," The Journal of Chemical Physics, Vol.130, Issue 12, (2009). Also, this paragraph ends with our paper published by SciTopics, which covers this issue in some details.

Any liquid can be described as homogeneous media with certain visco-elastic properties. Acoustic sensor and DTI software allows determination of these properties at high frequency on MHz range.

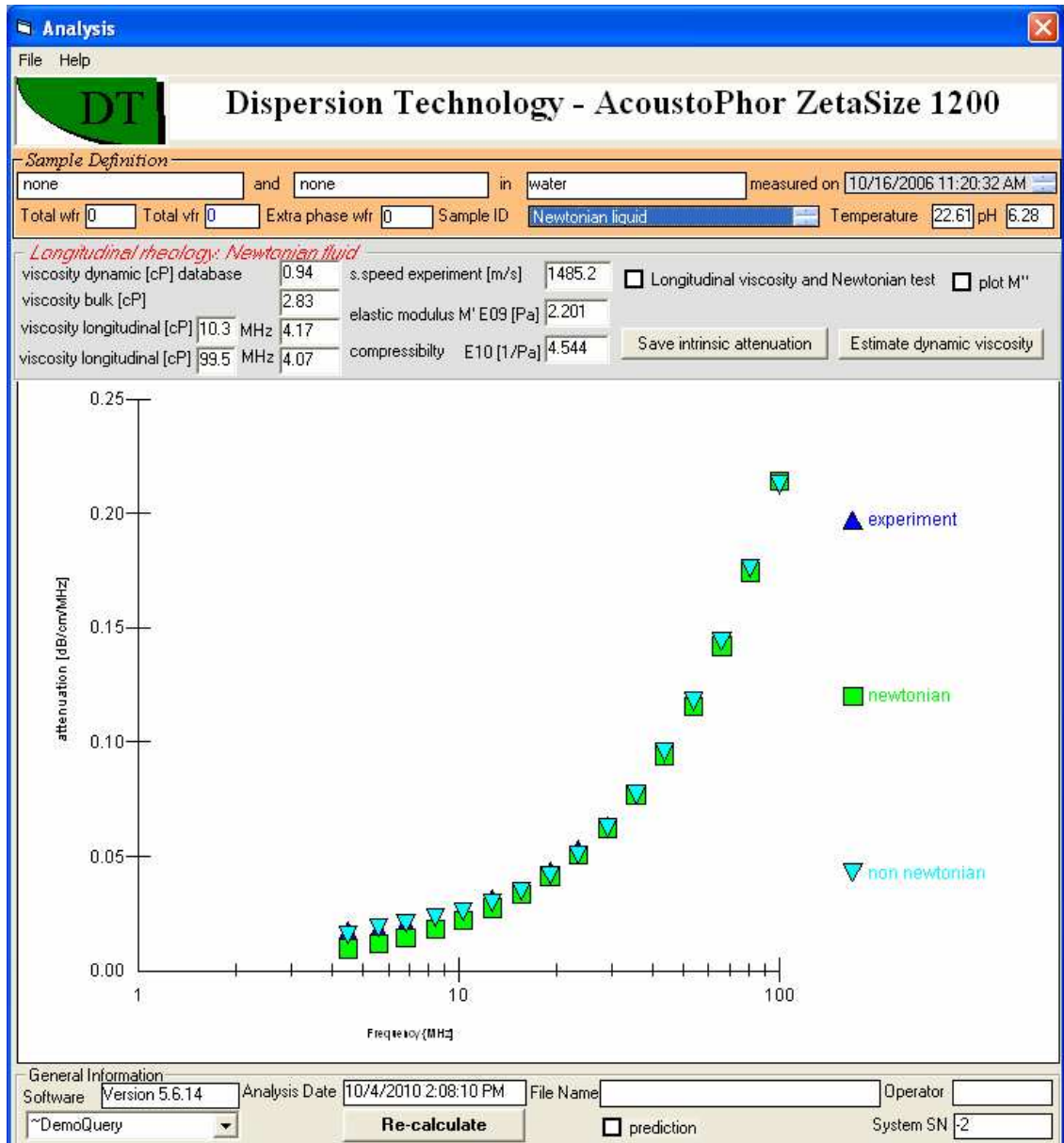
If this liquid already exists in the Material database, then user can simply select it from the list of material in Liquid Media box, as shown below with "solvent demo".

If this is a new liquid then user should simply create a new liquid using Define Material form. Important input parameter is only density, unless "bulk viscosity" is the purpose of the study. Then "dynamic viscosity" must be specified. Prototype "liquid medium for solid particles" can be used as Prototype.



We recommend to make at least 3 measurement, as shown on the Home page above, especially if this material is measured first time. This recommendation is based on possible wrong sound speed, which is selected at default value when material is created from Prototype. In the case when sound speed error would exceed 100 m/sec, measured attenuation would be wrong. However, sound speed will be correct. This corrected sound speed would make the second measurement in the set properly with correct attenuation as well. Then, sound speed can be saved as a property of this material using button "save intrinsic attenuation" on the Analysis form, see below.

This form shown below presents result of measurement for Newtonian liquid – water.

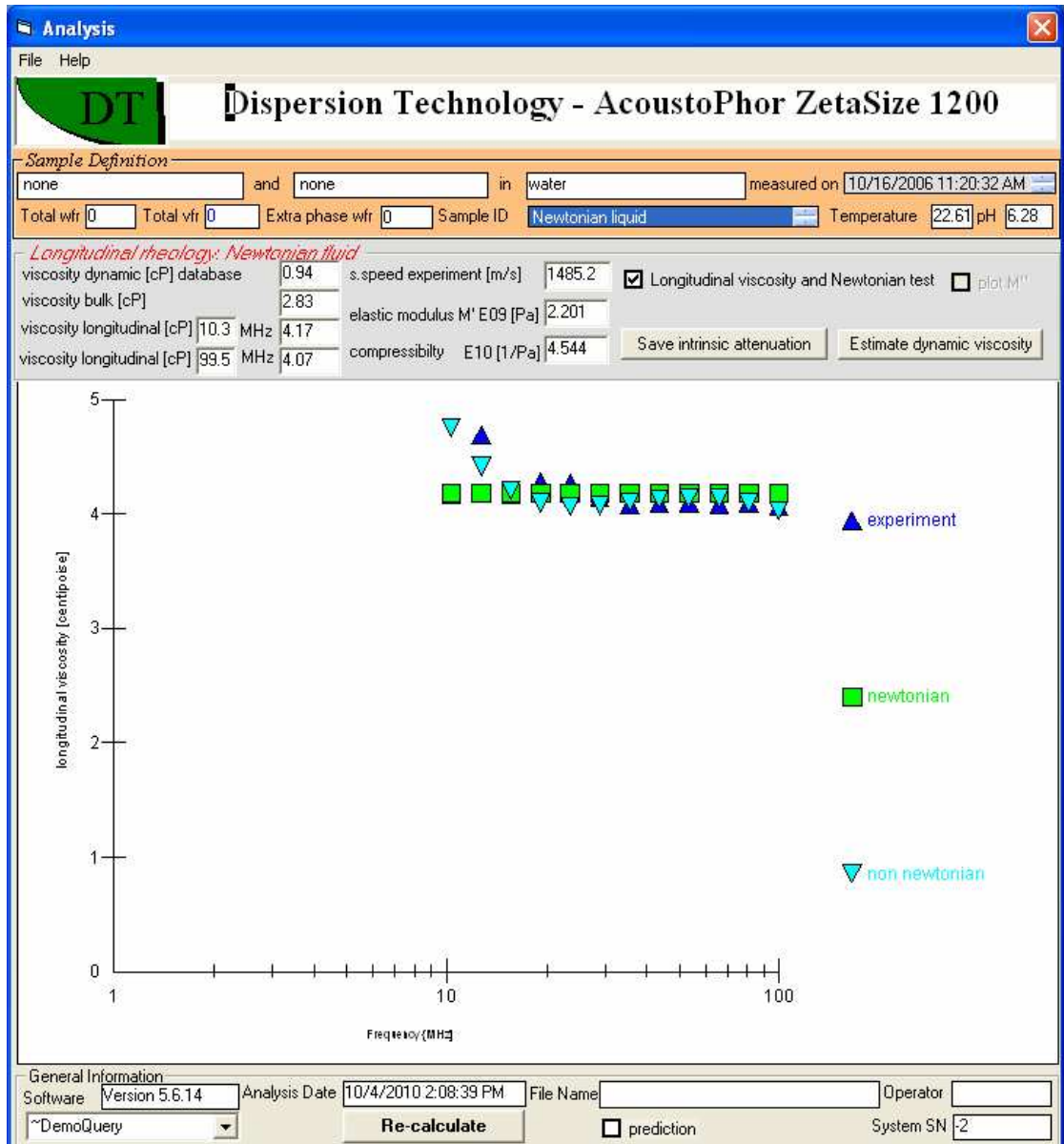


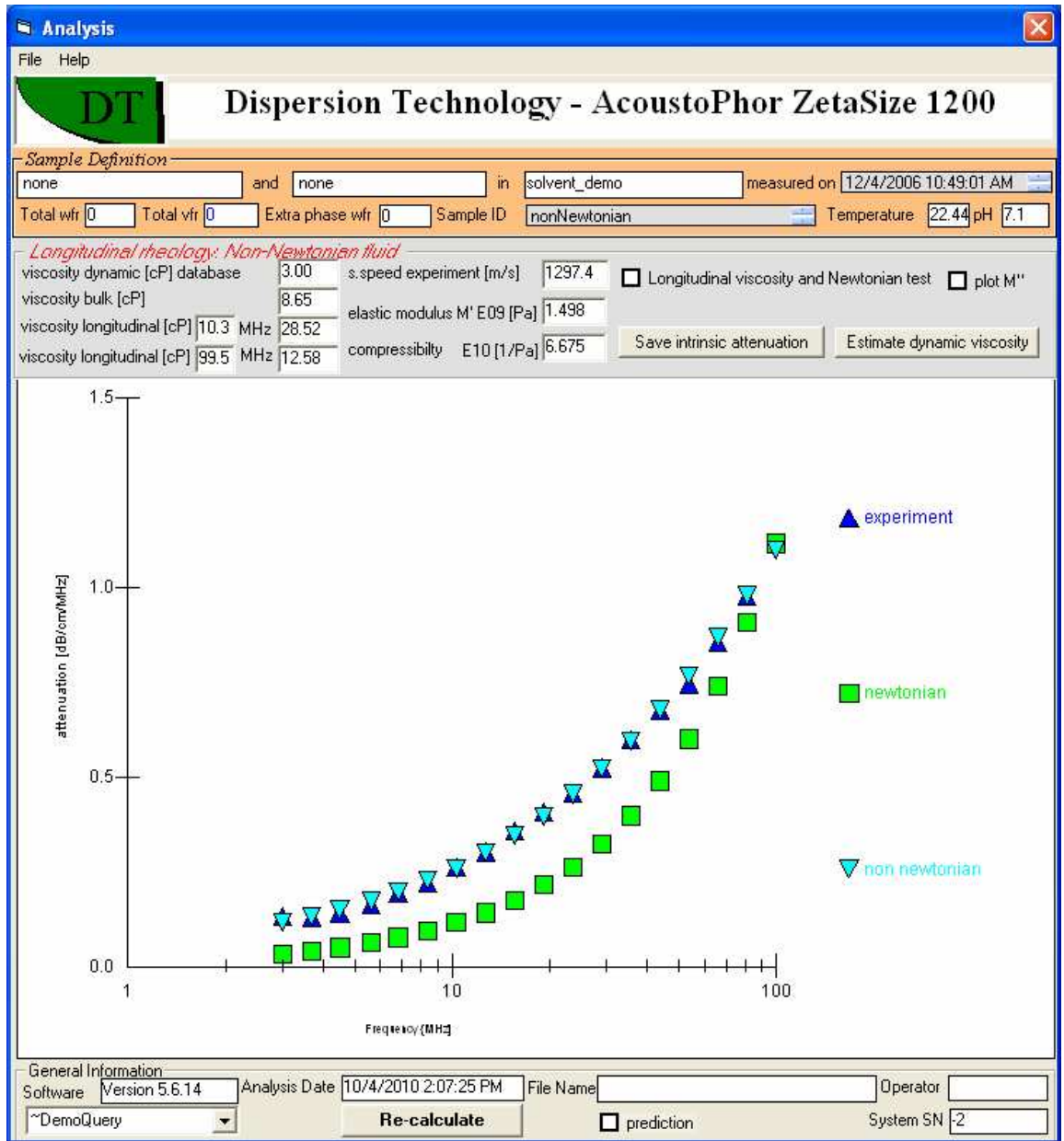
This form displays “bulk viscosity” calculated from attenuation, elastic modulus and compressibility calculated from sound speed.

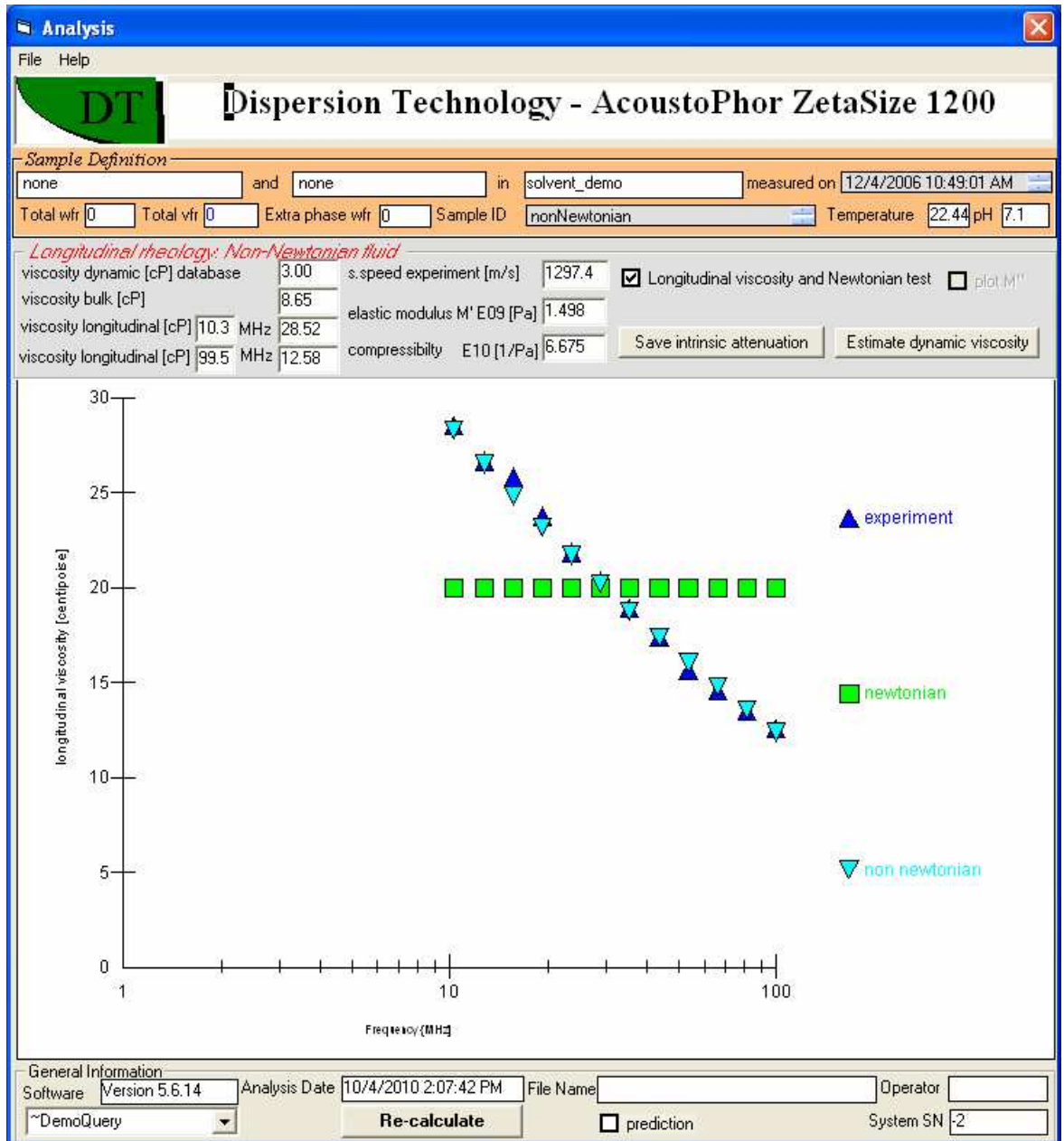
Next graph shows plot of “longitudinal viscosity” versus frequency. It is calculated from attenuation when check box “longitudinal viscosity and Newtonian test” are checked and button-re-calculates is pushed.

Liquid is Newtonian when viscosity is frequency independent.

For comparison, the following two graphs show similar results for non-Newtonian liquid.







## Rheology: shear, extensional, longitudinal

*Andrei Dukhin, Andrei Zelenev*

Rheology is the study of deformation and flow. Such definition is rather broad and implies that any process associated with deformation and flow falls within the subject of rheology. In reality, however, conventional rheology is typically restricted to the investigation of fundamental relations between force (stress) and deformation (strain) in different materials. In solid bodies, as well as in liquids, stresses can act in both tangential and normal directions. In certain cases both tangential and normal stresses are present simultaneously. However, in rheometric devices designed for measuring both stresses and deformation history, stresses are typically applied in either tangential (shear) or normal (extension/compression) direction, which leads to either **shear** or **extensional** rheology.



These two types of rheological measurements can provide significantly different information about the material. For instance, many polymeric liquids have much higher viscosities in extension than in shear.

There are many different ways to classify rheological measurements, such as by type of straining (small strain, large strain, steady strain), by deformation geometry (e.g. cone-plate, cup-bob, etc.), or by the type of flow (drag flow or pressure-driven flow). An excellent review of these classifications is given by Macosco [1]. (*see list of references at the end of this section*).

Shear rheology is by far the most abundant type of rheology, particularly because of difficulties associated with performing extensional measurements, and it dominates this scientific field to such a degree that it often becomes a synonym for rheology in general. However, many processes are governed by extensional deformations, and the behavior of materials subject to extensional stresses can not be adequately addressed with shear rheology alone. For example, in polymer melts at low extension rates the viscosity in extension is three times the viscosity in shear (Trouton's rule). In addition, there is no "extensional thinning" at higher extension rates" in contrast to the well-known shear-thinning [1].

Despite the recognized importance of extensional measurements, there are relatively few extensional studies described in the literature primarily because it is difficult to generate homogeneous extensional flows, especially with low viscosity liquids. Nevertheless, to satisfy the need for understanding the behavior of various materials in extension, there are a number of commercially available extensional rheometers. Readers can find brief overview of various types of commercially available extensional rheometers at the web site:

[http://www.museumstuff.com/learn/topics/Rheometer::sub::Types\\_Of\\_Extensional\\_Rheometer](http://www.museumstuff.com/learn/topics/Rheometer::sub::Types_Of_Extensional_Rheometer)

An important aspect of small strain rheology is oscillatory measurements. In this type of measurements the sample is deformed sinusoidally with frequency  $\omega$ . After a short transient period the stress  $\tau$  (of the amplitude  $\tau_0$ ) oscillates sinusoidally with the same frequency as strain  $\eta$  (of amplitude  $\eta_0$ ), but shifted by a phase angle  $\delta$ . The data is analyzed by decomposing the stress wave into one wave that is in phase with strain and one that is  $90^\circ$  out of phase with strain:

$$\tau = \tau' + \tau'' = \tau_0' \sin \omega t + \tau_0'' \cos \omega t \quad (1)$$

As a result of such decomposition two dynamic moduli can be defined:

$$G' = \frac{\tau_0'}{\gamma_0}; \quad G'' = \frac{\tau_0''}{\gamma_0} \quad (2)$$

The in-phase modulus  $G'$  is an elastic modulus, while the out of phase modulus  $G''$  is a viscous (or loss) modulus. A complex shear modulus can then be defined as

$$G_{shear}^* = G_{shear}' + jG_{shear}'' \quad (3)$$

Oscillatory measurements are very popular, as they allow for the detection of formation of gel networks and characterization of elasticity and viscosity of gels.

A characteristic feature of classic oscillatory rheology is low frequency (typically ranging between tens of Hz and fractions of Hz) of oscillations. Frequency of oscillations in a rheological measurement defines a characteristic flow time,  $t_f$ , or time of observation, which is compared to the material's stress relaxation time,  $t_r$ , via Deborah number,

$$De = \frac{t_r}{t_f} \quad (4)$$

Deborah number is an important parameter in rheology, as it allows one to compare the time needed to cause flow in the material to the time of a rheological experiment.

When the frequency of oscillation is low, the displacement originated at the interface has enough time to fill the entire system during the first half of cycle. All parts of the system then experience displacement in one direction. Increasing the frequency eventually alters this situation. The direction of the interface motion changes before the displacement can fill the entire system. Consequently, there are areas in the system with opposite displacements. Such high-frequency (MHz) oscillating stress creates a “wave” of displacement.

Unfortunately, most liquids do not support shear waves very well. There is a special parameter to characterize the penetration of the stress into the liquid bulk: the penetration depth,  $\delta$ . The stress amplitude decays by a factor  $\ell$  from its initial value when the distance traveled equals the  $\delta$ , where  $\ell \approx 2.71$ , the basis of the natural logarithm. The penetration depth differs from the wavelength  $\lambda$ , which depends mostly on frequency and sound speed:

$$\lambda = \frac{2\pi V}{\omega} = \frac{V}{f} \quad (5)$$

Where  $V$  is sound speed and  $f$  is frequency in *Hertz* [Hz].

The penetration depth of the shear stress,  $\delta_{shear}$ , can be estimated using following well-known equation for a viscous boundary layer (see for instance Dukhin and Goetz [2]):

$$\delta_{shear} \approx \sqrt{\frac{\eta}{\rho\omega}} \quad (6)$$

where  $\eta$  is viscosity and  $\rho$  is density. In water, for instance, the shear penetration depth is approximately 0.04 mm at a frequency of 100 Hz. and quickly decreases with frequency.

One might ask the motivation for building rheometers functioning at increasingly higher frequencies. Higher frequencies allow us to examine rheological properties on increasingly smaller length scales that are determined by wavelength. At high frequencies the characteristic flow time scales are very short, on the range of  $10^{-8}$  to  $10^{-7}$  s. In contrast to low-frequency measurements suitable for studying macroscopic changes in materials, these short time scales of high frequency measurements are suitable for probing changes on the *molecular* level, which is indeed the motivation behind developing high-frequency rheometers operating in the MHz range. Such short time scales correspond to processes like conformational rearrangement of segments in polymer chains and diffusion of strongly bound hydration water [3]. Relaxation processes with time scales longer than  $10^{-8}$  to  $10^{-7}$  s will appear “frozen” in the high frequency rheological measurements.

High frequency shear rheology has been made possible via the use of quartz crystal microbalance (QCM). The fundamental principal for QCM-based rheological measurements is the fact that mechanical properties of a vibrating quartz resonator are coupled to its electrical properties by means of the piezoelectric effect, and rheological properties of liquid in contact with the resonator can be determined by measuring electrical impedance and inductance of the vibrating crystal. The impedance and inductance are, respectively, related to  $G'$  and  $G''$  of the medium in contact with the resonator. The values of  $G'$  and  $G''$  determined by QCM are frequency-dependent and can be several orders of magnitude higher than the moduli determined by conventional low-frequency rheology. Indeed, this is not surprising in view of deficiencies associated with submicron penetration depths of shear waves existing at high frequencies. These depths can easily become comparable to the size of particles or macromolecules forming networks, which makes longer-range interaction undetectable, and puts serious constraints on the types of systems that can be analyzed. Another concern for high frequency shear measurements is related to the overemphasized role of interfacial phenomena taking place at the surface of the resonator. Formation of adsorption layers with differing visco-elastic properties makes rheological characterization of bulk material impossible.

Thus the quest for information about a material's microstructure drives frequencies higher and higher. However, the limitations associated with short penetration depths of shear waves put a restriction on the effective and universal use of QCM shear rheometers.

The answer to these limitations comes from extensional rheology. Extensional stress can penetrate into the liquid at much longer distances. The penetration depth for extensional stress,  $\delta_{ext}$ , can be estimated using Stokes's law [4] for ultrasound attenuation in a Newtonian liquid in a simple expression:

$$\delta_{ext} = \frac{\rho V^3}{\omega^2 \eta} \quad (7)$$

The extensional penetration depth is much longer than the shear penetration depth. In water, it is about 1000 meters at 1 MHz; at a frequency as high as 100 MHz, it is 10 cm. These dimensions are comparable to the macroscopic size of a typical sample chamber and by far exceed dimensions of particles and molecules making networks. Consequently, there should be no issues with the propagation of networks beyond the penetration of extensional stresses.

Waves of extensional stresses are usually called longitudinal waves, which are easy to generate and measure. There is a basic difference between the stress at the steady-state condition and the stress within an oscillating longitudinal wave. Traditional extensional steady-state stress generates deformation and flow in isochoric conditions, which means that liquid retains its volume. In contrast, extensional stress within a longitudinal wave causes variation in the liquid volume. Liquid behaves as a compressible material in the longitudinal wave. This drastic difference between steady-state and oscillating modes justifies the introduction of a special term for the oscillating extensional rheology: longitudinal rheology.

Longitudinal high frequency rheology characterizes the visco-elastic properties of liquids as well as the shear rheology. In a manner similar to shear rheology, longitudinal visco-elastic properties are characterized by a complex longitudinal modulus,  $G^*_{long}$ , with components similar to  $G^*_{shear}$ :

$$G^*_{long} = G'_{long} + jG''_{long} \quad (8)$$

Despite a general similarity with conventional  $G'$  and  $G''$ , the values for longitudinal moduli may be substantially different from the former, as was the case with high frequency shear moduli determined by QCM. The similarity between longitudinal and shear rheology appears in the general relationship between complex modulus and penetration depth

$$G'_{long} = \rho \omega^2 V^2 \delta_{long}^{shear} \frac{\omega^2 \delta_{long}^{shear} - V^2}{[\omega^2 \delta_{long}^{shear} + V^2]^2} \quad (9)$$

$$G''_{long} = 2\rho \omega^3 V^2 \delta_{long}^{shear} \frac{V}{[\omega^2 \delta_{long}^{shear} + V^2]^2} \quad (10)$$

It is possible to introduce shear and longitudinal viscosities for non-Newtonian liquids using the same expression for both parameters:

$$G^* = G' + jG'' = G' + i\omega \eta_{long}^{shear}(\omega) \quad (11)$$

Equations 9 and 10 for shear rheology were derived in a paper by Williams and Williams [5-7] who, in turn, make reference to an earlier book by Whorlow [8]. The derivation of identical equations for the longitudinal rheology case was taken from a review by Litovitz and Davis [9]. They, in turn, make reference to earlier works by Miexner [10,11], who apparently derived the general thermodynamic theory of stress-strain relationship in liquids.

According to these theories, the main difference between conventional shear rheology and longitudinal rheology is in the specifics of the penetration depth. This parameter is not easily measurable but it is linked to another parameter that is easy to measure—attenuation coefficient,  $\alpha$ . The attenuation coefficient determines how quickly the stress amplitude decays with distance due to various dissipative effects. Attenuation coefficient and penetration depth are simply reciprocal parameters:  $\delta = \frac{1}{\alpha}$ .

The longitudinal attenuation coefficient can be easily measured with modern acoustic spectrometers ([www.dispersion.com](http://www.dispersion.com)). These devices make longitudinal rheology as readily available as traditional shear rheology, although at much higher frequencies (on MHz scale). Consequently longitudinal rheology, rather than high frequency shear rheology, is a valuable source of information about the microstructure of materials.

### **Conclusions:**

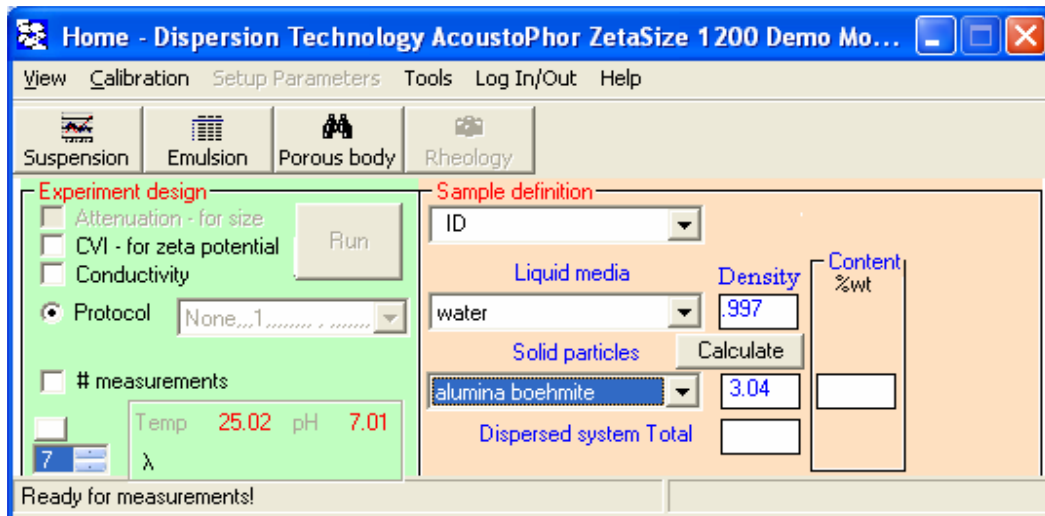
Conventional shear rheology, conventional extensional rheology, and their high frequency analogues (longitudinal rheology and high frequency shear rheology) are complementary rheological methods that are applicable at distinctively different frequency ranges, generate different types of stresses and cover completely different relaxation time scales that may differ by orders of magnitude. Conventional shear rheology works at the low-frequency range below 1 KHz and is most suitable for studying relaxation processes occurring on the time scales between  $10^{-3}$  and  $10^4$ . Longitudinal and high frequency shear rheology become viable tools at the high frequency range (on MHz scale) and are therefore methods of choice for probing processes with very short relaxation times, such as conformations of segments within a polymer backbone. Longitudinal rheology opens up the possibility of studying much shorter relaxation times than possible by shear rheology and provides deeper insight into the material microstructure. Also, longitudinal rheology is non-destructive—the structural network shakes but does not break. Shear rheology analyses breaks inter-particle bonds. This difference enforces the complementary nature of these two methods.

### **References**

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11. Meixner, J. *Kolloid-Zt.*, 134, 47 (1954)

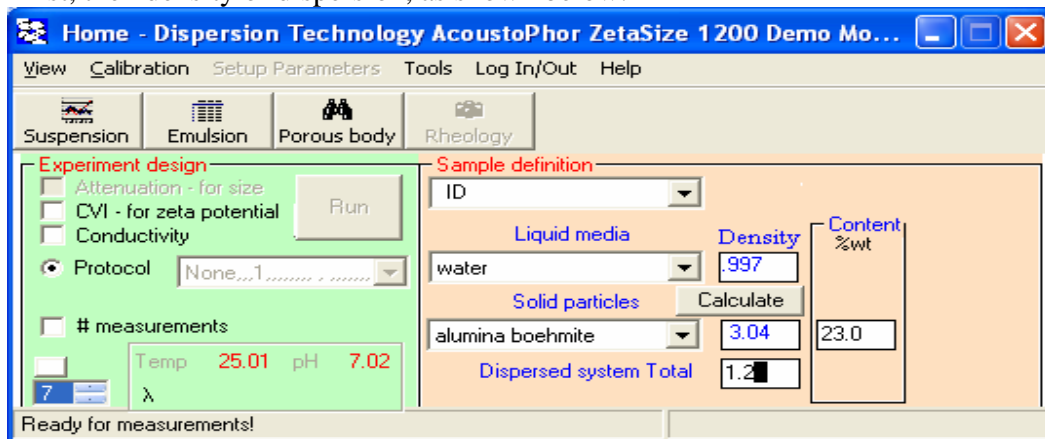
## Sample Content Wizard and Pycnometer

The time may come when you need to measure a sample for which you do not know weight fraction or the density of the disperse phase material. You can use a built-in software pycnometer function. There are several modes of this software. The simplest one is based on 4 boxes on the Home page Sample definition frame. Three boxes are for densities of the liquid, particles and dispersion. The fourth box is weight fraction of particles. These 4 parameters are constrained by matter conservation law. If one knows value of any 3 of the boxes, then the value for the 4<sup>th</sup> box can be calculated.

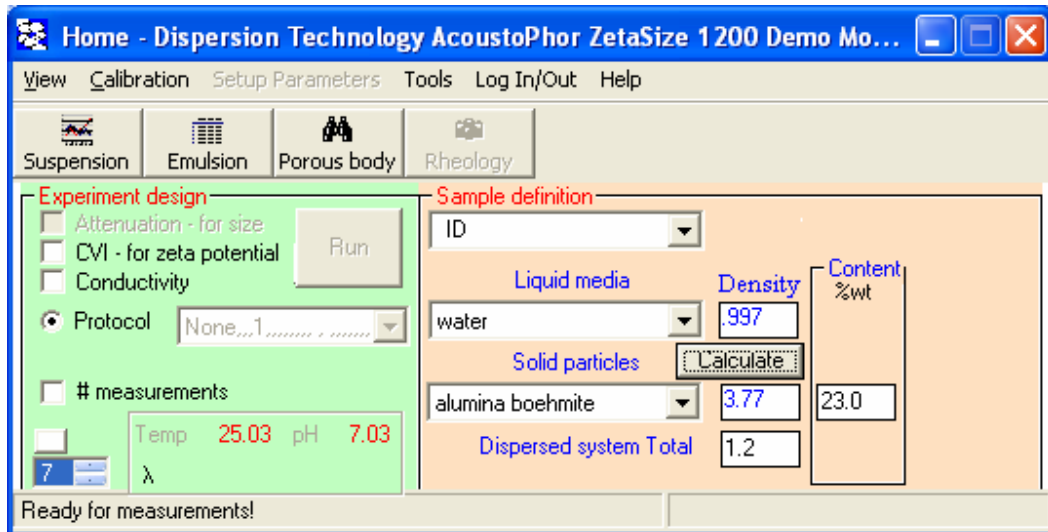


### Calculating density of particle

If one want to calculate density of the particles, then the density of dispersion, the density of your media and the weight fraction of the dispersion must be known. You should specify weight fraction first, then density of dispersion, as shown below.

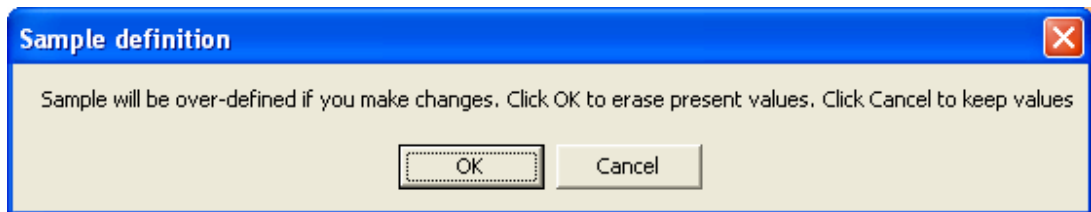


Click “Calculate”. You will see new density of particle in its box, see below.

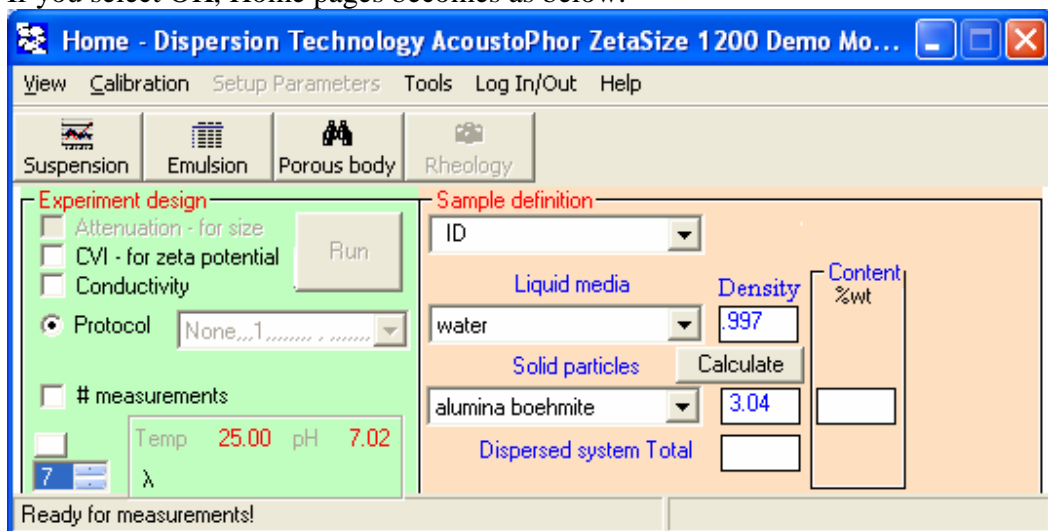


You can save this value by opening Define Material form from Home page Tools. Goto Modify Material there, find you material and type the new density value.

If you do not want this new value and want to recover the old one, just click on weight fraction box. The following message appears. It always would appear warning you about erasing data from all boxes of the Content Wizard if they becomes over-defined.

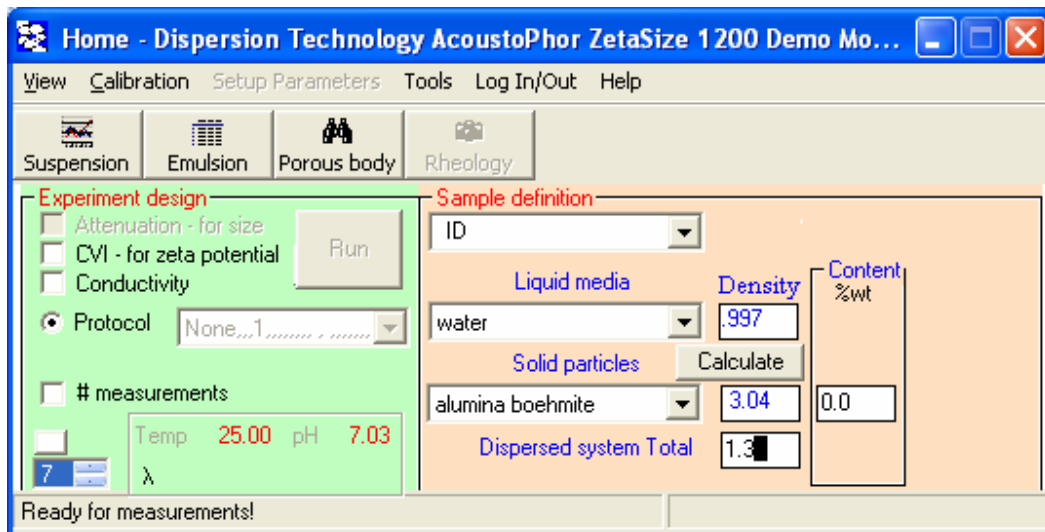


If you select OK, Home pages becomes as below.

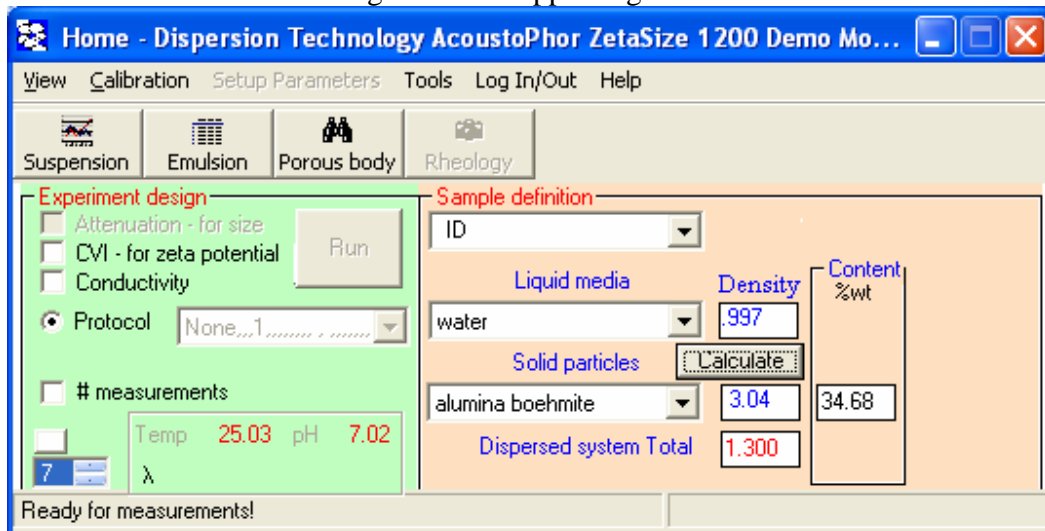


### Calculating weight fraction from the density of dispersion

For this purpose you should place measured density of dispersion in the box as shown below and click Calculate.



You can see value of the weight fraction appearing in the box on the frame Content.



### Calculating weight fraction from the weights and volumes

In some cases you might know weight or/and volumes of phases that compose particular dispersion. You can calculate weight fraction using these parameters.

Goto Home page, Tools click Content Wizard.

Home page would become as shown below.

Now you can fill any two boxes in the frame Content, click Calculate and software would fill all others.

Home - Dispersion Technology AcoustoPhor ZetaSize 1200 Demo Mode User: usr

View Calibration Setup Parameters Tools Log In/Out Help

Suspension Emulsion Porous body Rheology

**Experiment design**

- Attenuation - for size
- CVI - for zeta potential
- Conductivity
- Protocol None,,1
- # measurements
- Temp 25.03 pH 7.01
- $\lambda$

Run

**Sample definition**

ID Preparation temp 25

Liquid media	Density	Content				SSpeed
		%wt	%vl	gm	ml	
water	.997	65.32	85.17			1497.
alumina boehmite	3.04	34.68	14.83			8000.
Dispersed system Total		1.300				1419.1

Calculate

Ready for measurements!



## Glossary of attenuation spectra for different particle size distributions and material properties.

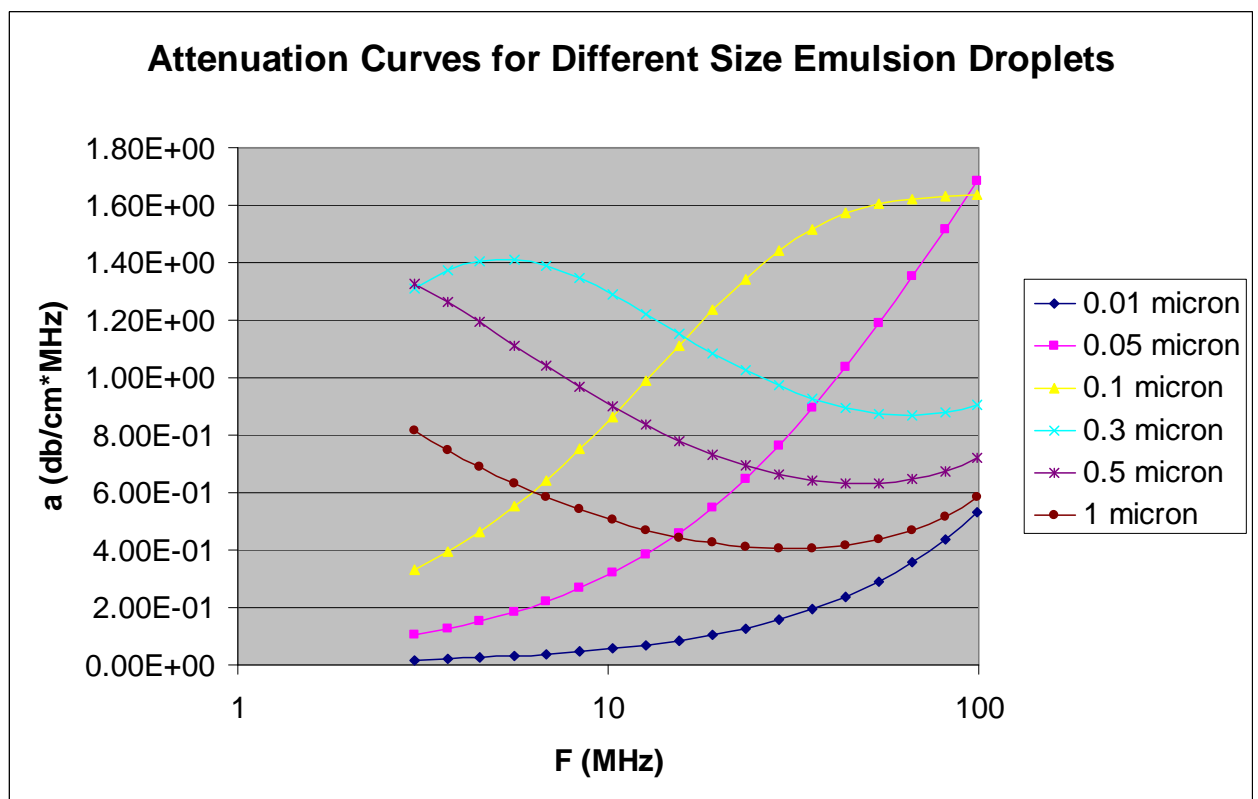
Acoustic particle sizing is based on calculation of particle size distribution from the attenuation frequency spectra, similarly as it is done for dynamic light scattering using autocorrelation function. Attenuation spectrum is an analog of autocorrelation function.

In this section we present typical attenuation spectra. It would be possible for user to compare these curves with whatever measured and made some preliminary judgment about particle size. We present these curves separately for emulsions and solid particles dispersions.

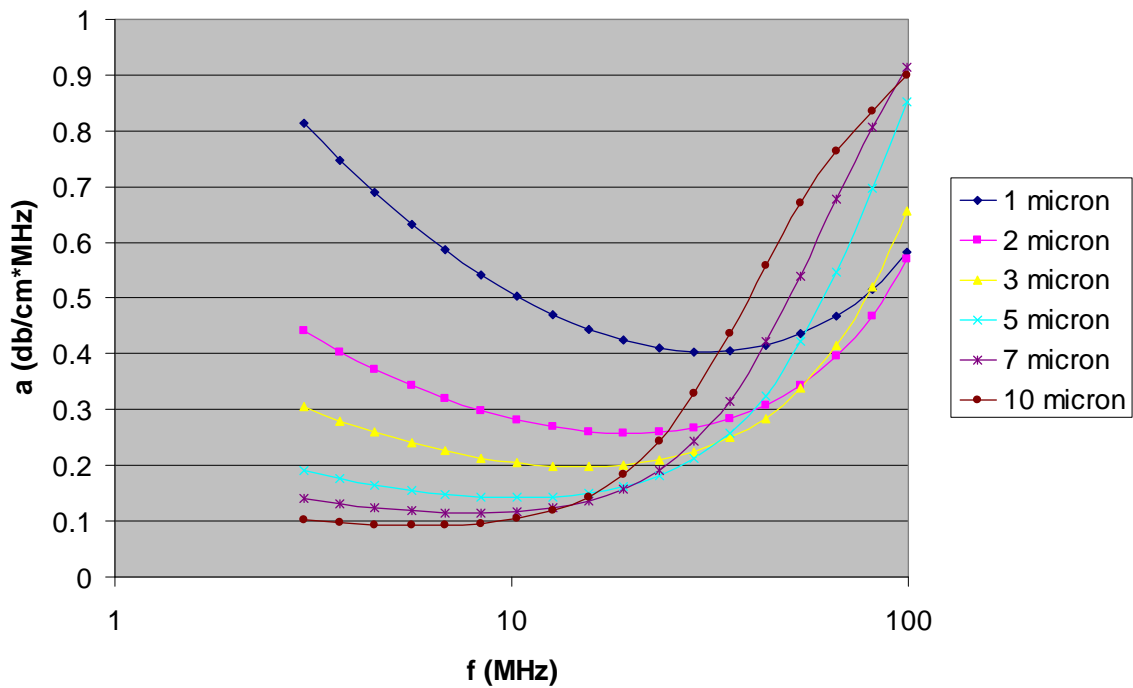
### *Emulsions.*

All emulsions attenuation spectra calculated here were made assuming them being oil-in-water with 10% of oil weight, density of water 1 g/cu.cm, density of oil 0.95 g/cu.cm, intrinsic attenuation of oil being Newtonian and 2 dB/cm/MHz at 100 MHz, st.dev of the particle size distribution is 0.12.

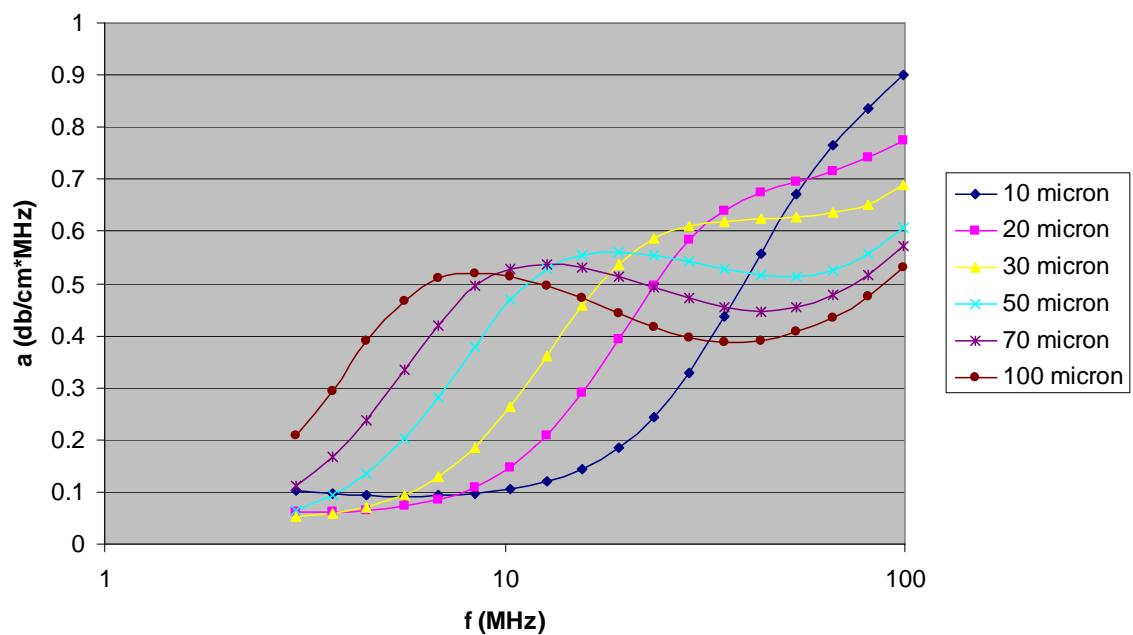
For first 3 graphs illustrating effect of the droplet size “scattering coefficient” is 1. Heat capacity was 2 J/g/C, heat conductance is 1.5 mW/cm/C, thermal expansion is 9 [1/Ko]\*10E-4.



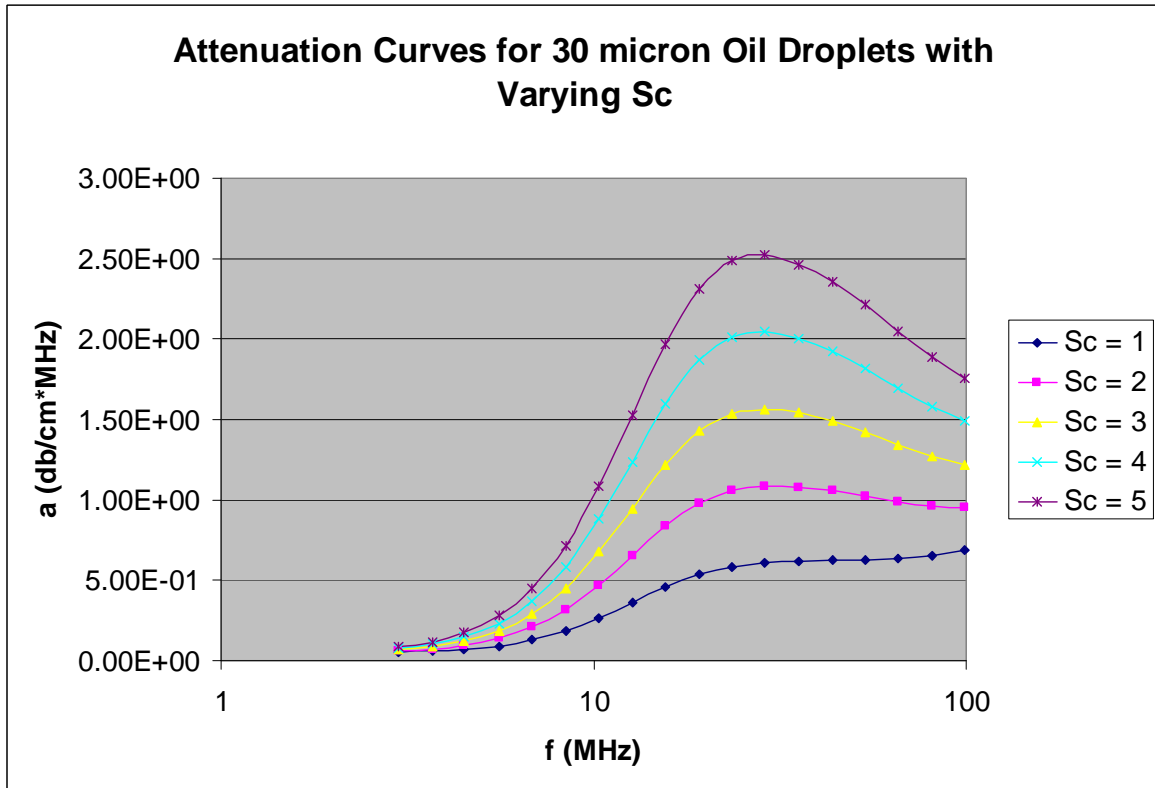
**Attenuation Curves for Large Emulsion Droplets**



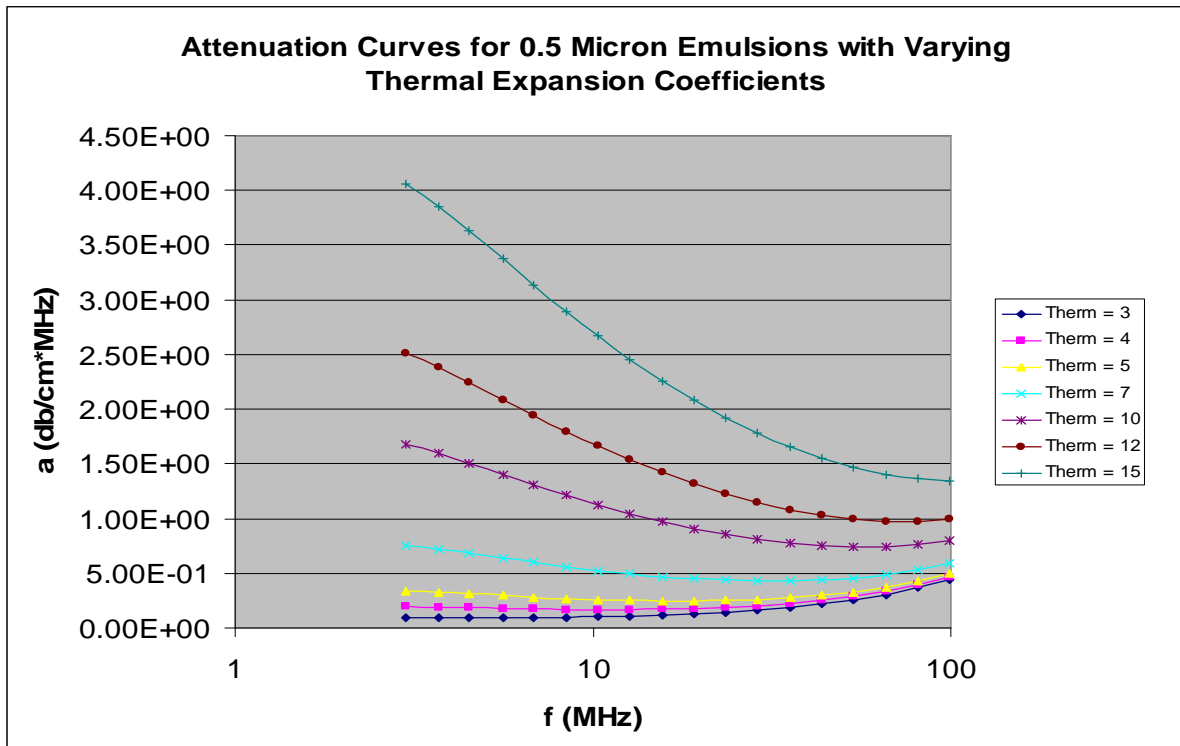
**Attenuation Curve for Very Large Emulsion Droplets**



The following attenuation curves illustrate role of the “scattering coefficient” for large droplets. Droplet size is 30 microns for all curves.



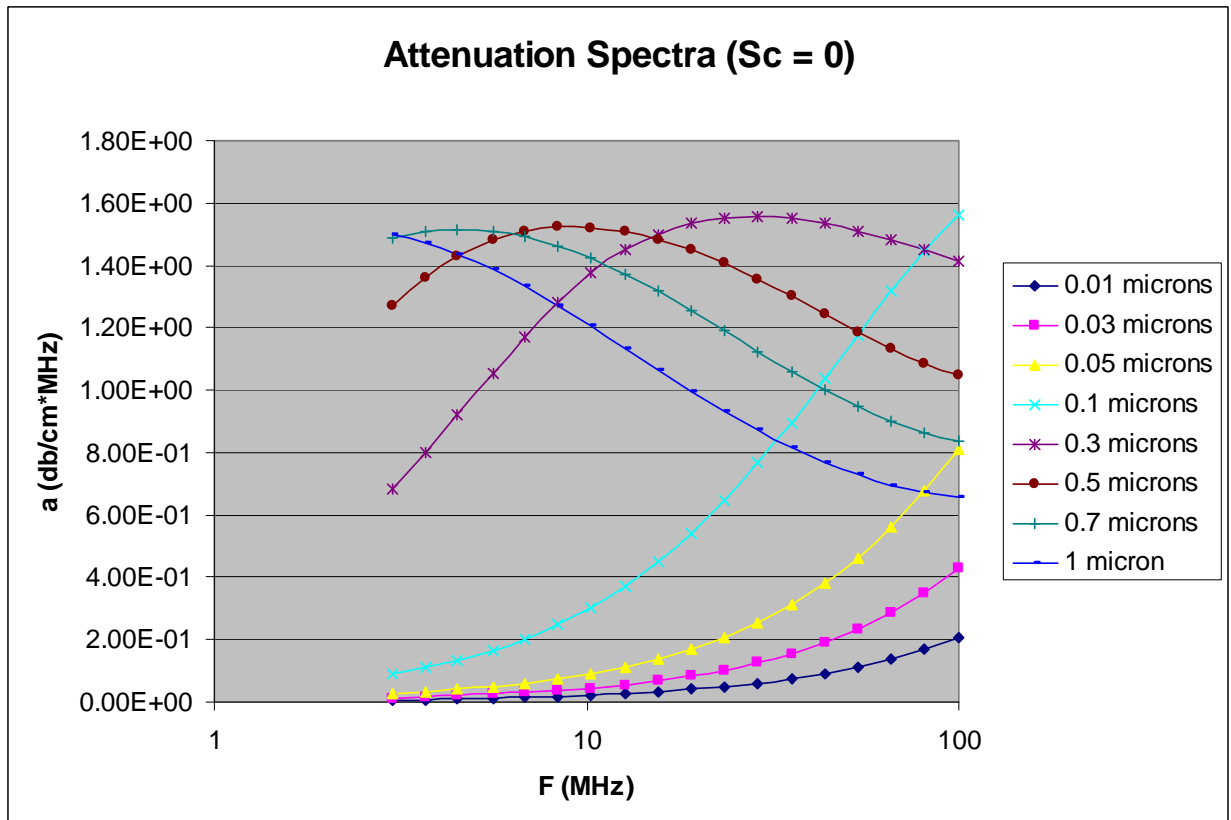
The following attenuation curves illustrate role of the “thermal expansion coefficient. Droplet size is 0.5 microns for all curves.

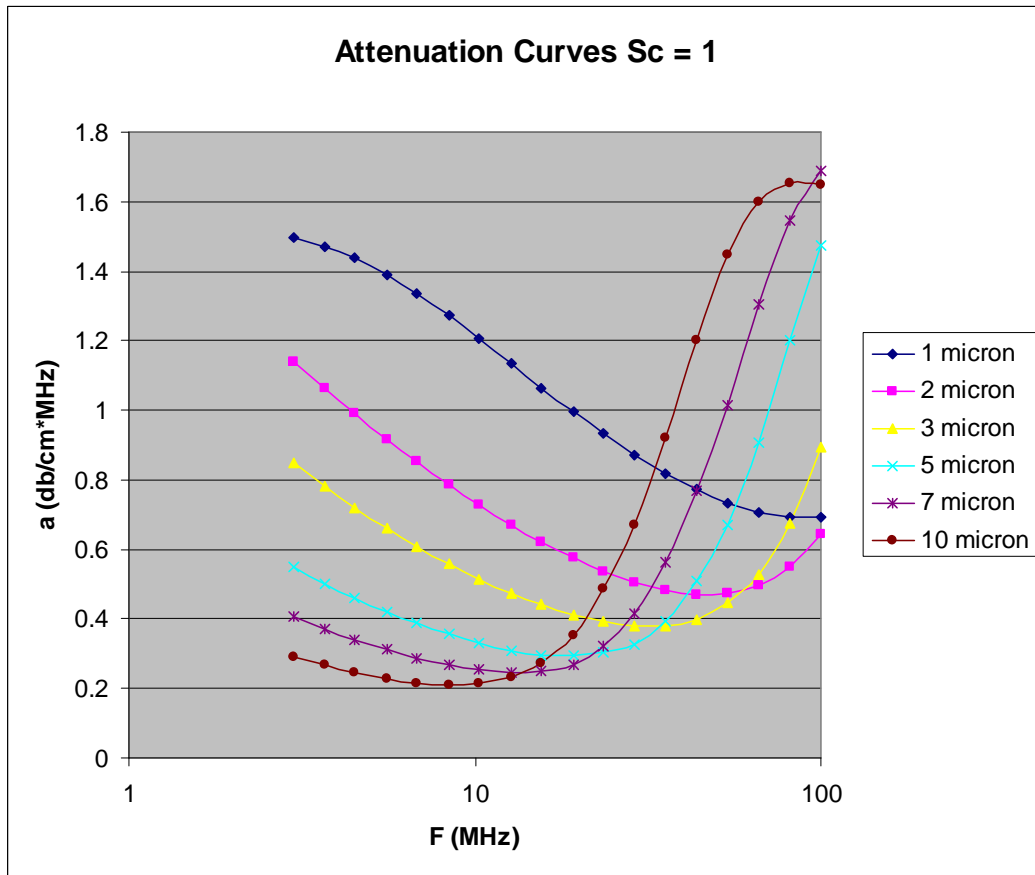


### *Dispersions of solid particles.*

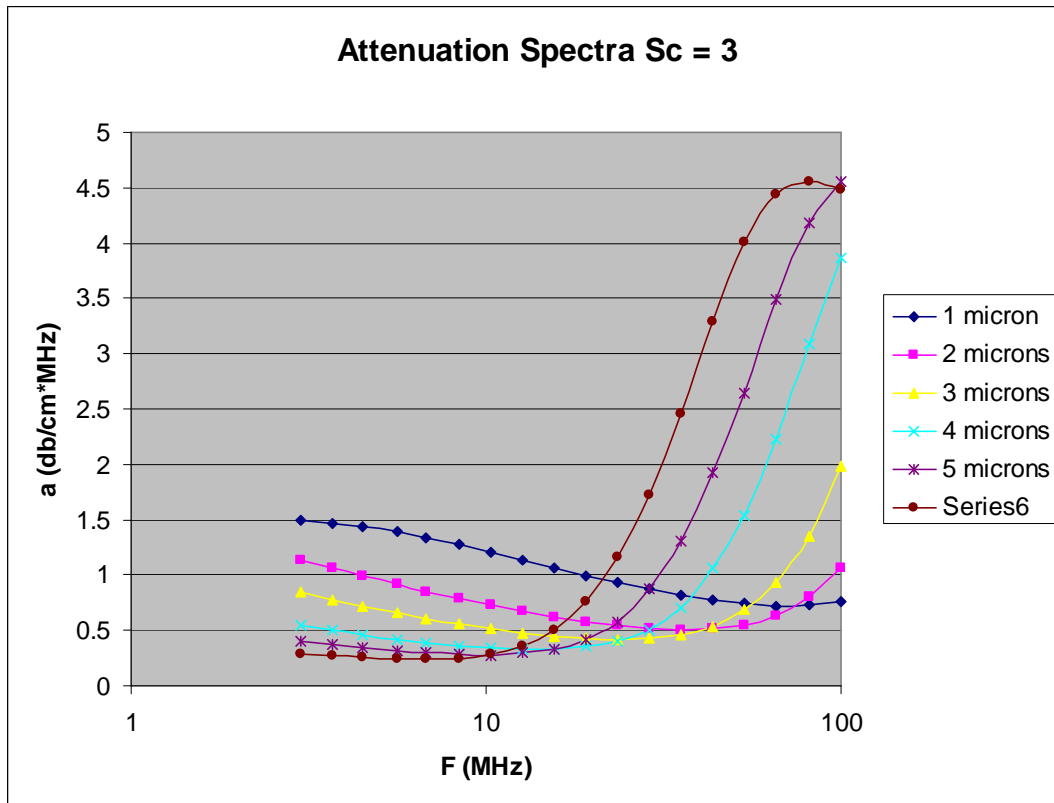
All attenuation curves shown below were calculated for dispersions of solid particles with weight fraction 10%, density of particles 2.5 g/cu.cm, density of liquid 1 g/cu.cm, viscosity 1 cP, st.dev of particle size distributions is 0.1.

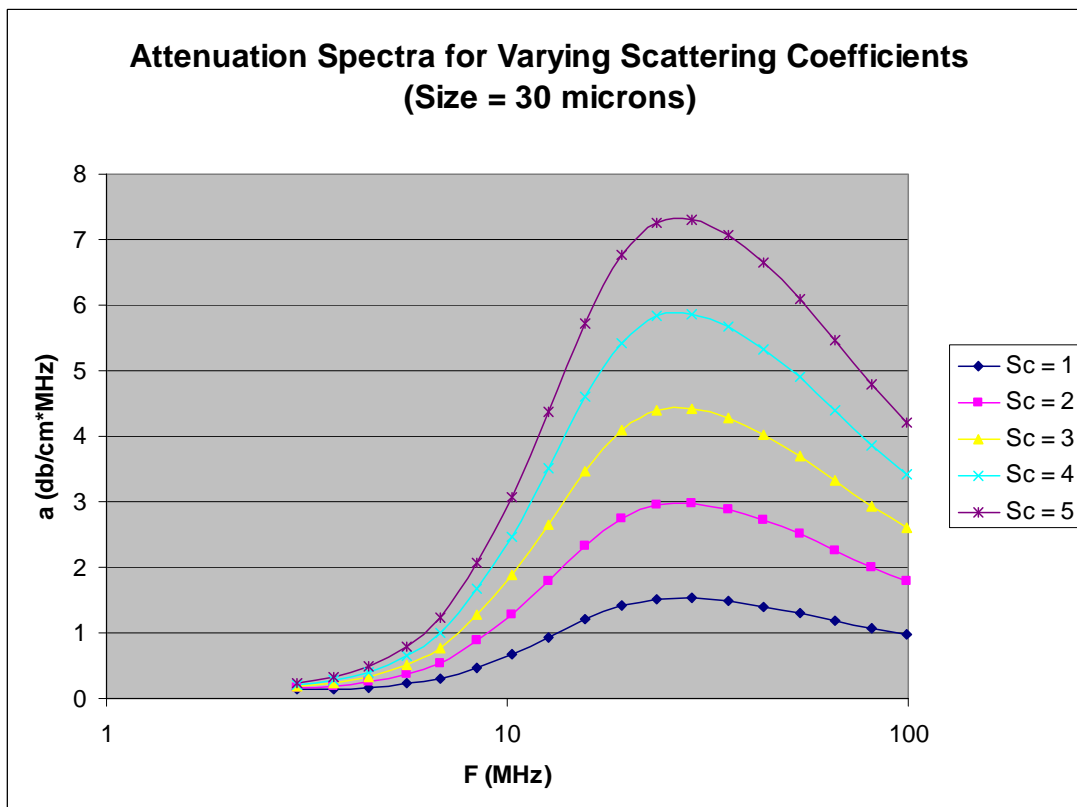
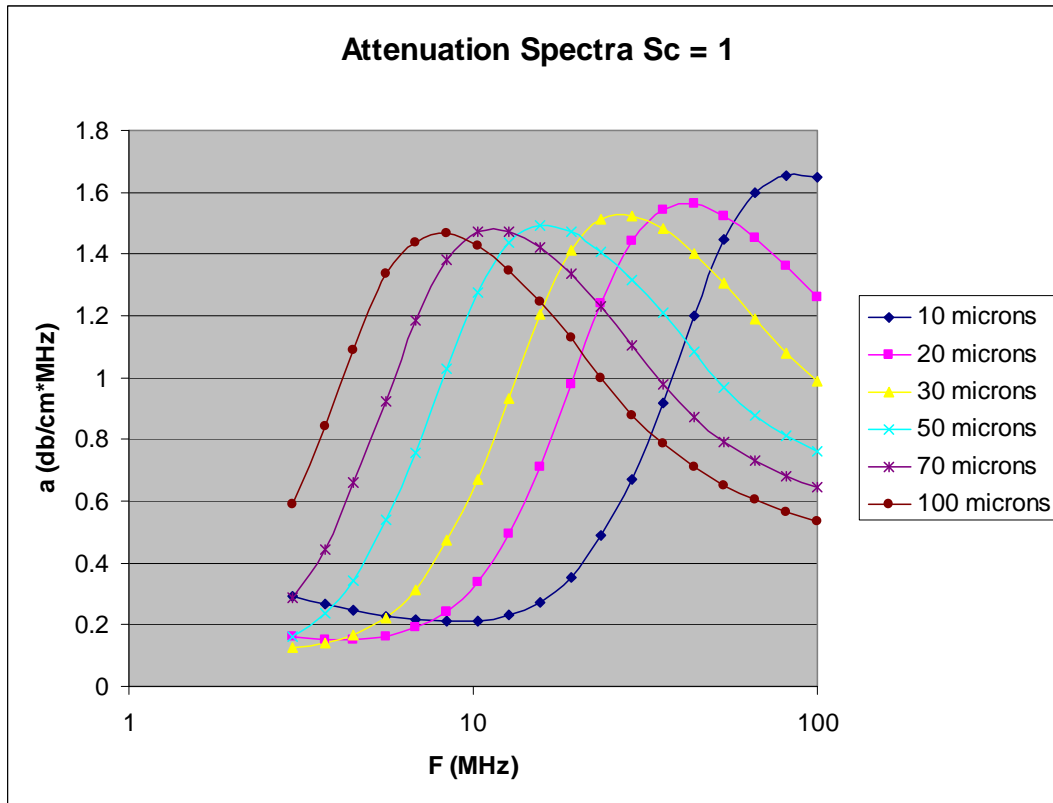
Parameter Sc is “scattering coefficient” that corrects scattering loss for non-elastic scattering. If Sc=0, then curve is combination of intrinsic and viscous attenuations only.





P





## Analysis form for calculating particle size, zeta potential, rheological properties.

There are several purposes for Analysis form.

1. Analysis form performs calculation of particle size, zeta potential, rheological parameters from the measured data.
2. It displays results of calculation and serves as the main Report.
3. It allows re-calculation of old results and re-building old Reports
4. It has tools for calculating key input parameters – thermal expansion coefficient, scattering coefficient, intrinsic attenuation and saving them to Material database, see the book, section 4.6, page 174.
5. It can function in Prediction mode calculating theoretical analogs of measured parameters for user specified input parameters.

This form appears right after the raw data has been collected.

If the Analysis form is not currently displayed, click on the menu item **File Analysis** on the Home page.

The form's appearance depends on which raw data are measured for a particular record.

There are four main display setups, namely:

Setup 1) Acoustic attenuation and colloid vibration current are both measured.

Setup 2) Only acoustic attenuation is measured;

Setup 3) Only colloid vibration current is measured;

Setup 4) Only acoustic attenuation is measured, but no disperse phase is specified on the Home form

There is also possibility to see this form in Report setup or in Research mode. This can be achieved by checking or un-checking menu item View, Report.

The following Figures illustrate all these display Setups, as they would normally appear automatically after a measurement is finished and the analysis of the raw data is complete. Each setup display Setup is largely fixed by the properties that are measured, but some modifications are possible by making selections from the View menu as will be described.

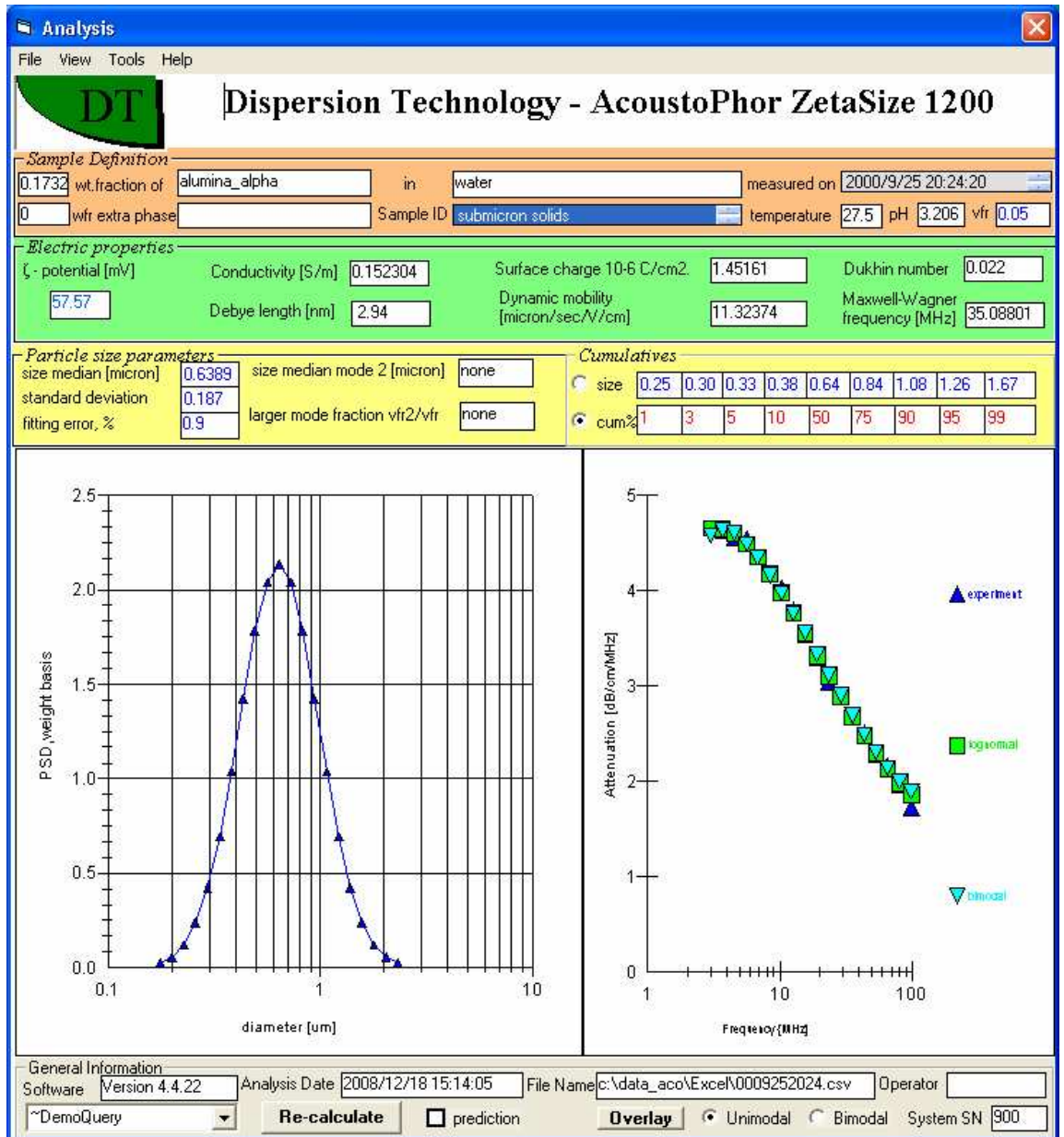


Figure 1. shows Setup 1 for which attenuation, CVI, and conductivity are all measured. Whether the unimodal or bimodal PSD is shown depends on which provides the most realistic picture of the sample based on the respective fitting errors. Report mode.



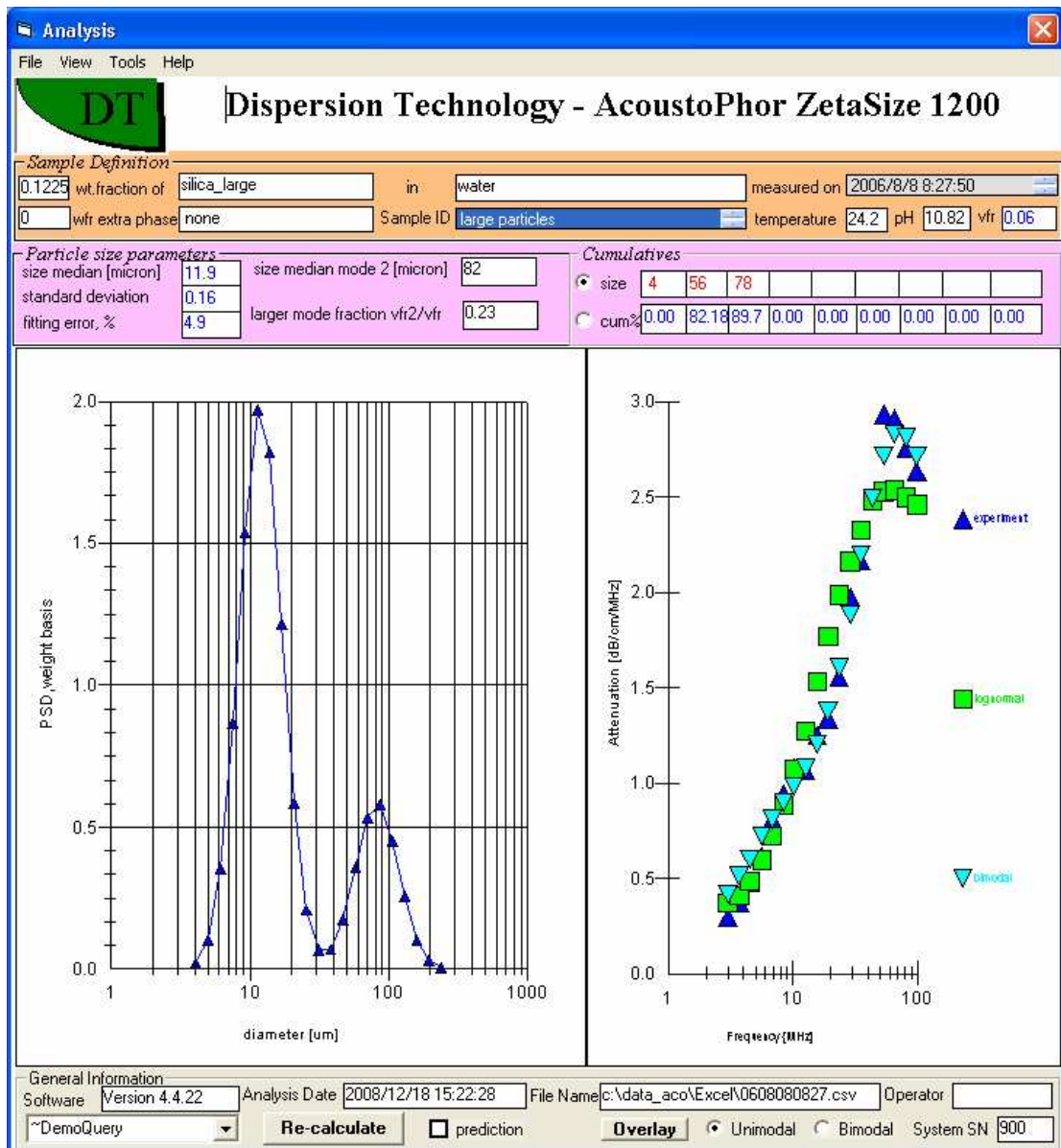


Figure 2 shows Setup 2 for which only attenuation measured. In this case the fitting errors suggested that the bimodal solution provided a better representation. Report mode.

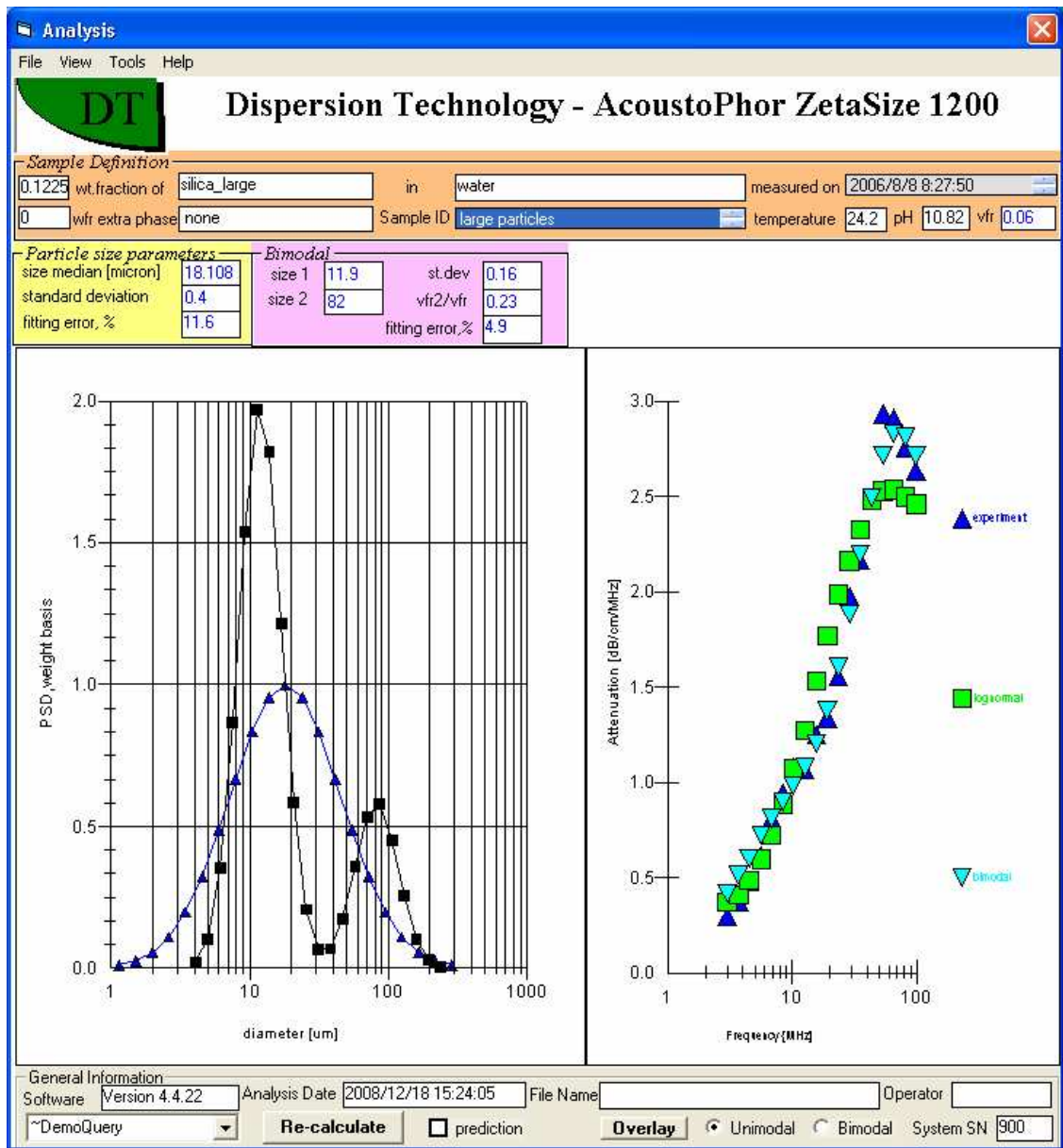


Figure 3 shows Setup 2 again, but in this case the user has clicked on the menu item **View both unimodal and bimodal** to see both. Research mode.

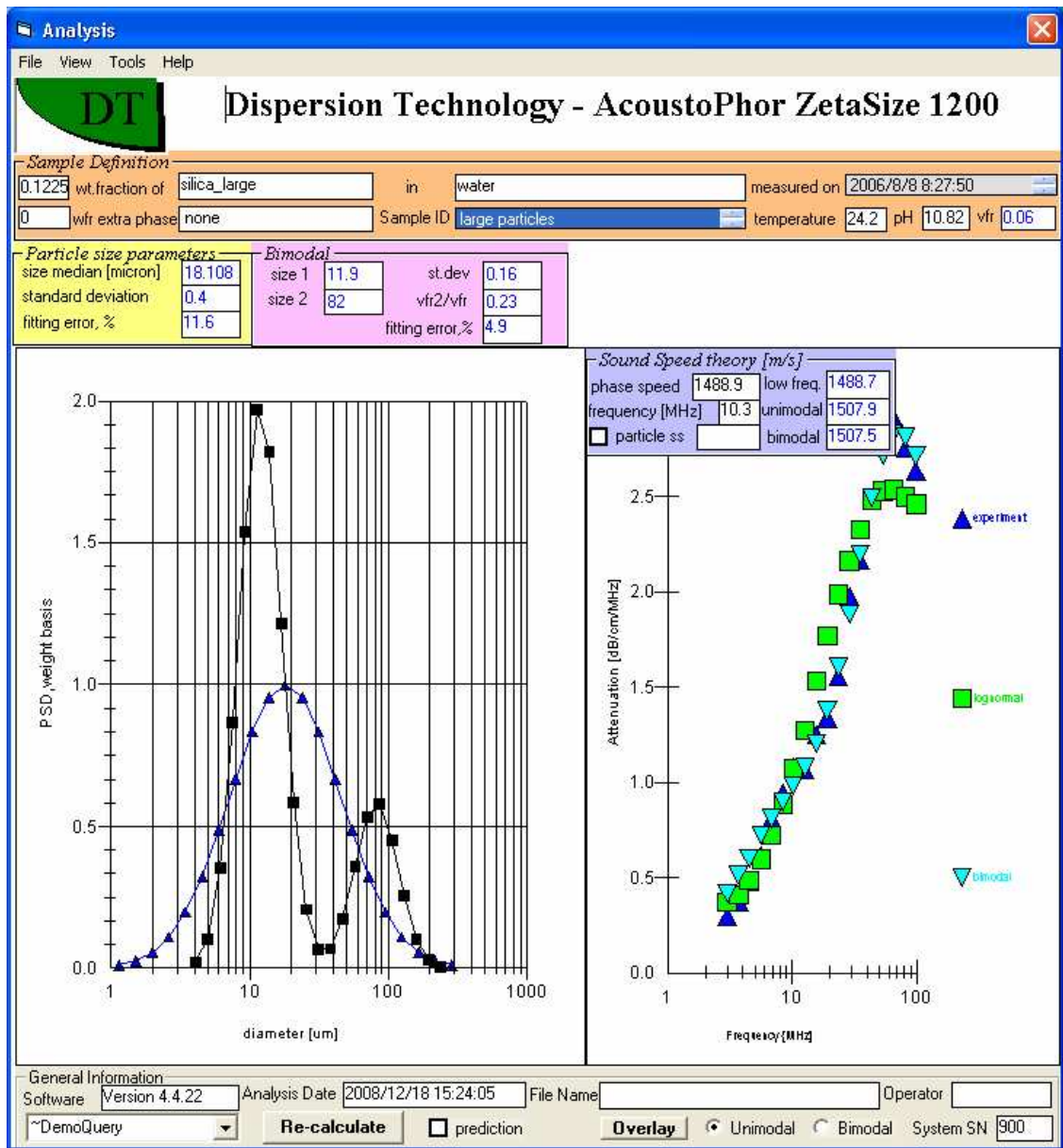


Figure 4 shows Setup 2 yet again, but in this case the user has clicked on the menu item **View Sound speed** to display sound speed information. Research mode.

The Analysis software can utilize altogether three separate theories of increasing sophistication for calculating zeta potential, namely the simple classic theory of Smoluchwsky, an Advanced CVI aqueous theory, and a theory for non-aqueous or nano-colloids. Figure 5 shows Setup 3, for which we measure only CVI, and by default calculate zp using this classic theory.

The more sophisticated theories yield more accurate zp values only when the conductivity of the sample is known, which in turn allows us to calculate the Debye length and Dukhin number  $Du$ ,

both important characteristic of surface conductivity. The Advanced CVI aqueous theory works when  $ka > 3$ . For lower  $ka$  values, due to the small size or low ionic strength, the third theory becomes employed. It takes into account the overlap of thick double layers. The best output for the third theory is surface charge. This parameter can be calculated without conductivity, assuming nevertheless a  $ka < 3$ . The calculation of a zeta potential would require conductivity data, which might be hard to obtain in non-polar liquids. In addition, this calculation is very non-linear. The software restricts the highest value of zeta potential for this third theory at 200 mV.

You can see the results for the two more sophisticated theories by clicking on the menu item **View Advanced CVI**. Figure 6 shows Setup 3 for case where user has indeed measured conductivity and selected Advanced CVI.

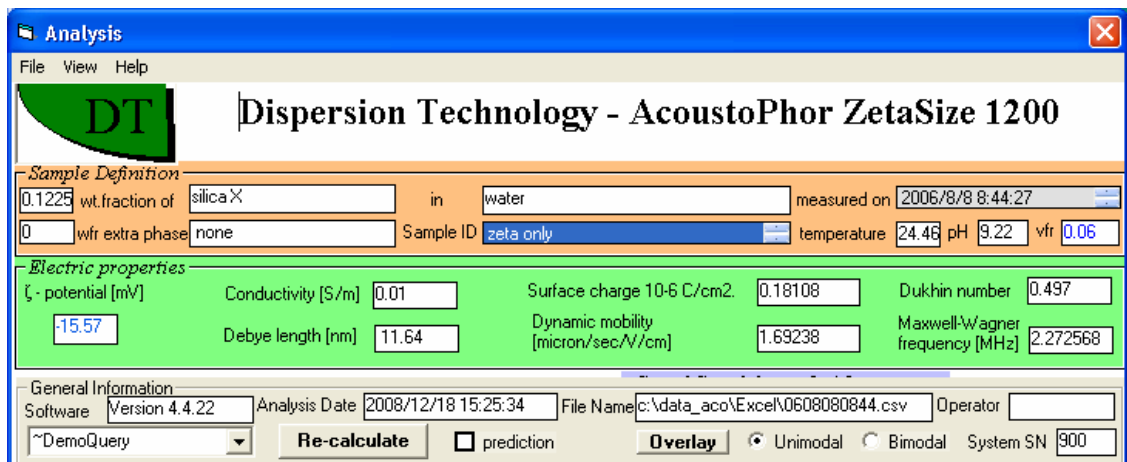


Figure 5.

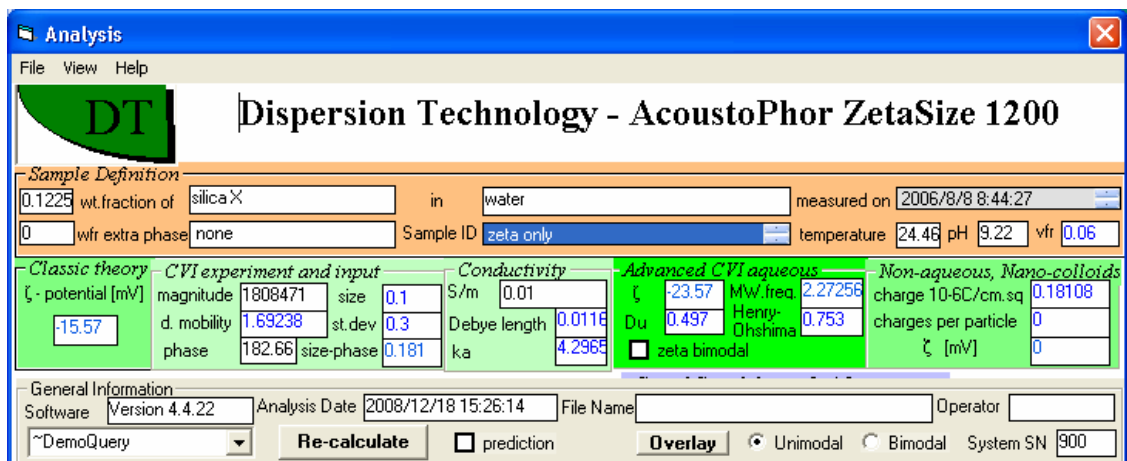


Figure 6

The Advanced CVI aqueous frame has a check box labeled “zeta bimodal”. If you check this box a Bimodal frame appears as shown in Figure 7 below. This would allow you to recalculate zeta for given CVI using a bimodal PSD that you specify in this Bimodal frame

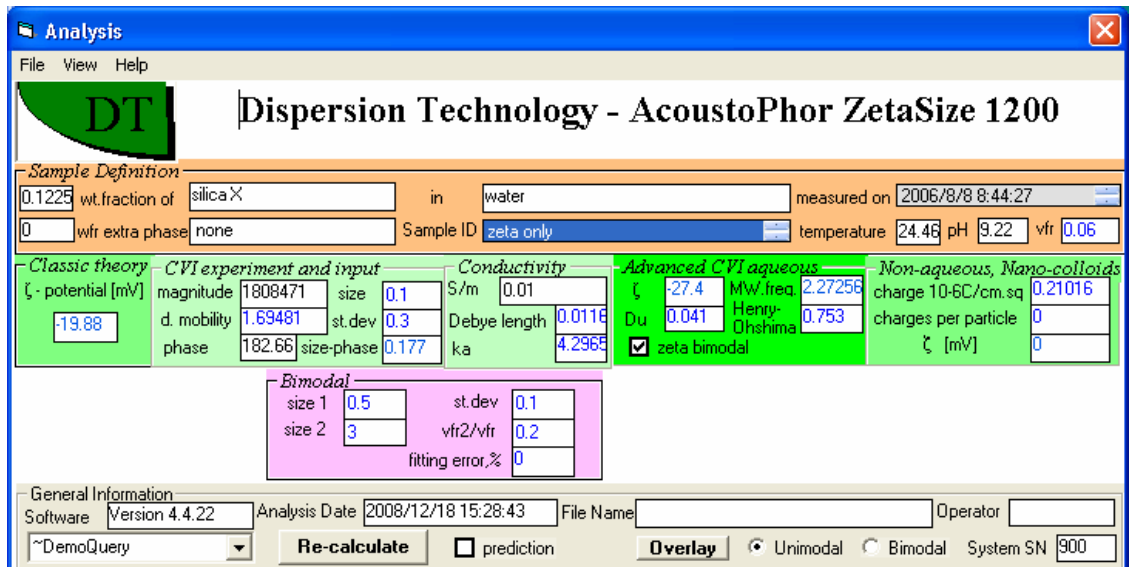


Figure 7

Figure 8 shows Setup 4 for case where only attenuation is measured and there is no disperse phase. This Setup is used for intrinsic attenuation measurement.

Once attenuation data is displayed in Setup 4 it can be converted to a rheological format by checking the box **rheology output** and then pressing **RUN** ( If necessary click on Recalculate to make RUN visible) This will convert attenuation and sound speed into visco-elastic moduli as shown in Figure 9

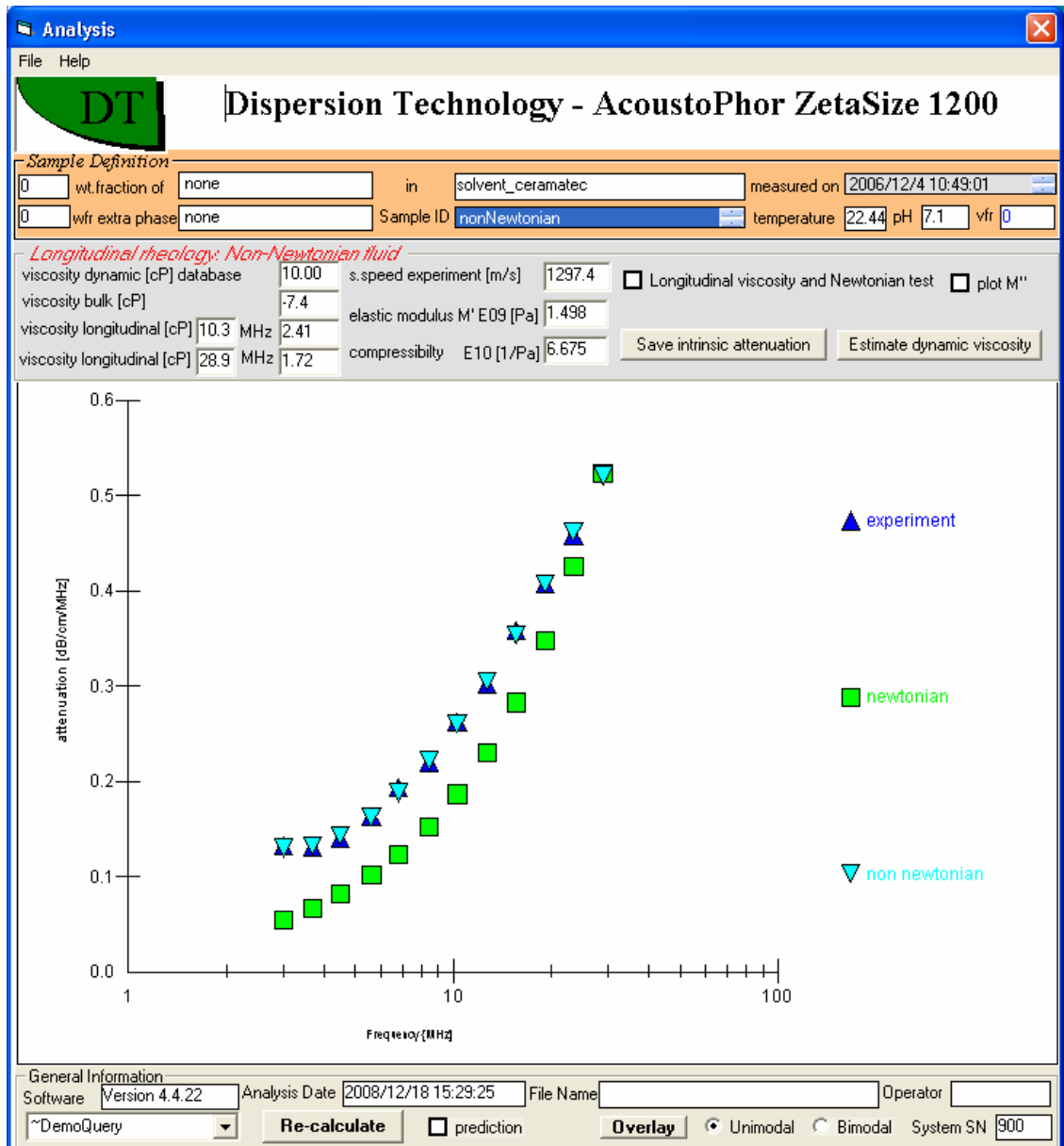


Figure 8

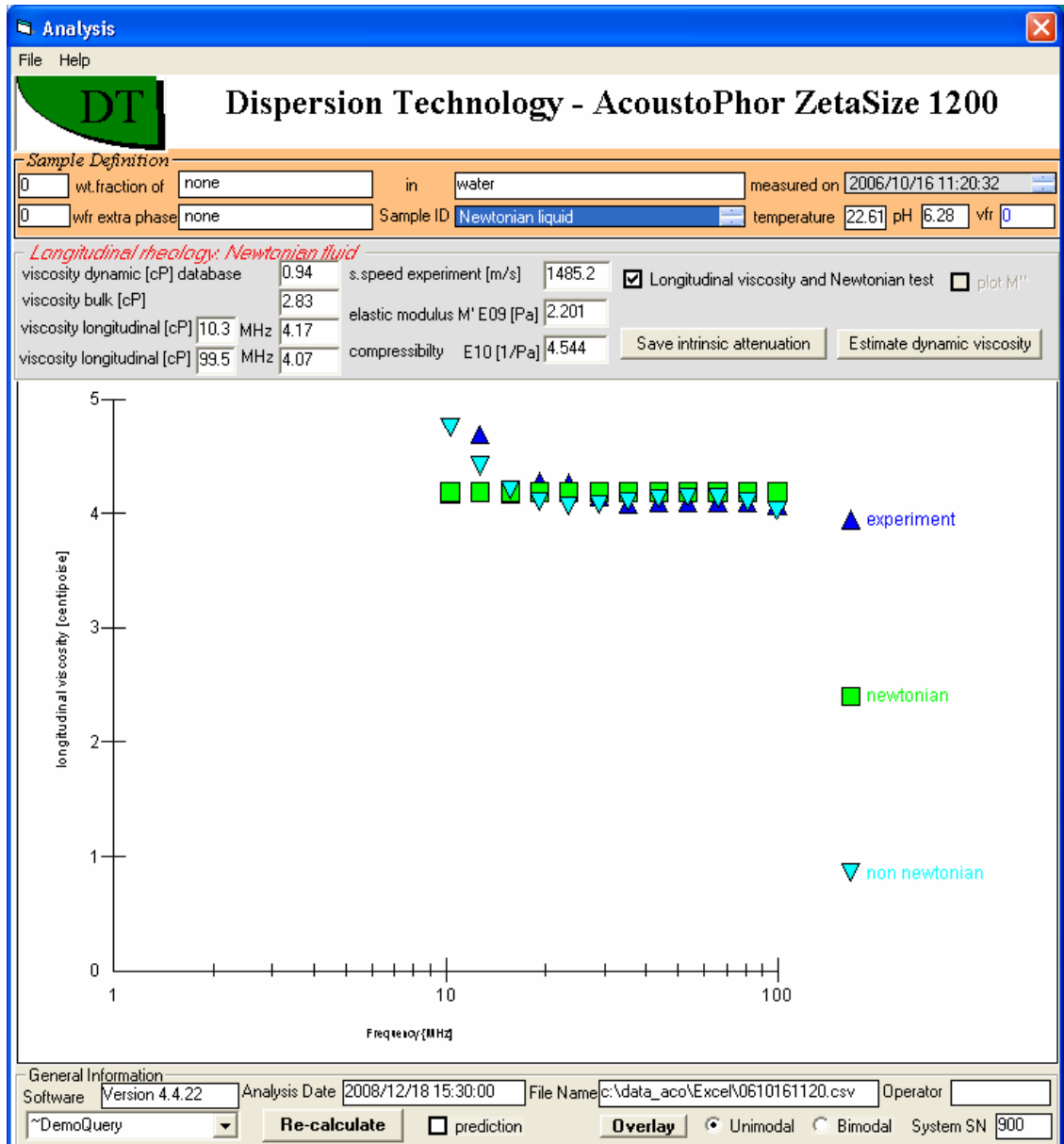


Figure 9

## Printing and Saving Analysis as a Test Report

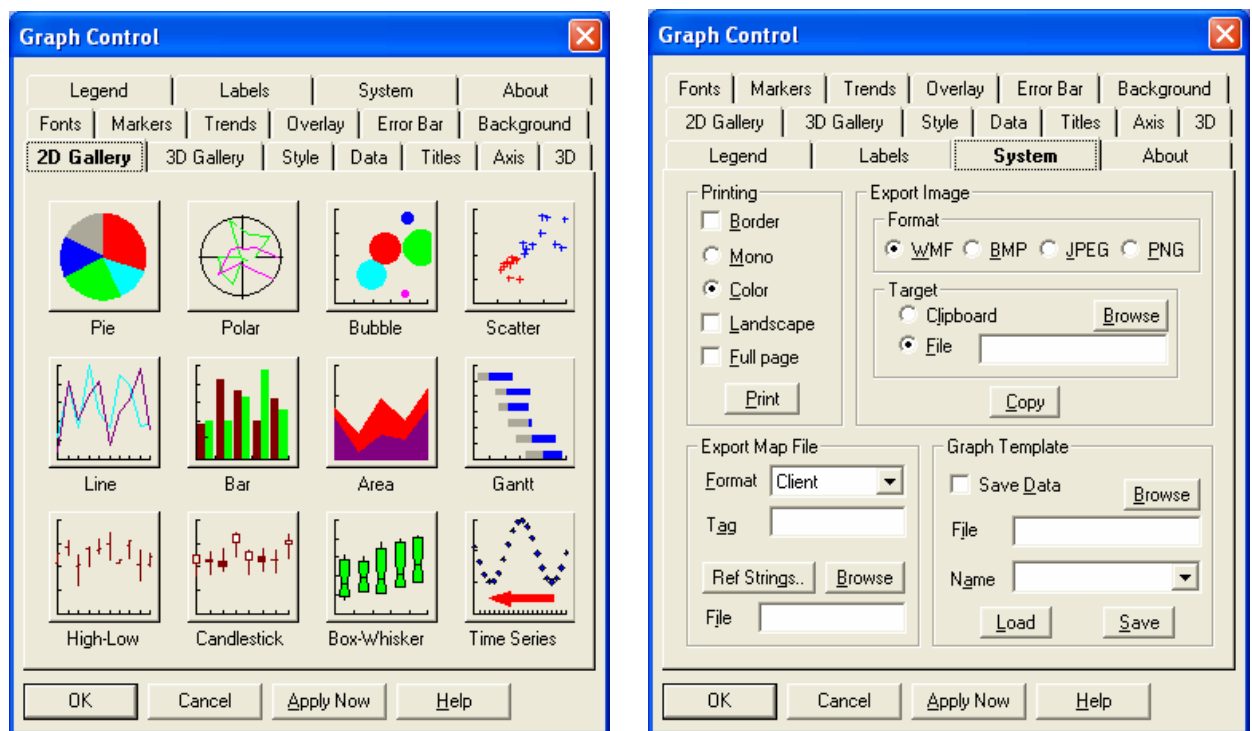
Click on **File - Print analysis** as report to print a copy of the present report to the printer  
Check **File- AutoPrint** if you want to generate this report automatically each time you make a measurement.

If you modify the report format you can save this so that it will be used next time the system is started by clicking **File-Save report setup**.

You can Paste this form as a report into the MS Word file. In order to do this, use **Alt-PrintScr** keys. This would copy the form to the clipboard. The form must have focus at this point, which is indicated by top bar being blue. Then open MS Word file and do Paste command. Analysis appears there as a picture.

## Saving any graph on the Analysis as separate picture

You can save any graph on the Analysis form as a stand alone picture. In order to do this, click right mouse button. The following form appears, left Figure. .

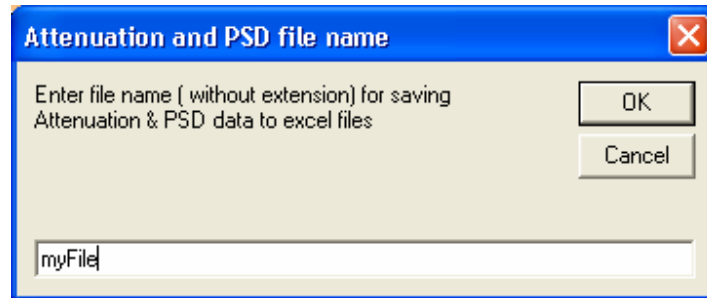


Select tab **System**. The form would become like following. Frame **Export Image** allows you to save this particular graph as metafile. Use button **Browse** to select directory and then type name of the file.



## Saving and retrieving Excel files from the Analysis form

The analysis of a particular measurement can be easily saved to an Excel file. Corresponding functions are available from the Analysis, File. You can save file with your own name using following form.



If saved automatically, the name of the file would be constructed from the measurement data and time.

This one file contains information on all parameters involved in characterization procedure.

The columns are defined as follows

Ft(i)	Frequency, MHz
At(i)	Attenuation, db/cm/MHz
Atlog(i)	Attenuation of best fit unimodal distribution, db/cm/MHz
Atbi(i)	Attenuation of best fit bimodal distribution

d-log	Diameter for unimodal distribution data, microns
psd-log	Unimodal particle size distribution function
cum-log	Unimodal cumulative particle size
d-bi	Diameter for bimodal distributions, microns
psd-bi	Bimodal particle size distribution
cum-bi	Cumulative bimodal particle size
psd-1	Particle size distribution of mode 1 of bimodal distribution
psd-2	Particle size distribution of mode 2 of bimodal distribution

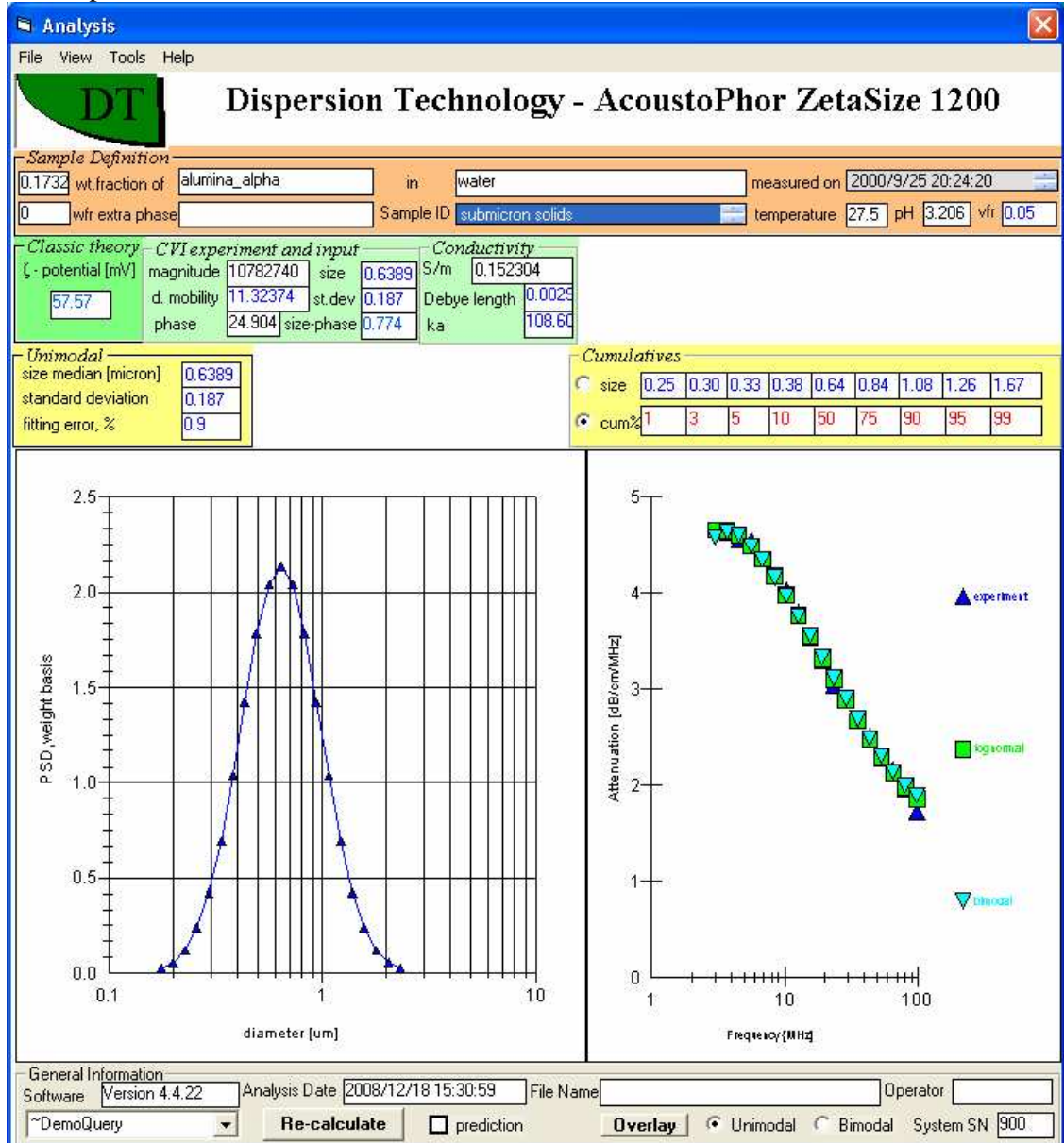
If you want to save attenuation and PSD excel files for every measurement, the check **File - Autosave**

If you want to see the last attenuation data that was analyzed, click on **File- Open last attenuation with Excel.**

If you want to see the last PSD information that was generated by the Analysis form, click on **File - Open last PSD with Excel.** If you want to explore all Excel files created by the Analysis form, click on **File - Explore saved Excel files.**

### Re-calculate option - Selecting a particular record.

Analysis form can re-analyze existing raw data. In order to do this Analysis must be connected to the particular record in the database. It is done with 3 objects on the Analysis page, shown on this picture.



As a first step you should select a Query that contains this record. This is done with the object that is linked to the query “~DemoQuery” now. Clicking on arrow would open a list of available queries.

As a second step you should select a database field for choosing the record. It is done with the two list objects: Sample ID and Measurement Date. Each of them contains list of all records in particular Query. Right button click expands them. In order to connect Analysis to particular record just click on this record in Sample ID or Measurement date.

## Re-calculate option - Prediction.

Button “Re-calculate” starts calculation for the selected record.

Option “prediction” allows you to plot attenuation that corresponds to particular PSD. For this purpose you should check this box and specify desired PSD in the frame Unimodal or Bimodal. This would disconnect searching program and plot theoretical attenuation corresponding to the PSDs in those frames. Analysis must be in Research mode (view, report – unchecked) for this function available.

## Analysis Tools

Analysis has several software tools for performing various calculations. They can be opened using menu Tools. This opens right hand side of the Analysis.

Objects on the very bottom frame allow:

- restricting frequency range for cutting off attenuation points that are in doubt. Use boxes “fmin” and “fmax” for this purpose.
- restricting size range using boxes “dmin” and “dmax”.
- recalculate automatically all records in the given query by checking box “recordset”

**Dispersion Technology - Zeta & Size 1202**

**Sample Definition**  
 latex and in water measured on 9/21/2000 10:52:21 AM  
 Total vfr 0.146 Total vfr 0.12 Sample ID soft particles Temperature 26.2 pH 9.393

**Electric properties**  
 Conductivity [S/m] 0.1232021 Surface charge 10-6 C/cm2 6.96075 Dukhin number 0.227  
 zeta potential [mV] -94.38 Dynamic mobility [micron/sec/V/cm] 1.81513 Maxwell-Wagner frequency [MHz] 28.21645  
 Debye length [nm] 3.32

**Particle size parameters**  
 size median [micron] 0.0626 size median mode 2 [micron] none  
 standard deviation 0.011 larger mode fraction vfr2/vfr none  
 fitting error, % 7.6 mean size [micron] 0.0624

**Cumulatives**  
 size 0.059 0.060 0.060 0.061 0.063 0.064 0.065 0.065 0.066  
 cum% 1 3 5 10 50 75 90 95 99

**Attenuation (dB/cm/MHz) vs Frequency (MHz)**  
 The plot shows experimental data (blue triangles) and theoretical fits (green squares) for unimodal and bimodal distributions. The x-axis is logarithmic from 1 to 100 MHz, and the y-axis is linear from 0.0 to 0.4 dB/cm/MHz.

**General Information**  
 ~DemoQuery Software Version 5.6.31 File Name  
 Analysis Date 7/31/2012 10:29:52 AM Operator System SN 356

**Re-calculate**  prediction

**Right-hand side panels:**  
 - **Vfr search:** automatic, vfr only, 0.12, Save  
 - **Structure:** automatic, Hook coeff. only, 0, microviscosity, 0 cP, porosity/fractal, 3  
 - **Large particles:** automatic, 1, Save  
 - **Thermal properties:** automatic, th. expansion only, 2.700, Save  
 - **Setup parameters:** fmin 3, fmax 99.5, recordset, dmin 0.001, dmax 1000, error fit threshold 3

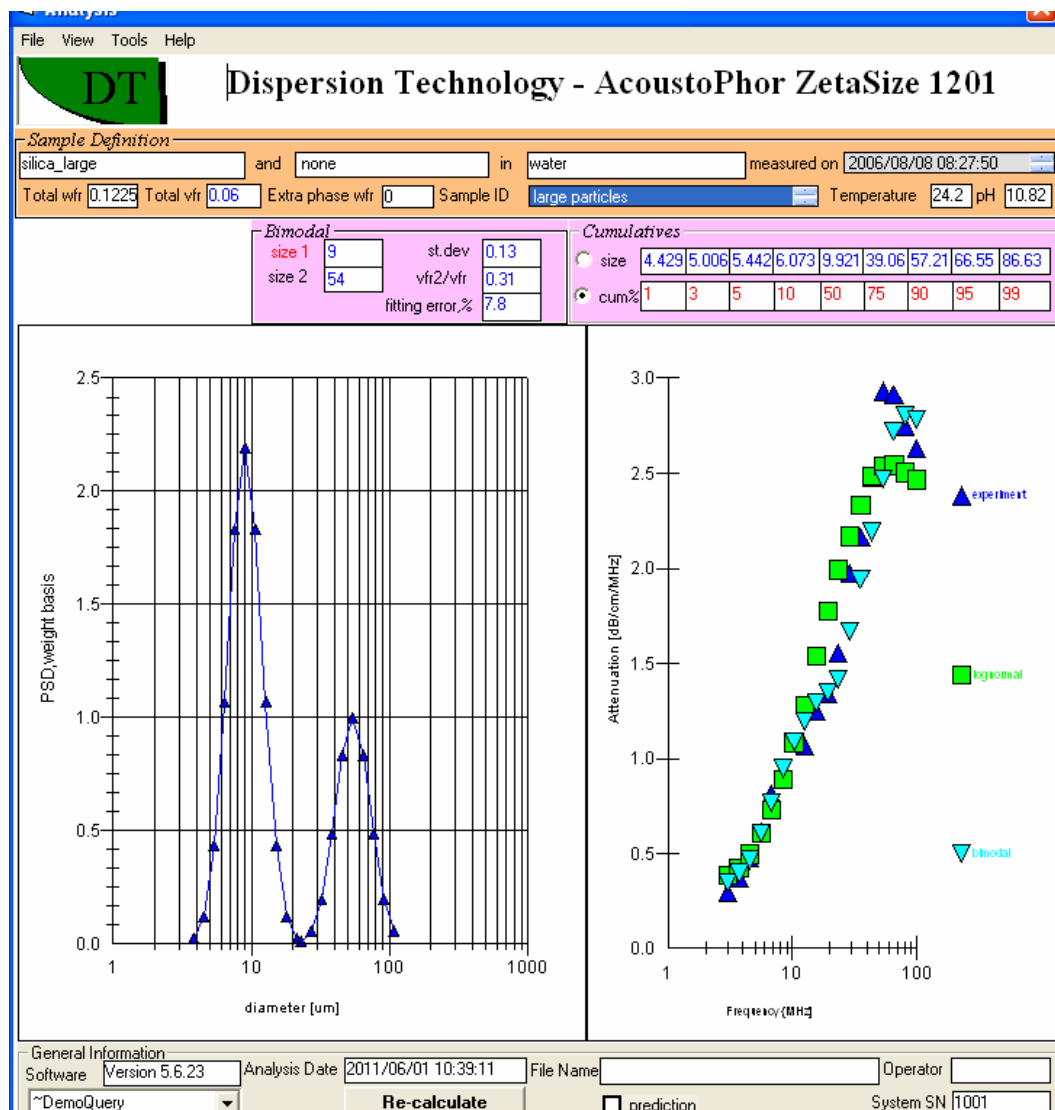
## Enforcing unimodal or bimodal PSD

Object “error fit threshold” determines minimum improvement in the fitting error that allows software to claim bimodal PSD over unimodal one. If this parameter is set to high value, for instance 100, software would always select unimodal PSD only.

Alternatively, if this parameter is very small, bimodal PSD would be predominant. Default value is 3%. It is our experience that this value is the most realistic.

## Analysis Tools - Primary particle size known.

In some cases size of the primary particles is known. The main purpose of the Acoustic measurement is determination size of the aggregates. Software allows setting up size of the primary particles and perform search for aggregates size only. In order to achieve this you should click on the label “size 1”. It becomes red as shown on Figure below. Then, place known size of primary particles in the box for the size 1. Click re-calculate. Software would search only for aggregates size. In order to restore original setup, click on the “size 1” again. It will become black. This is a flag for the full bimodal search.



### **Analysis Tools - Volume fraction search.**

Usually volume fraction or weight fraction must be input parameters on HOME page with precision roughly 0.1%. However, in some cases attenuation contains enough data for extracting this parameter together with PSD.

The volume fraction can be calculated from the Attenuation data for solid particles having a particle size between 0.1 to 1 microns where for slurries that are not structured.

The VFR search frame contains a box “automatic”. Checking this box will run search for that vfr and PSD that provides the best fit between theoretical and experimental attenuation spectrum.

In some cases, one might know the PSD for the given sample, but not the weight fraction. The VFR search frame also includes a check box “vfr only” and allows one to compute the vfr that provides the best fit between theoretical and experimental attenuation given the known particle size.

User can also change wfr and vfr for given sample using the same frame. New VFR must be placed in the text box and then click SAVE button. You will see that both WFR and VFR for this particular record will change to the new values. Then you can recalculate this record for these new values of WFR and VFR.

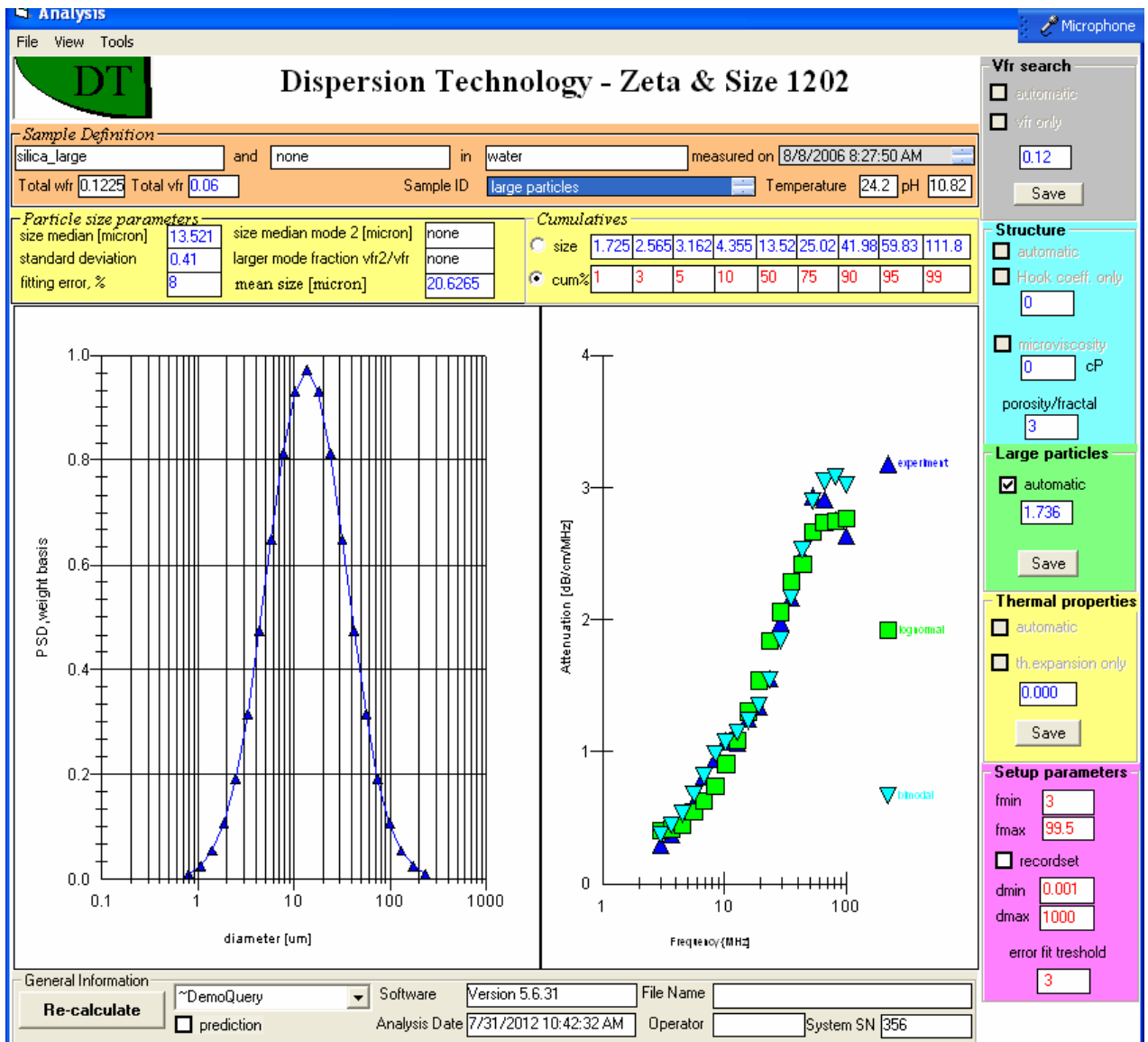
### **Analysis Tools - Scattering coefficient search**

The Scattering coefficient is a property of large particle material. This parameter accounts for scattering deviation from the pure elastic mode.

The border between large and small particles is 10 microns, but in some cases even 3 microns particles might be considered large. That is why search for scattering coefficient might be important for both prototypes of solid particles (see section Defining New Material): solid submicron particles, material type 1 and large particles – material type 2. .

This parameter set to 1 for the prototype 1 and 2 for prototype 2, when a new material created using Define Material form, see menu Tools of the Home page. Searching for large particles is very different than for small ones, that is why we have 2 different material prototypes with border line around 3 microns. However, in the range from 1 to 10 microns, sometimes prototype 1 gives better results, sometimes prototype 2. That is why we recommend to try both prototypes when you start with the new material.

After one creates a new material for large particles and uses it for measurement, Analysis would automatically search for large sizes recognizes that material type is 2. It would also recognize that scattering coefficient is in default. It would run “automatic” search box this parameter and PSD. You can re-run this search if you check box “automatic” on the frame “Large particles”.



After it finds the best value of “scattering coefficient” it would display message asking you if you are happy with the size. If not, you have option to search for scattering using known size, check box “scatter only” and specify this size in Unimodal frame.

Button “save” allows you to save the best value of the scattering coefficient directly to the database.

### Analysis Tools - Thermal expansion coefficient search.

Thermal expansion coefficient is a material property of soft particles, such as emulsion droplets and latex particles. These are material types 3 and 6 when New Material is created in the Define Material form, see Home page, menu TOOLS.

When Define Materials create a new material record in Material database it assigns default value for this parameter. If user do not correct it, software would know that this parameter has not been modified and remains at default value.

Then, when user measures dispersion with this material as particle, software would recognize necessity to adjust this parameter, because it is at default value. Analysis would automatically start search for the optimum value of thermal expansion and would ask user at the end if he likes results. If he does like result, then he can save it using Save button next to the box “th.exp.only”. If this button is not visible you can activate it by clicking and then un-checking the box “automatic”.

If user do not like results, then he can start re-calculating using boxes “automatic” and th.exp.only” on the frame “Thermal properties particle”.

The screenshot displays the 'Dispersion Technology - Zeta & Size 1202' software interface. The main window is divided into several sections:

- Sample Definition:** Includes fields for 'latex', 'in water', 'measured on' (9/21/2000 10:52:21 AM), 'Total wfr' (0.146), 'Total vfr' (0.12), 'Sample ID' (soft particles), 'Temperature' (26.2), and 'pH' (9.393).
- Electric properties:** Fields for zeta potential (-22.53 mV), conductivity (0.1232021 S/m), surface charge (-2.73315 C/cm<sup>2</sup>), Dukhin number (0), Debye length (3.32 nm), dynamic mobility (1.81513 micron/sec/V/cm), and Maxwell-Wagner frequency (28.21645 MHz).
- Particle size parameters:** A table with columns for size median [micron] (0.0155), size median mode 2 [micron] (none), standard deviation (0.239), larger mode fraction vfr2/vfr (none), fitting error, % (4.9), and mean size [micron] (0.0179).
- Cumulatives:** Radio buttons for 'size' and 'cum%', with a list of cumulative percentages (1, 3, 5, 10, 50, 75, 90, 95, 99).
- Graphs:**
  - PSD weight basis vs diameter [um]:** A plot showing a unimodal distribution peaking at approximately 0.015 micrometers.
  - Attenuation [dB/cm/MHz] vs Frequency [MHz]:** A log-log plot showing a linear relationship between attenuation and frequency, with data points for 'experiment', 'lognormal', and 'unimodal' fits.
- Right-hand panels:**
  - Vfr search:** Checkboxes for 'automatic' and 'vfr only', a text field (0.12), and a 'Save' button.
  - Structure:** Checkboxes for 'automatic' and 'Hook coeff. only', a text field (0), and a 'Save' button.
  - Large particles:** Checkboxes for 'automatic' and '1', and a 'Save' button.
  - Thermal properties:** A checked 'automatic' checkbox, an unchecked 'th.expansion only' checkbox, a text field (6.09), and a 'Save' button.
  - Setup parameters:** Text fields for 'fmin' (3), 'fmax' (99.5), 'dmin' (0.001), 'dmax' (1000), and 'error fit threshold' (3).
- Bottom section:** 'General Information' with a 'Re-calculate' button, a 'prediction' checkbox, 'Software' (Version 5.6.31), 'File Name', 'Analysis Date' (7/31/2012 10:37:25 AM), 'Operator', and 'System SN' (356).

Check boxes “automatic” and “th.exp. only” works the same way as similar boxes on the vfr and scattering coefficient searches. Box “automatic” ensures that PSD parameters and ThExp.Coefficient are used for fitting attenuation. Box “th.exp.only” performs search of th.exp. only for PSD specified in Unimodal PSDF frame.

### **Analysis Tools - Structure.**

Some dispersed systems are structured. This can be modeled as if particles are connected with strings, polymers or other specific forces. Oscillation of these strings contributes to attenuation. There is theoretical model of such attenuation, see the book, section 4.3.4 and 6.3. If box “structure” checked, software accounts for this additional attenuation. It searches for the best value of the second Hookean coefficient that determines dissipative properties of the strings. This parameter is shown in the box next to the “structure” check.

Newsletter 8 on the [www.dispersion.com](http://www.dispersion.com) describes result of this option for structured alumina dispersion.

One should use it if regular searching procedure fails to fit high frequency attenuation, as described in the section 6.3 in the book. This extra attenuation comes from structure. This is raw data for calculating structuring coefficient.

### **Analysis Tools - Microviscosity for sizing and zeta potential in non-Newtonian liquids, polymeric solutions.**

There are samples where solid particles are dispersed in non-Newtonian liquid. This raises question of viscosity that should be used for extracting correct particle size distribution from the measured attenuation spectra. Quite often this viscosity is not the same as macroscopic viscosity measured with regular viscosity meter. Section 4.7 in the book provides estimates of the particles motion in ultrasound field. Displacement is very small. It means that small particles would not sense presence of viscosity modifiers if their chain length exceeds particle size. In such case viscosity of the basic liquid must be used.

In the opposite case of large particle with sizes much larger than chain length, macroviscosity must be used.

Introduction of the new liquid for particle sizing and zeta potential might be complicated in the case if this liquid is non-Newtonian. Many aqueous polymeric solution can serve as examples. The most complicated part is selection of the proper dynamic viscosity. Viscosity measured with low frequency rheological instruments is not adequate and applicable at high frequency due to non-Newtonian nature of such liquids.

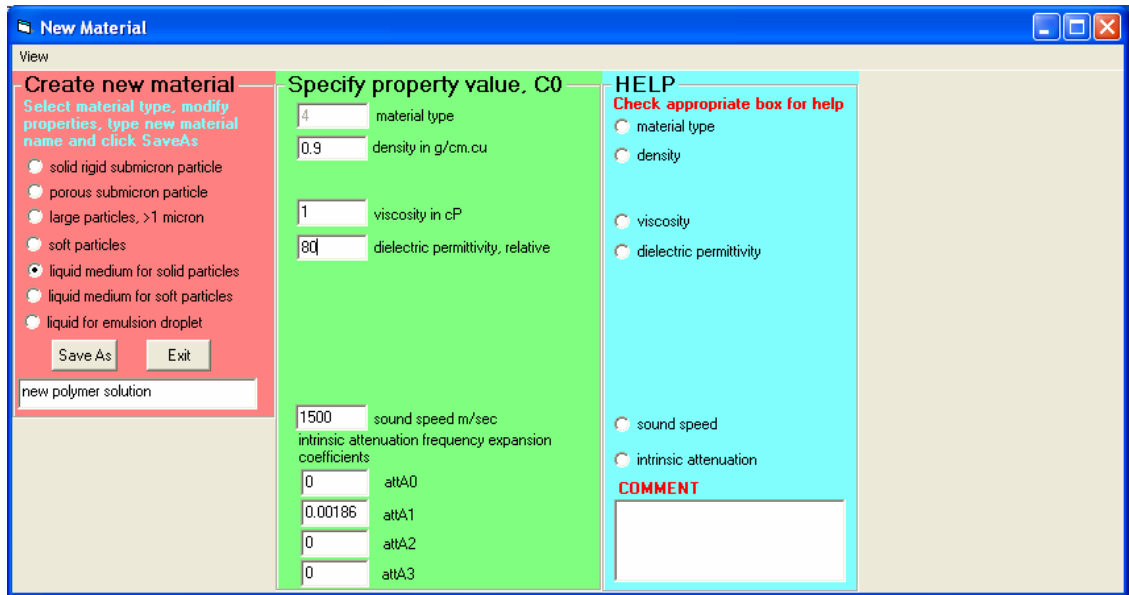
We suggest procedure here that at least specifies high limit of the “microviscosity”. Concept of “microviscosity” is used for instance by Harvard scientists: M.L. Gardel, M.T. Valentine and D.A. Weitz from Department of Physics and Division of Engineering and Applied Sciences, who published large review on this subject.

In order to define this new liquid properly you MUST measure this new non-Newtonian liquid by itself, with no particles. This would yield “intrinsic attenuation”, “sound speed” and “microviscosity”.

Fill Acoustic sensor measuring chamber with this liquid.

First of all you should define a new liquid using Home page, Tools, Define material. When this form appears: select “liquid medium for solid particles”. This form would look like below.



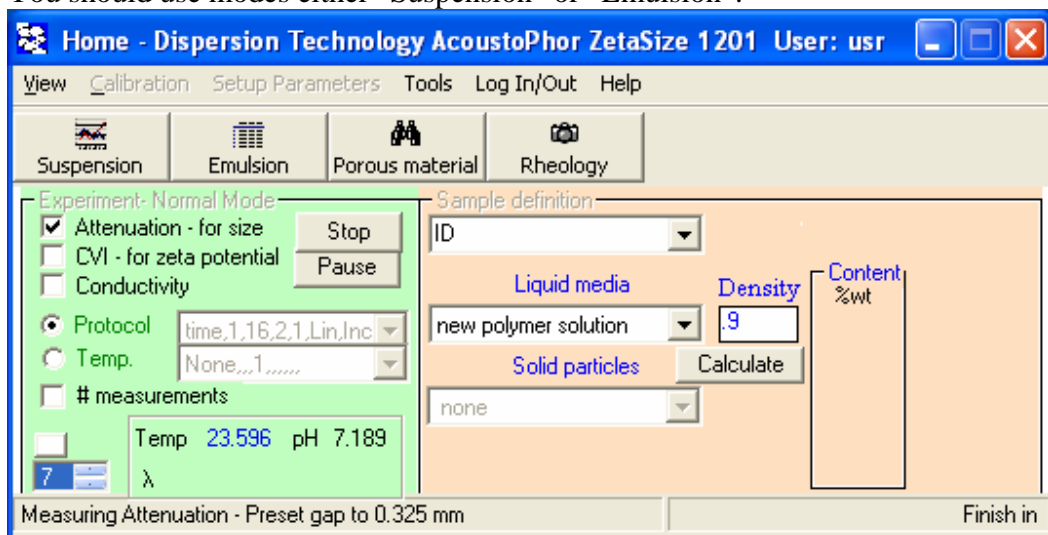


In the case if you are dealing with aqueous solution – change dielectric permittivity to 80, which will be important for zeta potential only, not sizing.

I defined material name as “new polymer solution”. You can use any other name that is appropriate for you.

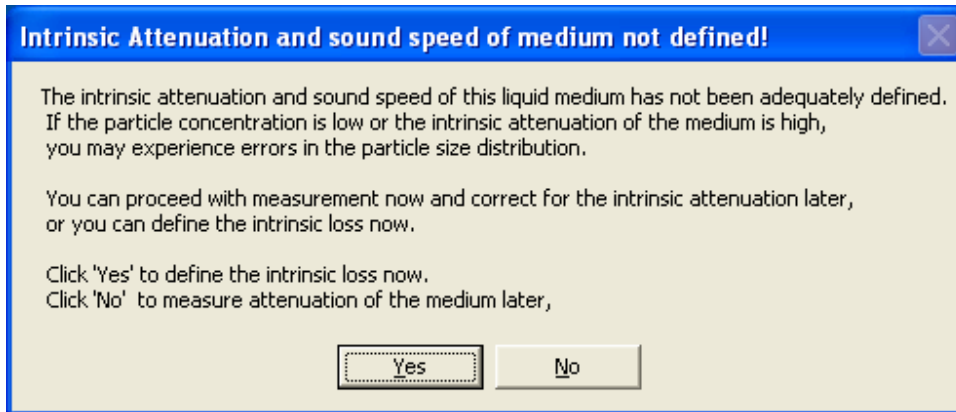
Click Save AS.

The next step – selecting this new liquid on the Home page – Liquid media. You should use modes either “Suspension” or “Emulsion”.



When you select this new material the following text message appears. It is warning that this liquid is not yet properly defined and software would object using it with particles.

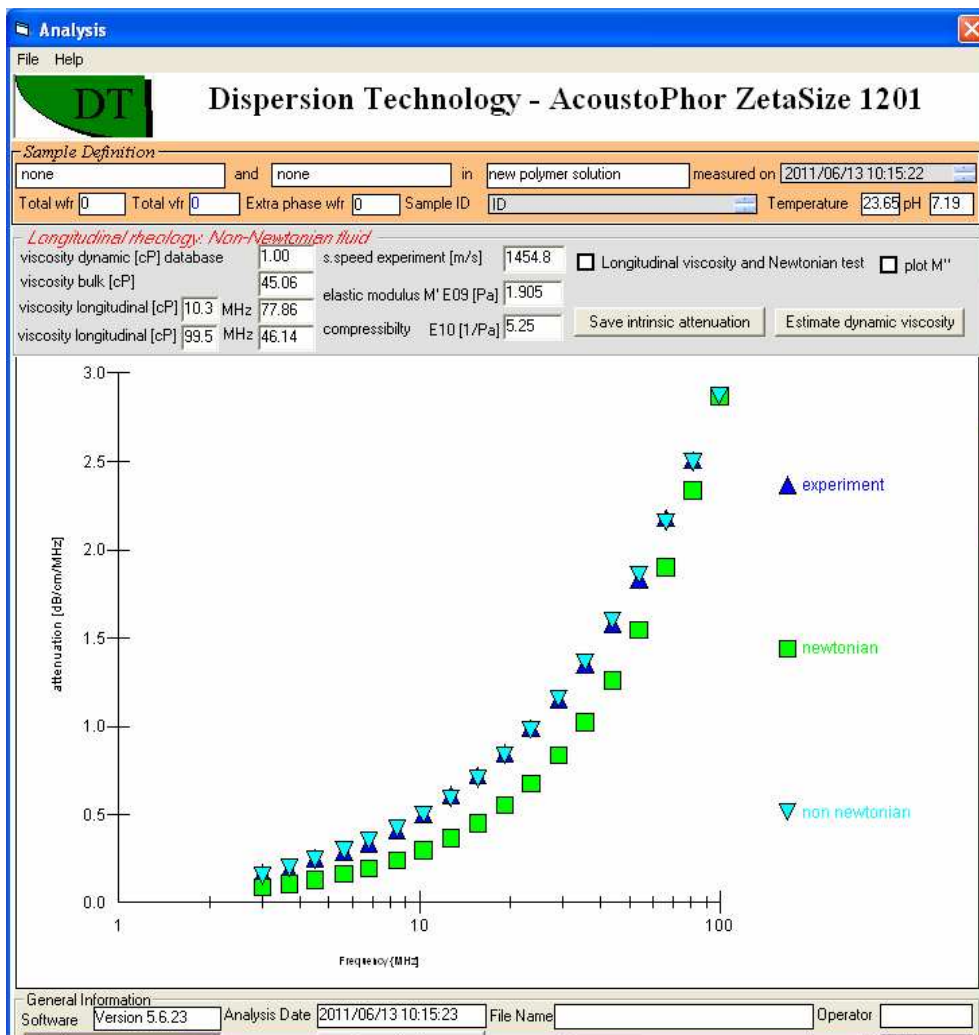
Click YES.



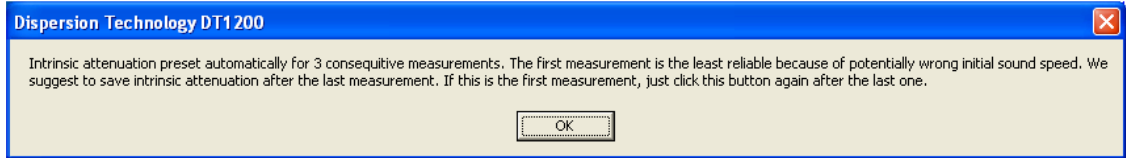
There will be 3 more messages with obvious warnings – Yes for all of them.

Instrument will perform 2 measurements in raw. The first measurement might be affected with a wrong sound speed. Only result of the second measurement is reliable.

After the second measurement finished, Analysis page as following appears.



Then, message. Click Yes.



The first step – Saving “intrinsic attenuation” and “ sound speed”. Analysis has a button “save intrinsic attenuation”. Click on it. This saves acoustic properties of the liquid.

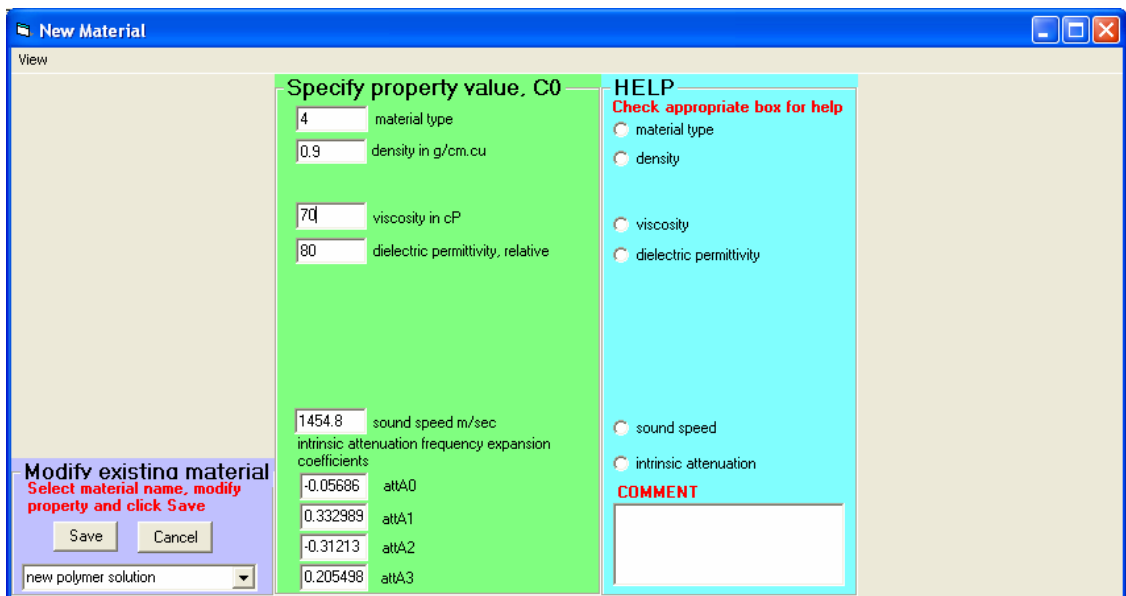
The next step is determination of the “dynamic viscosity” that would be adequate for high frequency range.

We can use relationship between “dynamic viscosity” and “bulk viscosity”. Definition of the “bulk viscosity” (introduced 150 years ago) is overviewed in the paper “Dukhin, A.S. and Goetz, P.J. Bulk viscosity and compressibility measurement using acoustic spectroscopy," The Journal of Chemical Physics, Vol.130, Issue 12, (2009)”

On the form above bulk viscosity is 45.06 assuming dynamic viscosity as 1. However, for many polymeric solutions measured viscosity at low frequency is much higher than 1. Let us assume that it is 70.

Open Define Materials again, Goto “Modify existing material, find your newly defined material, and change viscosity to 70 see below.

Save.

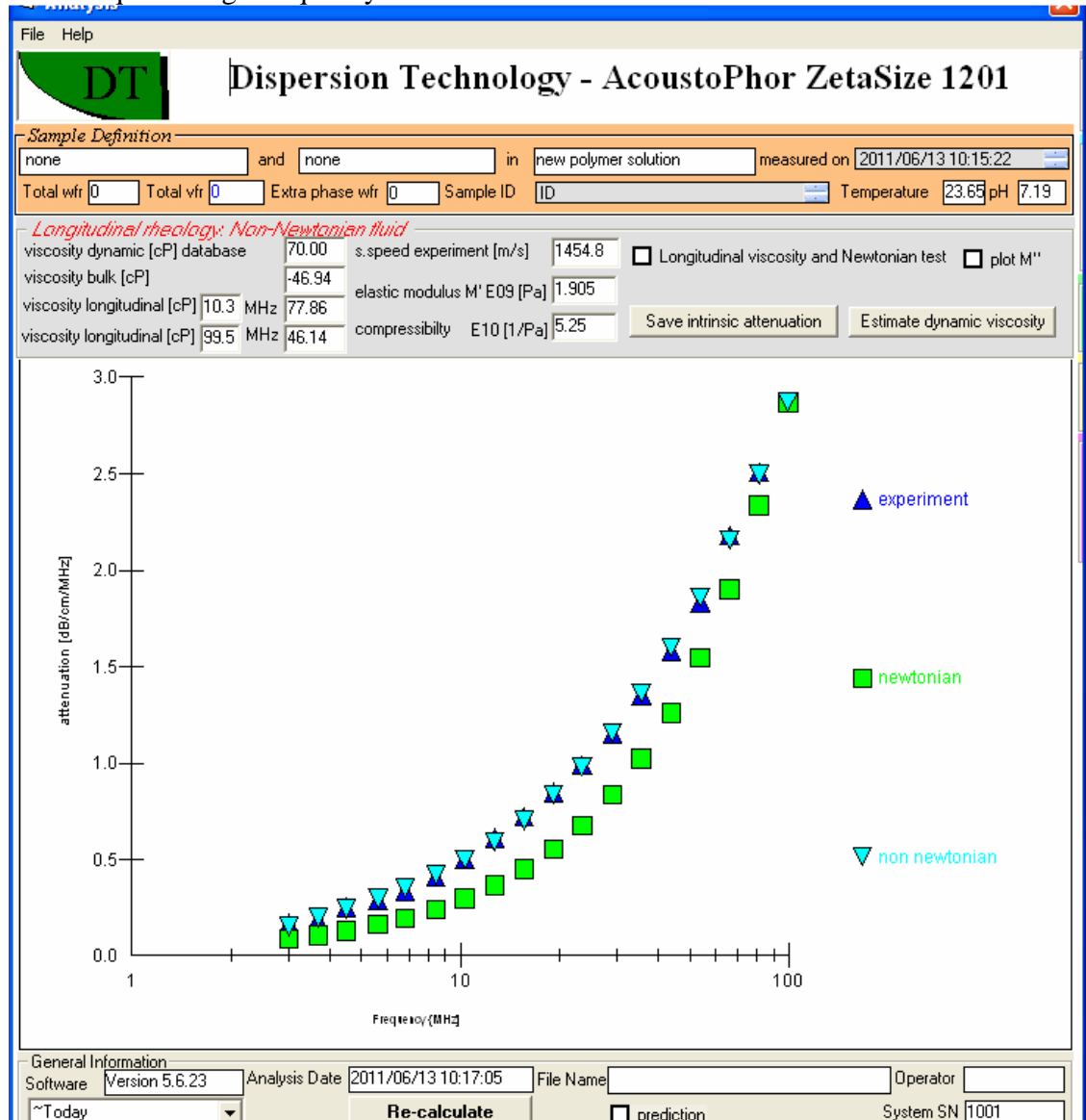


Go to Analysis again and click Re-Calculate at the bottom of the page.

Analysis will change viscosity values as shown below.

You can see that bulk viscosity is negative now. IT IS IMPOSSIBLE. It occurs due to too high dynamic viscosity.

We can use this constrain for determining how possibly high dynamic viscosity might be for this particular liquid at high frequency.



The higher “dynamic viscosity” the more negative will be “bulk viscosity”.

Lowering “dynamic viscosity” would eventually make “bulk viscosity” positive. The highest number for “dynamic viscosity” will be the one when “bulk viscosity” equals value in pure water, which is 2.4. This is condition for polymer aqueous systems. For non-aqueous we suggest to use just 0 for “bulk viscosity”.

With this assumption we restrict polymers molecules only to transitional motion. Bulk viscosity reflects rotational and vibrational degrees of freedom. It is quite possible that they are not important for large polymers molecules.

This relationship between “dynamic” and “bulk viscosity” is result of fixed “longitudinal viscosity”. There is a general notion of longitudinal viscosity  $\eta_{long}$ , which determines dissipation due to longitudinal stress in non-Newtonian liquid:

$$\eta_{long} = \frac{2\alpha_{long}\rho V^3}{\omega^2} \quad (10)$$

We can use also the following approximate equation leaning this parameter to “bulk and dynamic” viscosities:

$$\eta_{long} = \eta^b + \frac{4}{3}\eta$$

We assume the last relationship when calculate bulk viscosity for non-Newtonian liquid. It is the best we can do.

For the last Analysis longitudinal viscosity equals:

$$\eta_{long} = -47 + \frac{4}{3}70 = 46.3$$

Now, if we assume that bulk viscosity is 2.4, as for water, this equation can be used for determining “dynamic viscosity:

$$46.3 = 2.4 + \frac{4}{3}\eta$$

which yields  $\eta = 32.9$ .

Actual dynamic viscosity might be even smaller. However, even this number works very well in many cases.

In order to verify that this simple calculation works, we go to Define material, change dynamic viscosity to 32.9 and then Re-Calculate with Analysis.

This procedure is illustrated with following two Figures.

**New Material**

View

**Specify property value, C0**

4 material type  
 0.9 density in g/cm.cu  
 32.9 viscosity in cP  
 80 dielectric permittivity, relative

1454.8 sound speed m/sec  
 intrinsic attenuation frequency expansion coefficients

-0.05686 attA0  
 0.332989 attA1  
 -0.31213 attA2  
 0.205498 attA3

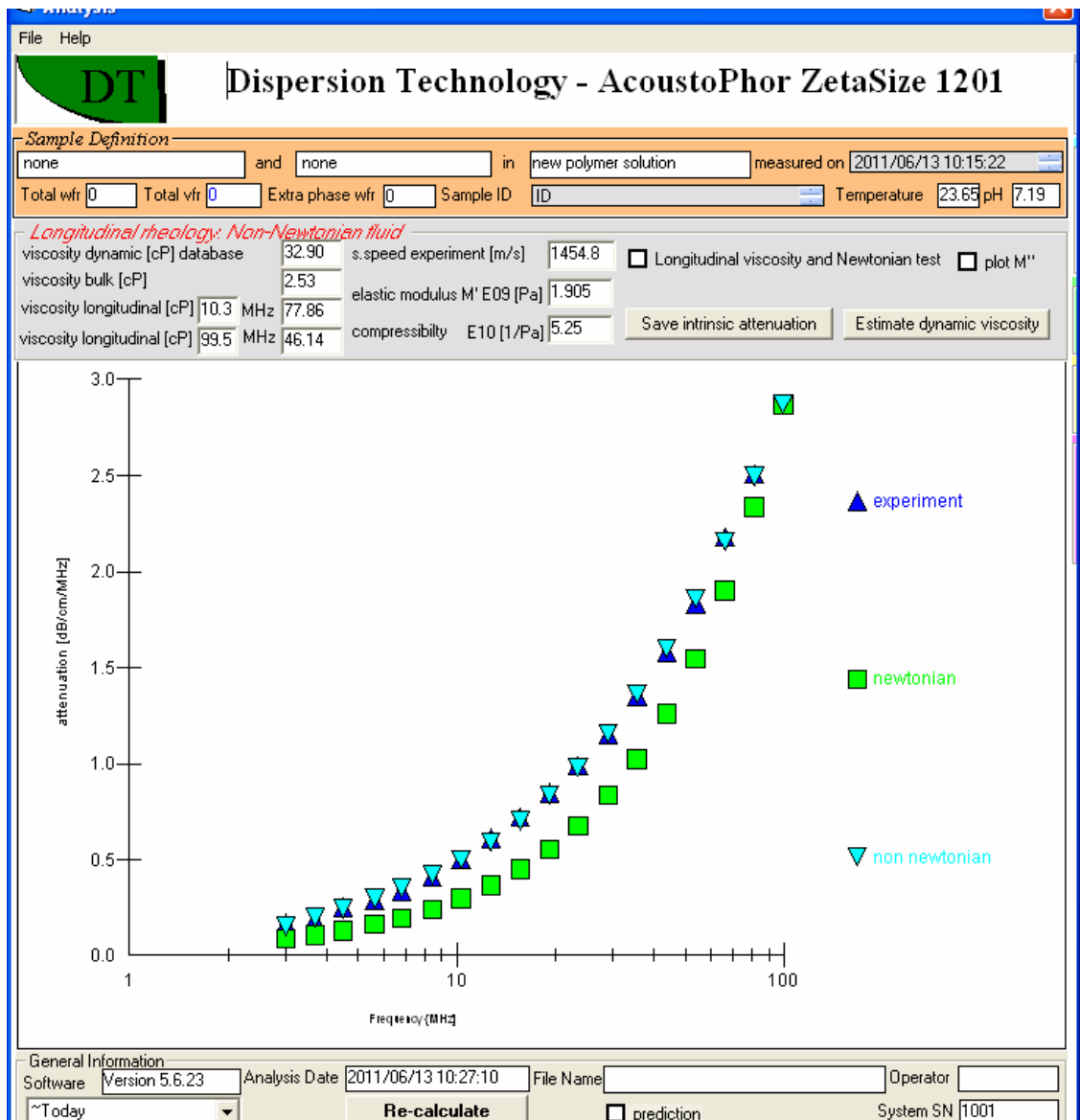
**HELP**  
 Check appropriate box for help  
 material type  
 density  
 viscosity  
 dielectric permittivity  
 sound speed  
 intrinsic attenuation

**COMMENT**

**Modify existing material**  
 Select material name, modify property and click Save

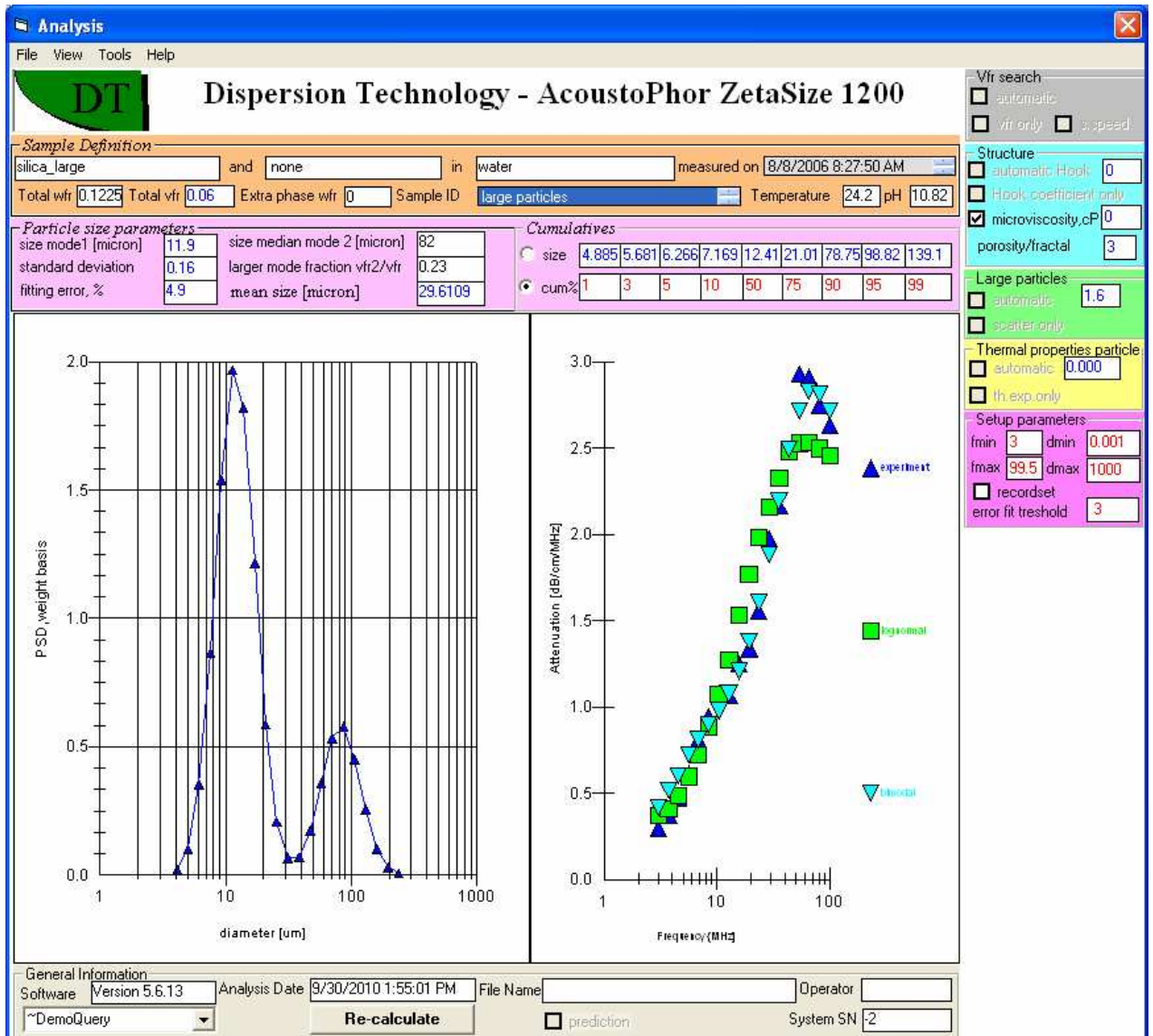
Save Cancel

new polymer solution



If this value of the dynamic viscosity is still would not provide good fit for attenuation spectra with particles in this liquid, we suggest even more reduction of the dynamic viscosity.

Analysis has a tool that helps finding microviscosity when particle size is known. If we would know particle size then we could calculate microviscosity as adjustable parameter providing the best fitting to experimental attenuation. This is achieved with the box "microviscosity" on the frame "Structure, see Figure below. Particle size is placed in the boxes of Unimodal PSD.



### Analysis - Porous and fractal particles.

There are particles that contain trapped water. We have two models for them, see section 2.4.2 , page 37 in the book. One model is “porous particles”, the other model is “fractal” particles. There are two prototypes for creating such materials using “Define material” form.

Parameter “porosity” determines how much water is trapped in the particles in the “porous” model. Value of this parameter changes from 0.11 to 0.99. It is shown in the box next to pH on the top of Analysis. It can be changed only from Define Material form. It is saved in the Material table, field “fractal number”.

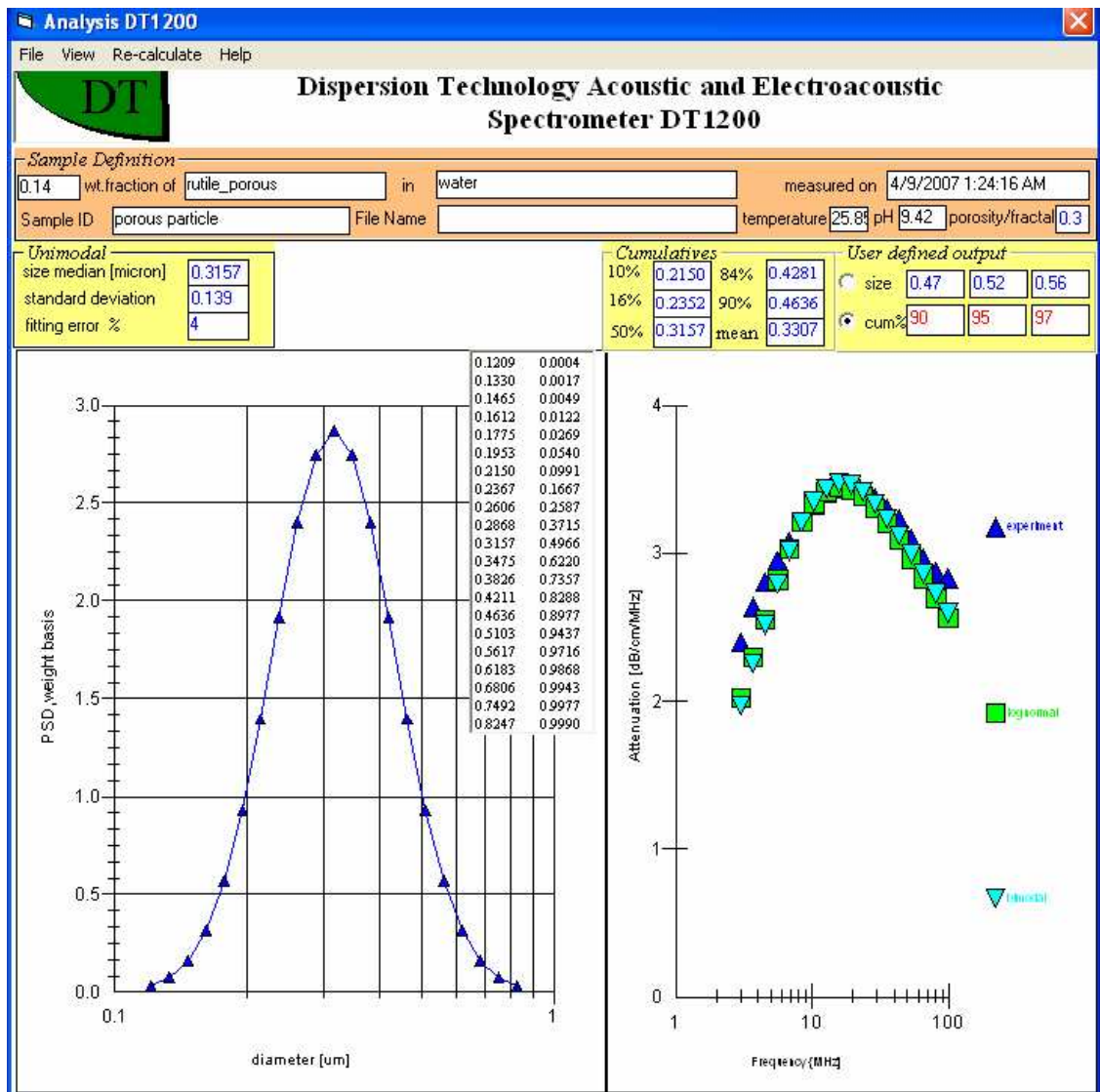
This parameter is used for sizing and zeta potential.

For sizing, all particles with material type 7 are assumed to be porous and large. In this sense the size search range is the same as for type 3.

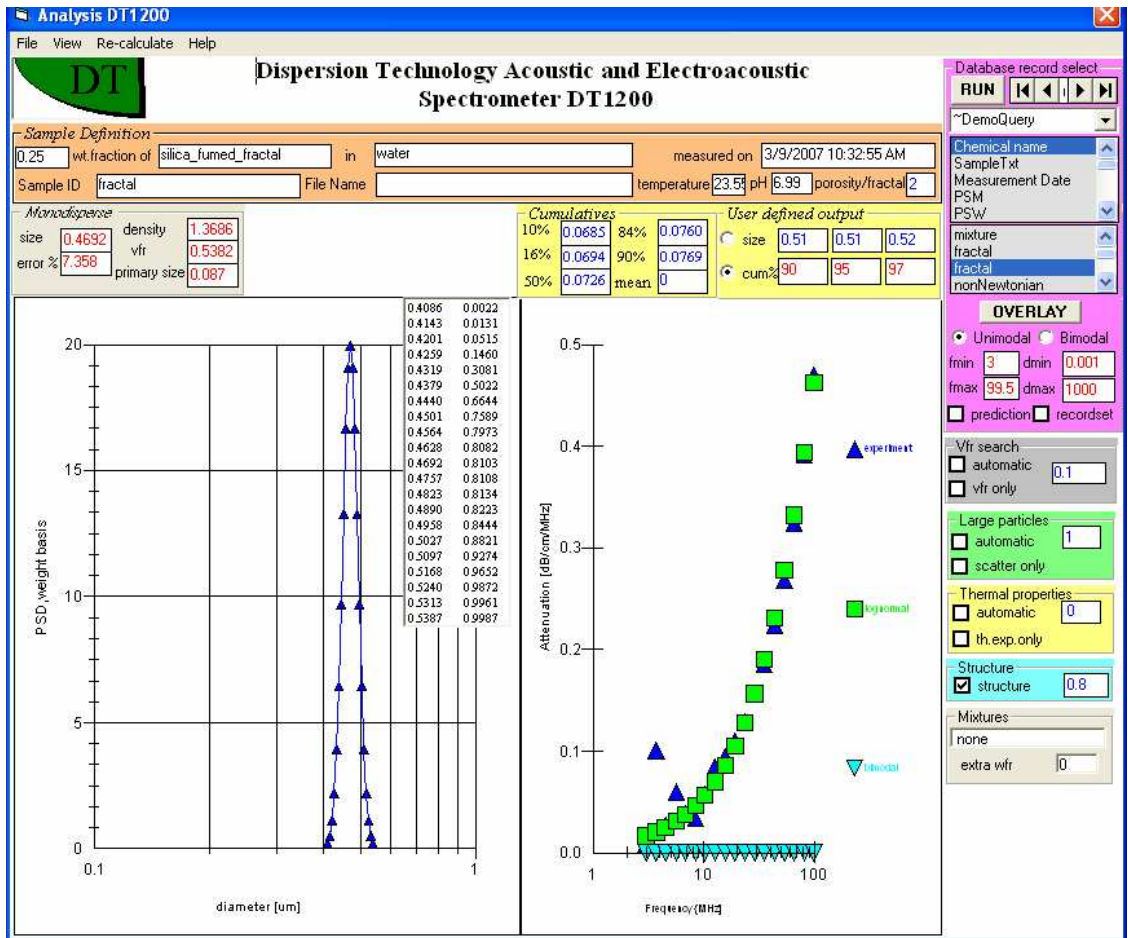


For zeta potential, there is section at the end of this manual “Characterization of Porous Materials. Measurement of Zeta potential”. There is difference between porous particles in flow and porous particles in the sediment.

Porosity is not adjustable. User can try various numbers, till fit is good and size is as expected. Size dependence on this parameter is very non-linear. It is our experience that it becomes important when porosity exceeds 0.3.



The other model assumes “fractal” particles. There is a paper on the web site that describes this notion. Basically, it is porous particles with trapped water and repeated symmetry of structure. Fractal model has a characteristic number- “fractal dimension”. For solid particles it is 3. For linear chains of particles it is 1. Fractal particles belong to type 1. When New Material is created in Define Material form, fractal number setup to 3 – solid particles. You can change it to range from 1 to 3. This would run fractal search in Analysis. Output of the search is shown on frame “Monodisperse”. We have published paper indicating that the value of the fractal number does not affect size much. We suggest using number 2. However, it is very easy to go to the Define Material form and change this number. You can determine by yourself how influenced this parameter is for your system.



### Re-calculate option - Mixtures of two dispersed phases.

There are two approaches for characterizing dispersions with more than two dispersed phases – see Home page, menu Mixtures. There is also large section on mixtures in the book, chapter 8.2, page 312.

Analysis has a special program for the approach when two dispersed phases are determined on the Home page. There is a frame on the bottom re-calculate options column that displays name of the extra material and corresponding weight fraction. This is a flag that this approach has been selected.

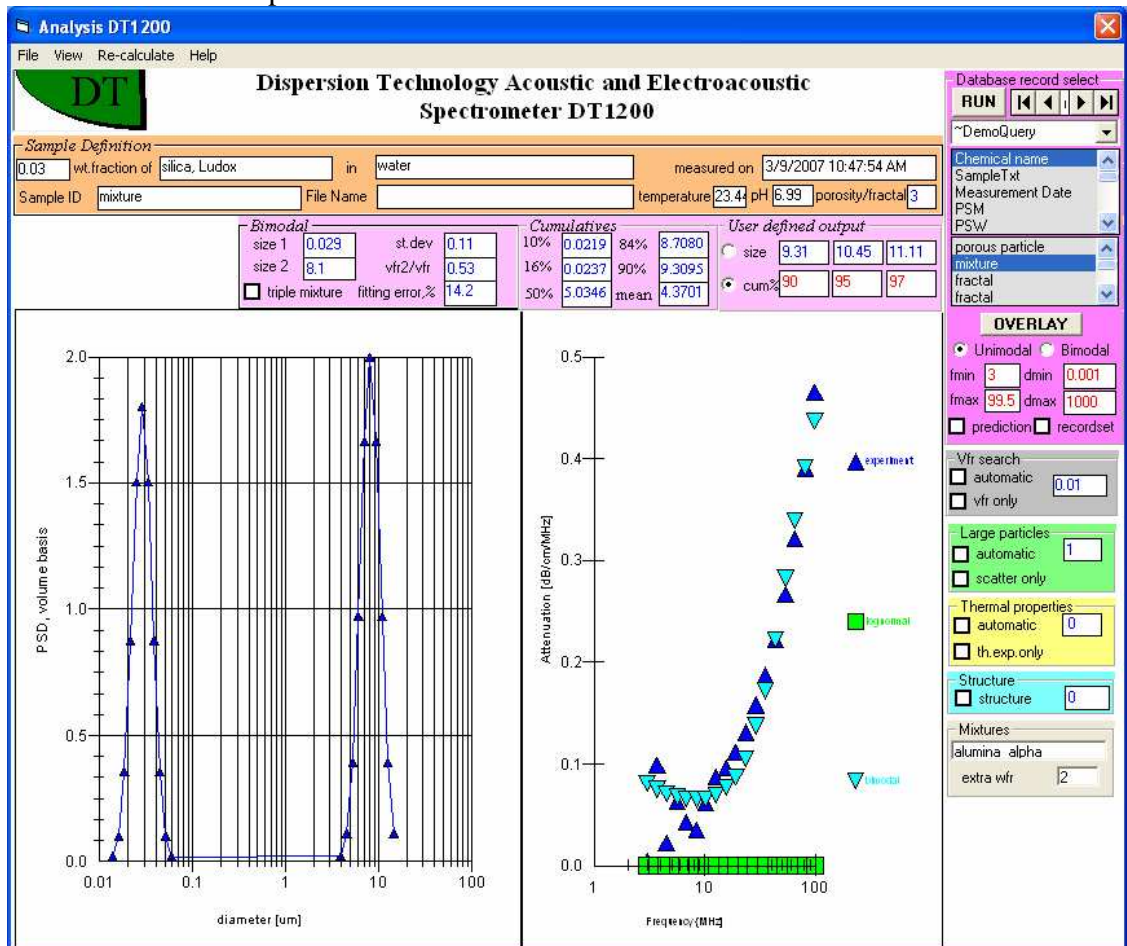
This program can be run by checking box “triple mixtures” on the Bimodal frame. It would run search for tri-modal PSD. The third mode is for aggregates.

The first mode is reserved for the smaller phase particles. This size must be specified in the box “size 1”. It is assumed to be known.

The second mode is reserved for the larger phase particles. This size must be specified in the box “size 2”. It is assumed to be known.

The third mode – aggregates of modes 1 and 2.

Software searches for the amount of free particles for the each mode, and PSD of aggregates. It is shown in the frame “Triple Mixture”.

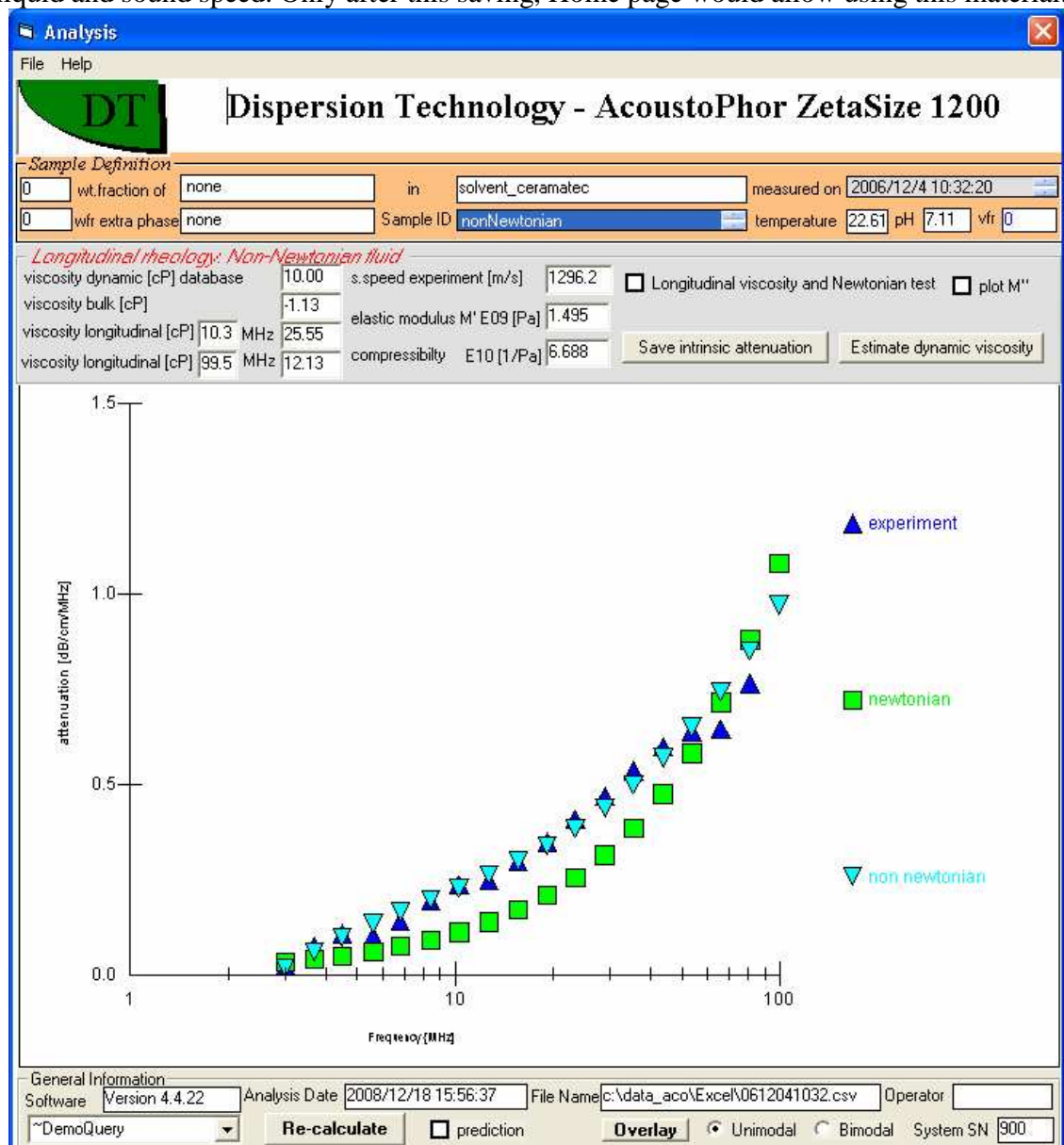


## Saving intrinsic attenuation.

There is a form for creating a new material – Define Material, Home page, Tools. There are three prototypes for the new liquids.. When we use these prototypes for creating a new material, software would know that the liquid by itself must be measured. It would not allow using this liquid with particles unless intrinsic attenuation has been measured.

When you select this new liquid on the Home page, software would setup measurement protocol for three consecutive measurements. The first measurement might be affected by unknown sound speed.

After the second measurement Analysis would offer a possibility to save intrinsic attenuation of the liquid and sound speed. Only after this saving, Home page would allow using this material.



## Defining a new material with relevant input parameters

Proper definition of a new material is extremely important for reliable calculation of the particle size distribution and zeta potential. When we define the new material we **MUST** specify certain input parameters that play key role in calculation. These input parameters are properties of the new material, such as density, viscosity, thermal expansion coefficient etc. List of all parameters that **MIGHT** be relevant in some cases is long. There is detail discussion of the input parameters in the book, chapter 4.6, page 174.

However, it turns out that for particular material only a very few parameters are truly required. The most important are **density** for solid rigid particles, **thermal expansion coefficient** for soft particles, droplets, latex, **intrinsic attenuation** of liquid for sizing, **scattering coefficient** for large particles, **viscosity** of liquid for submicron solid particles, **dielectric permittivity** of liquid for zeta potential measurements. Meaning of all these parameters are given on our book.

Some of these parameters must be measured independently. Some of them can be extracted directly from the measured attenuation spectra. We have created a special program that simplifies introduction of the new material and enforces later user to make proper calculation for determining unknown yet material properties. In order to achieve this purpose we use a set of default **PROTOTYPES**. The form that allows usage of Prototypes for introducing a new material is called Define Material. This form becomes available from menu Tools, Home page, **Define Material**:

The screenshot shows a software dialog box titled "New Material". It is divided into two main sections. The top section, titled "Create new material" (highlighted in red), contains the instruction "Select material type, modify properties, type new material name and click SaveAs". Below this are seven radio button options: "solid rigid particle", "porous particle", "large particles, >10 micron", "soft particles (latex)", "aqueous medium for solid particles", "emulsion medium (oil)", and "emulsion droplet (oil)". At the bottom of this section are "Save As" and "Exit" buttons, and an empty text input field. The bottom section, titled "Modify existing material" (highlighted in blue), contains the instruction "Select material name, modify property and click Save". Below this are "Save" and "Cancel" buttons, and a dropdown menu with "zirconia" selected.

You can either **Create** a new material, or **Modify** existing material.

There are 7 Prototypes for new material as shown below on the Figure of this Form as it appears initially.

In order to **CREATE** a new material user should select appropriate Prototype.

In order to **MODIFY** existing material user should select material from the list of existing material.

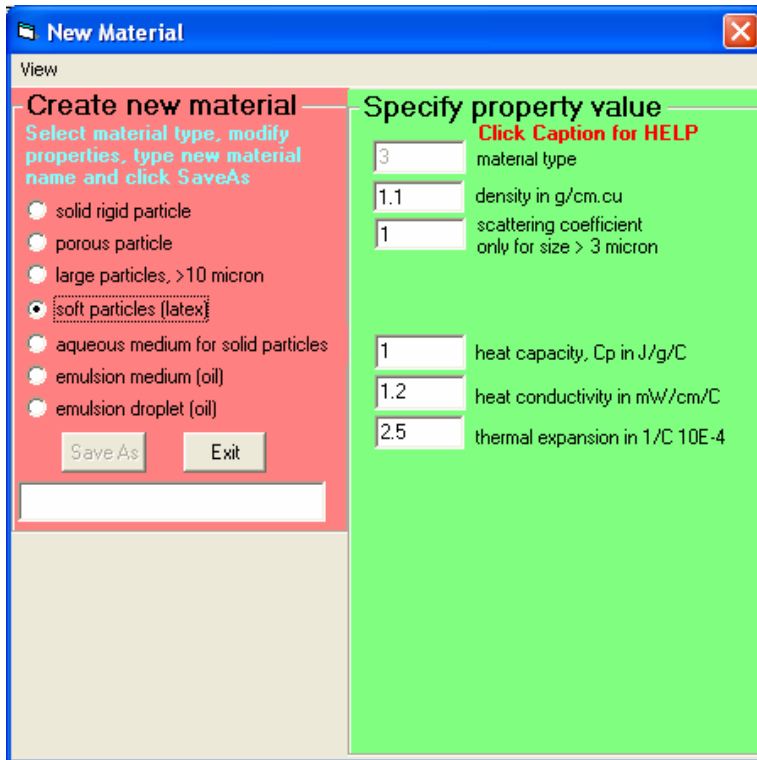
When you selected a particular Prototype, the following form appears. It shows only those material properties that are important for this prototype.

There are Help messages that explain meaning of the every parameter. For instance if you check box “fractal number” the following message appears that explains meaning of fractal number.

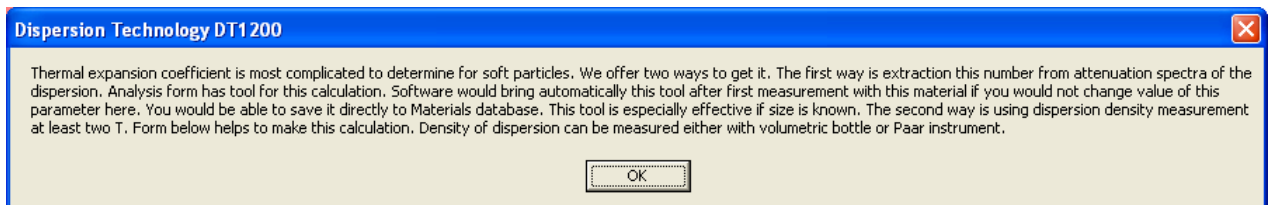
In order to introduce a new material you simply type name of this material, as shown below “my new solid”.

Solid submicron particles require least input parameters- just density. It is also very easy to measure and it is also known in many cases. There is instruction for Content Wizard explaining how to extract density of particles from the density of dispersion. Density of dispersion can be measured with Pycnometer. There is instruction on Pycnometer calibration. There is also well advanced Pycnometer form available from Home page, Tools, Options. It provides means for calculating density even at different T.

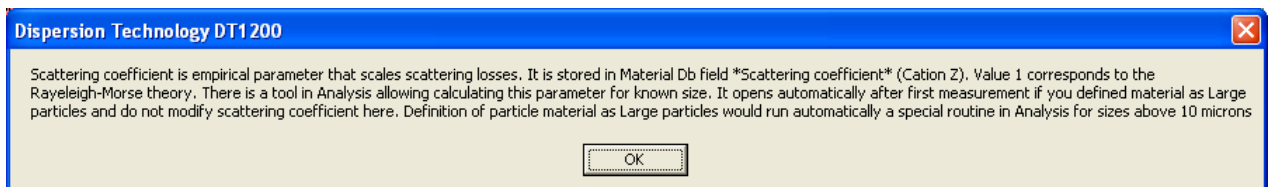
Soft particle require more information, as follows from the form below. Among 3 thermal properties only thermal expansion coefficient varies in rather wide range of values. Two other parameters are almost constant, as shown in the book, page 29.



There is a message that describes ways of getting information on the thermal expansion coefficient. Basically software would force using Analysis software tool for calculating thermal expansion coefficient from the FIRST TIME measured dispersion or emulsion that includes this material.



Similar method is used for calculating scattering coefficient of large particles. There is section 4.4.6 page 170 in the book that describes meaning of this parameter. It can be extracted from the measured attenuation spectra that is FIRST TIME measured with this material, see help message below.



In the case of the new liquid material, software would insist on measuring intrinsic attenuation of this liquid and sound speed. These parameters are properties of all liquid Prototypes, as shown below.



**New Material**

View

**Create new material**  
Select material type, modify properties, type new material name and click SaveAs

- solid rigid particle
- porous particle
- large particles, >10 micron
- soft particles (latex)
- aqueous medium for solid particles
- emulsion medium (oil)
- emulsion droplet (oil)

Save As    Exit

\_\_\_\_\_

**Specify property value**  
Click Caption for HELP

4    material type

0.997039    density in g/cm.cu

0.8904    viscosity in cP

78.85    dielectric permittivity, relative

1496.7    sound speed m/sec

***Intrinsic attenuation of liquid***

frequency expansion		T expansion	
0	A0	0.18635	att C0
0.01	A1	-0.0052	att C1
0	A2	0.00012	att C2
0	A3		

Help message below explains how Analysis would allow saving these properties of the liquid material after they measured with acoustic sensor.

**Dispersion Technology DT1200**

Intrinsic attenuation using DT1200 acoustic sensor. Analysis software has provision for saving these parameters to the database after measurement with pure liquid is finished. We suggest to run 3 measurements and use the second or the third. Results of the first one could be affected because of unknown sound speed, which becomes known after the first measurement has finished. Software would not allow using this material unless at least value of the box attA1 has been changed, or intrinsic properties will be measured and stored using Analysis. For newtonian liquid it is attenuation at 100 MHz divided by 100. Attenuation is defined as product of frequency function by temperature function

OK

When you create material for emulsion droplets you have possibility to distinguish between emulsions and microemulsions. If you start name of the material with word “emulsion” as shown below than Analysis software will search only for sizes above 0.5 microns when this material is used for sample definition.

If you want to modify existing material, then you should select it from the combo box that contains all materials in the Material database, EXCEPT so-called standard material that are not allowed to be modified. For instance, you are not allowed to modify properties of “water”.

You can change value of any parameter and save it.

However, there is protection against using unrealistic values. It is built into the software, which would not allow user to save values outside of reason ranges.

You can see all material properties that are relevant to our measuring techniques. Goto View, all properties. They are shown on Figure below.

The screenshot shows a software window titled "New Material" with a "View" button in the top right corner. The window is divided into two main sections: "Create new material" on the left and "Specify property value" on the right.

**Create new material:** This section includes instructions: "Select material type, modify properties, type new material name and click SaveAs". It features a list of material types with radio buttons:

- solid rigid particle
- porous particle
- large particles, >10 micron
- soft particles (latex)
- aqueous medium for solid particles** (selected)
- emulsion medium (oil)
- emulsion droplet (oil)

Below the list are "SaveAs" and "Exit" buttons, and a text input field for the material name.

**Specify property value:** This section has a green background and a "Click Caption for HELP" link. It contains several input fields with corresponding property names:

- material type: 4
- density in g/cm.cu: 0.997039
- scattering coefficient only for size > 3 micron: 1
- viscosity in cP: 0.8904
- dielectric permittivity, relative: 78.85
- heat capacity, Cp in J/g/C: 4.179613
- heat conductivity in mW/cm/C: 6.0648
- thermal expansion in 1/C 10E-4: 2.57
- fractal number /: 3
- sound speed m/sec: 1496.7

Below these is a section titled "Intrinsic attenuation of liquid" with two columns: "frequency expansion" and "T expansion".

frequency expansion		T expansion	
0	A0	0.18635	att C0
0.01	A1	-0.0052	att C1
0	A2	0.00012	att C2
0	A3		

All material properties are temperature dependent. We have temperature correction know from literature for standard material built into the Material database. User can insert such corrections for the new material if he knows them. Goto to View, T corrections.

**New Material** [X]

View

**Specify property value**  
Click Caption for HELP

4 material type

0.785 density in g/cm.cu

1 scattering coefficient  
only for size > 3 micron

1.096413 viscosity in cP

24.3 dielectric permittivity, relative

2.47 heat capacity, Cp in J/g/C

1.71 heat conductivity in mW/cm/C

8.4 thermal expansion in 1/C 10E-4

fractal number /

1207 sound speed m/sec

*Intrinsic attenuation of liquid*

frequency expansion	T expansion
0 A0	0.42 att C0
0.01 A1	0 att C1
0 A2	0 att C2
0 A3	

**Temperature Correction**

If you do not know T correction, default 0 value would be saved to the database

All properties assume the same polinomial expansion around reference T=25C

$$C0 + C1(T-25) + C2(T-25)^2$$

	C1	C2
density	-0.00	0
viscosity	-0.01	0.0003
diel.per.	-0.27	0
Cp	0	0
heat cond.	-0.00	0.0000
th. expansion	0	0
s.speed	0	0

**COMMENT**

**Modify existind material**  
Select material name, modify property and click Save

Save Cancel

ethano

There is a very detail program that presents T dependences of various parameters graphically. It is called Material Wizard. It is available from Home page, Tools, Options.

## Storing numerical data in the database and excel files

All results are automatically saved in the MS Access database located in the C/DATA\_ACO as **data\_aco.mdb** file. Structure of this file and related tools are discussed in the two following sections.

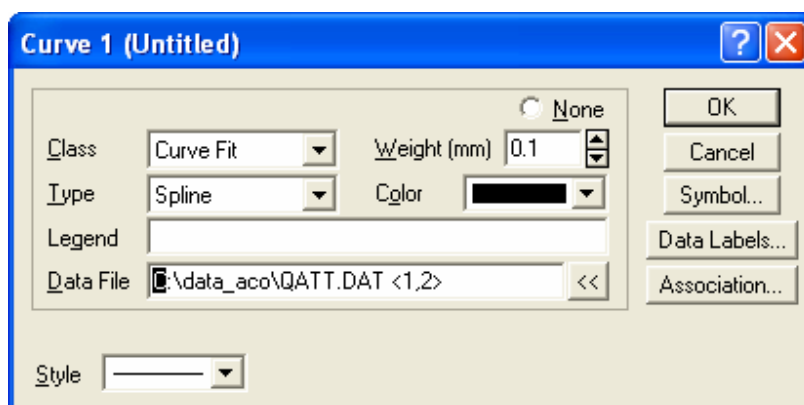
In addition several programs could create excel files for particular purposes.

**Analysis** program has File menu items allowing saving excel files for the last single measurement treated with Analysis. These files are saved to the directory C/data\_aco/Excel. They can be saved automatically or with different names.

**On-Line Continuous data** program saves data shown on graphs located on this program to file c/data\_aco/report-continuous.csv. It re-writes this file every time when File. Save to Excel file menu item clicked. This file contains column formatted in time units, which is very convenient for plotting kinetic curves.

**Titration Control** allows saving of the last made titration to two files: C/data\_aco/titdata.csv that contains data on zeta potential conductivity, pH etc, and c/data\_aco/titcurve.csv, which contains data on pH versus meq of the added chemical. The first file corresponds to the top Graph on the Titration Control form, the second one to the bottom Graph. These files become overwritten each time Save command is triggered.

**Graph overlay** program creates several excel files for selection of records and graph types chosen by user. Each curve has X and Y values saved in one of this files. User can click on any of the curves and small form appears as showing below.



This form contains line Data File. It specifies data file where X and Y values for this curve are stored and numbers of columns. User can open this file with excel and retrieve these numbers.

### *Database*

User can open this file using standard Access interface. Data is organized in several tables. The simplest way to browse through the data is using “queries” instead of tables. Query is selection of the records based on certain criteria. There are several pre-defined queries based on date of measurement, such as ~Today, ~Today and Yesterday, ~Last 7 days, ~Last year, etc. User can create his own Query using Access Interface.

Several DTI software programs are linked to the queries. **Analysis** and **Graph** are linked in default either to ~!Today query, if any measurements are made today. Alternatively they are linked to ~Demo Query, which contains a set of records illustrating main features of these methods. These programs has options of being re-connected to any other Query.

Program **On-Line Continuous data** is also linked to query, such as ~Last Experiment or ~Today. This link can be also changed.

Instead of using MS Access interface we have developed our own interface program called **Data View**, which can be start from Home page, View. Next section presents detail description of this interface.

Sometimes it is useful to know meaning of particular field in the database. Table below presents this meaning and field format for all fields in the database.

<b>Name</b>	<b>Meaning</b>	<b>Format</b>
wf	weight fraction, as %. Do not use for correcting existing record	<i>Number (Single)</i>
Chemical name	optional sample ID info entered on Meas Form	<i>Text</i>
C1	measured CVI magnitude at first freq, for zp using probe design	<i>Number (Double)</i>
SampleTxt	short definition of the sample created from user-entered info	<i>Text</i>
Measurement Date	date and time of the measurement	<i>Date/Time</i>
PSM	median unimodal size.(but not for modified unimodal. See Book)	<i>Number (Single)</i>
PSW	standard deviation of unimodal PSD	<i>Number (Single)</i>
zp	zeta potential, mV	<i>Number (Single)</i>
err_th_con	CVI phase, with theoretical correction, determines sign of zeta	<i>Number (Double)</i>
pH	pH	<i>Number (Single)</i>
V1	measured sound speed from time of flight, m/s	<i>Number (Single)</i>
C2	measured CVI magnitude at second frequency, for zp using split sensor	<i>Number (Double)</i>
K1	conductivity, S/m	<i>Number (Single)</i>
Temp	temperature, degrees Celcius	<i>Number (Single)</i>
Medium	name of the liquid	<i>Text</i>
Experiments.System ID	system ID	<i>Number (Long)</i>
Experiments.Sample Date	date and time when sample is defined	<i>Date/Time</i>
Dispersed Phase	name of the particle material	<i>Text</i>
Sample id	*****	<i>Text</i>
ExpTxt	*****	<i>Text</i>
MeasTxt	*****	<i>Text</i>
measurements.System ID	serial number of system	<i>Number (Long)</i>
measurements.Experiment Date	date and time when experiment started	<i>Date/Time</i>
Analysis Cond Date	*****	<i>Number (Integer)</i>
Density	density of particles material, kg/m3. Use when correcting existing record	<i>Number (Double)</i>
Part_S_Speed	sound speed in the particle, m/sec	<i>Number (Double)</i>
Weight_Fraction	weight fraction (not in %). Use when correcting existing record	<i>Number (Double)</i>
K2	zeta potential with Du and MW corrections calculated with conductivity	<i>Number (Single)</i>
K3	std deviation of unimodal PSD, for calculating zeta potential	<i>Number (Single)</i>
K4	th.phase for Du=0 and no MW correction	<i>Number (Single)</i>
K5	ka, ratio of the particle radius to the Debye length	<i>Number (Single)</i>
K6	Debye length, microns	<i>Number (Single)</i>
K7	dynamic mobility, micron/sec/V/cm	<i>Number (Single)</i>
K8	phase angle of complex conductivity, degrees	<i>Number (Single)</i>
K9	Imaginary part of complex conductivity, S/m	<i>Number (Single)</i>
K10	Magnitude of complex conductivity, S/m	<i>Number (Single)</i>
K11	Maxwell-Wagner frequency, MHz	<i>Number (Single)</i>
K12	Du calculated using measured conductivity	<i>Number (Single)</i>
K13	diffusion coefficient for calculating zeta in non-aqueous, *10E-5. In cm2/sec	<i>Number (Single)</i>
K14	zeta potential, for non-aqueous media	<i>Number (Single)</i>
K15	zeta potential, for Du=0 but MW freq from K	<i>Number (Single)</i>
K16	zeta potential phase for Du=0 but MW from K	<i>Number (Single)</i>
K17	CVI gap, m	<i>Number (Single)</i>

K18	CVI Phase size	<i>Number (Single)</i>
K19	surface charge for non-aqueous, microCoulombs/cm square	<i>Number (Single)</i>
K20	th.phase for non-aqueous	<i>Number (Single)</i>
K21	th.phase DU and MW taken into account	<i>Number (Single)</i>
E1	measured CVI phase at frequency #1 for probe	<i>Number (Single)</i>
E2	measured CVI phase at frequency #2 for probe	<i>Number (Single)</i>
E3	measured CVI phase at frequency #3 for probe	<i>Number (Single)</i>
E4	measured CVI phase at frequency #4 for probe	<i>Number (Single)</i>
E5	d10log	<i>Number (Single)</i>
E6	d16log	<i>Number (Single)</i>
E7	d50log	<i>Number (Single)</i>
E8	d86log	<i>Number (Single)</i>
E9	d90log	<i>Number (Single)</i>
E10	d10bi	<i>Number (Single)</i>
E11	d16bi	<i>Number (Single)</i>
E12	d50bi	<i>Number (Single)</i>
E13	d86bi	<i>Number (Single)</i>
E14	d90bi	<i>Number (Single)</i>
E15	surface charge for non-aqueous, per particle	<i>Number (Single)</i>
E16	calibration constant for different Analysis strategies	<i>Number (Single)</i>
E17	added reagent volume, ml	<i>Number (Single)</i>
E18	added meq	<i>Number (Single)</i>
E19	total sample volume, ml	<i>Number (Single)</i>
E20	scattering adjustment resonance parameter	<i>Number (Single)</i>
E21	CVI Phase correction for frequency #2 for split sensor	<i>Number (Single)</i>
C3	measured CVI magnitude at frequency #3	<i>Number (Double)</i>
C4	measured CVI magnitude at frequency #4	<i>Number (Double)</i>
C5	measured CVI magnitude at frequency #5	<i>Number (Double)</i>
C6	measured CVI magnitude at frequency #6 for probe, CVI phase for freq #1 for split sensor	<i>Number (Double)</i>
C7	measured CVI magnitude at frequency #7 for probe, CVI phase for freq #2 for split sensor	<i>Number (Double)</i>
C8	measured CVI magnitude at frequency #8 for probe, CVI phase for freq #3 for split sensor	<i>Number (Double)</i>
C9	measured CVI magnitude at frequency #9 for probe, CVI phase for freq #4 for split sensor	<i>Number (Double)</i>
C10	measured CVI magnitude at frequency #10 for probe, CVI phase for freq #5 for split sensor	<i>Number (Double)</i>
C11	measured CVI magnitude at frequency #11	<i>Number (Double)</i>
A1	attenuation at frequency #1	<i>Number (Double)</i>
A2	attenuation at frequency #2	<i>Number (Double)</i>
A3	attenuation at frequency #3	<i>Number (Double)</i>
A4	attenuation at frequency #4	<i>Number (Double)</i>
A5	attenuation at frequency #5	<i>Number (Double)</i>
A6	attenuation at frequency #6	<i>Number (Double)</i>
A7	attenuation at frequency #7	<i>Number (Double)</i>
A8	attenuation at frequency #8	<i>Number (Double)</i>
A9	attenuation at frequency #9	<i>Number (Double)</i>
A10	attenuation at frequency #10	<i>Number (Double)</i>
A11	attenuation at frequency #11	<i>Number (Double)</i>
A12	attenuation at frequency #12	<i>Number (Double)</i>
A13	attenuation at frequency #13	<i>Number (Double)</i>
A14	attenuation at frequency #14	<i>Number (Double)</i>
A15	attenuation at frequency #15	<i>Number (Double)</i>
A16	attenuation at frequency #16	<i>Number (Double)</i>
A17	attenuation at frequency #17	<i>Number (Double)</i>
A18	attenuation at frequency #18	<i>Number (Double)</i>
A19	attenuation at frequency #19	<i>Number (Double)</i>
A20	attenuation at frequency #20	<i>Number (Double)</i>
A21	attenuation at frequency #21	<i>Number (Double)</i>

N1	attenuation error at frequency #1	Number (Double)
N2	attenuation error at frequency #2	Number (Double)
N3	attenuation error at frequency #3	Number (Double)
N4	attenuation error at frequency #4	Number (Double)
N5	attenuation error at frequency #5	Number (Double)
N6	attenuation error at frequency #6	Number (Double)
N7	attenuation error at frequency #7	Number (Double)
N8	attenuation error at frequency #8	Number (Double)
N9	attenuation error at frequency #9	Number (Double)
N10	attenuation error at frequency #10	Number (Double)
N11	attenuation error at frequency #11	Number (Double)
N12	attenuation error at frequency #12	Number (Double)
N13	attenuation error at frequency #13	Number (Double)
N14	attenuation error at frequency #14	Number (Double)
N15	attenuation error at frequency #15	Number (Double)
N16	attenuation error at frequency #16	Number (Double)
N17	attenuation error at frequency #17	Number (Double)
N18	attenuation error at frequency #18	Number (Double)
N19	number of records in the measurement table	Number (Double)
N20	attenuation error at frequency #20	Number (Double)
N21	attenuation error at frequency #21	Number (Double)
V2	frequency of sound speed measurement	Number (Single)
V3	relaxed low frequency sound speed, Wood eq.	Number (Single)
V4	s.speed calculated for unimodal psd	Number (Single)
V5	s.speed calculated for bimodal psd	Number (Single)
V6	error of the Newtonian fit, %	Number (Single)
V7	frequency number at which s.speed measured	Number (Single)
V8	primary size for fractals	Number (Single)
V9	fractal density	Number (Single)
V10	fractal dimension	Number (Single)
V11	elastic modulus	Number (Single)
V12	compressibility	Number (Single)
V13	number of points out of Newtonian fit range (>2 nonNewt)	Number (Single)
V14		Number (Single)
V15	zeta for calculating vfr from CVI	Number (Single)
V16	d50 for calculating vfr from CVI	Number (Single)
V17	stdev for calculating vfr from CVI	Number (Single)
V18	precision of Newtonian test [dB/cm/Mhz]	Number (Single)
V19	experimental phase s.speed	Number (Single)
V20	volume fraction from attenuation	Number (Single)
V21	volume fraction	Number (Single)
VN1	triple psd, size 1	Number (Double)
VN2	triple psd, size 2	Number (Double)
VN3	triple psd, size 3 (d90)	Number (Double)
VN4	triple psd, stdev1	Number (Double)
VN5	triple psd, stdev2	Number (Double)
VN6	triple psd, stdev3	Number (Double)
VN7	triple psd, fr1	Number (Double)
VN8	triple psd, fr2	Number (Double)
VN9	triple psd, fr3 (20% for milling)	Number (Double)
VN10	triple psd fitting error	Number (Double)
VN11	volume viscosity [cP]	Number (Double)
VN12	extensional viscosity at 10 MHz [cP]	Number (Double)
VN13	extensional viscosity at 100 MHz [cP]	Number (Double)
VN14	*****	Number (Double)
VN15	online attenuation at selected frequency (A15)	Number (Double)
VN16	online polynomial coef(0) to fit attenuation spectra over selected freq range	Number (Double)
VN17	online polynomial coef(1) to fit attenuation spectra over selected freq range	Number (Double)
VN18	online polynomial coef(2) to fit attenuation spectra over selected freq range	Number (Double)



VN19	online polynomial coef(3) to fit attenuation spectra over selected freq range	Number (Double)
VN20		Number (Double)
VN21	Hookean coefficient in structure search	Number (Double)
PSMBi	bimodal d50	Number (Double)
PSM_1	bimodal median of the mode 1	Number (Double)
PSM_2	bimodal median of the mode 2, larger size	Number (Double)
PSMM	monodisperse size	Number (Double)
PSMN	mean size, either bi or lo depending on program selection	Number (Double)
PSMW		Number (Double)
PSWBi	average bimodal	Number (Double)
PSW_1	bimodal, st.dev mode 1	Number (Double)
PSW_2	bimodal, st.dev mode 2, larger size	Number (Double)
Cont_1	*****	Number (Double)
Cont_2	weight fraction of the mode 2 relative to the total wfr	Number (Double)
SHELL	shell of the unimodal	Number (Double)
SHELLBi	shell of the bimodal	Number (Double)
SHELL_1	error subtracted from err-log for selecting it over bimodal	Number (Double)
SHELL_2	error of the fit produced by triple psd	Number (Double)
ERR_EXP	experimental error	Number (Double)
ERR_MONO	error of the fit produced by monodisperse assumption	Number (Double)
ERR_LOG	error of the fit produced by unimodal assumption	Number (Double)
ERR_BI	error of the fit produced by bimodal assumption	Number (Double)
ERR_AVE	average theoretical error	Number (Double)
min_size	minimum size defined by Analysis for unimodal PSD	Number (Double)
max_size	maximum size defined by Analysis for unimodal PSD	Number (Double)
min_size_mix	minimum size for global search if E16=1	Number (Double)
max_size_mix	maximum size for global search if E16=1	Number (Double)
F_low	lowest frequency for attenuation measurement	Number (Double)
F_high	highest frequency for attenuation measurement	Number (Double)
N_exp	number of frequencies used for attenuation measurement	Number (Double)
Lin_exp	scale for attenuation measurements, 1 - log, 0 - linear	Number (Double)
F_min	minimum frequency for theoretical fit defined by Analysis	Number (Double)
F_max	maximum frequency for theoretical fit defined by Analysis	Number (Double)
D15	triple mixture, %free mode 1	Number (Double)
D50	triple mixture, %free mode 2	Number (Double)
D85	triple mixture, size mode 3	Number (Single)
SQR(D85/D16)	triple mixture, stdev mode3	Number (Double)
F_z_min	*****	Number (Double)
F_z_max	*****	Number (Double)
F_c_low	lowest frequency for CVI measurement	Number (Double)
F_c_high	highest frequency for CVI measurement	Number (Double)
F_c_min	*****	Number (Double)
F_c_max	*****	Number (Double)
a_zeta	unimodal median size for zeta calculation	Number (Double)
K0	*****	Number (Single)
Rel	*****	Number (Double)
numfreq_c	number of frequencies for CVI measurement	Number (Double)
Us1	*****	Text
Us2	*****	Text
Us3	*****	Text
rawdata	raw data for attenuation measurement, optional	Memo
Experiments.Experiment Date	*****	Date/Time
Configuration Date	*****	Date/Time
Protocol Date	*****	Date/Time
Calibration Date	*****	Date/Time
SetUp Date	*****	Date/Time
View Date	*****	Date/Time
Samples.System ID	*****	Number (Long)
Samples.Sample Date	*****	Date/Time

Category	*****	Text
Dimension	*****	Number (Integer)
Class	*****	Text
Product Type	*****	Text
Use code	*****	Text
Industry code	*****	Text
Formula	*****	Text
Manufacturer	*****	Text
Trade Name	*****	Text
Method	*****	Text
Notes	*****	Memo
viscosity	*****	Number (Double)
vp	*****	Number (Double)
Extra Dispersed Phase	*****	Text
wf extra	weight fraction of the extra dispersed phase	Number (Double)
Used	*****	Number (Double) (uble)

## Viewing numerical data using Data View window

To view data in table form:

Click **View > Data** on the Home page.

The Data View window will appear similar to that shown below.

wt% Disperse Phase	xw% Ext Dis	Medium	ID	Experiment Date	Titr Prot	T Prot	N	Measurement Date	Temp	pH	K1	Cvi	zp	CviPha	zSize	V1	PSM	PSW
10.0	silica, Ludox	0.0	none	water				2006/06/27 22:57:56	26.8	5.6	0.000	3.813423	-37.4	180.01	0.010	1490.6	0.100	0.300
10.0	silica, Ludox	0.0	none	water				2006/06/27 22:58:22	26.8	5.6	0.000	3.826509	-37.5	180.27	0.037	1490.6	0.100	0.300
10.0	silica, Ludox	0.0	none	water				2006/06/27 22:58:46	26.8	5.6	0.000	3.834362	-37.6	179.89	0.000	1490.6	0.100	0.300
10.0	silica, Ludox	0.0	none	water				2006/06/27 22:59:10	26.8	5.6	0.000	3.851272	-37.8	180.14	0.026	1490.6	0.100	0.300
10.0	silica, Ludox	0.0	none	water				2006/06/27 22:59:35	26.8	5.6	0.000	3.832092	-37.6	179.96	0.000	1490.6	0.100	0.300
10.0	silica, Ludox	0.0	none	water				2006/06/27 23:00:02	26.8	5.6	0.000	3.821686	-37.5	179.95	0.000	1490.6	0.100	0.300
10.0	silica, Ludox	0.0	none	water	Silica ludox in p			2006/06/27 23:26:45	26.8	5.7	0.000	0.00	0.0	0.00	0.000	1495.1	0.035	0.133
10.0	silica, Ludox	0.0	none	water	Silica ludox in p			2006/06/27 23:35:07	26.9	5.7	0.000	0.00	0.0	0.00	0.000	1495.2	0.036	0.132
10.0	silica, Ludox	0.0	none	water	Silica ludox in p			2006/06/27 23:44:24	27.1	5.7	0.000	0.00	0.0	0.00	0.000	1495.0	0.035	0.142
10.0	silica, Ludox	0.0	none	water	Silica ludox in p			2006/06/28 00:24:29	27.3	5.7	0.000	0.00	0.0	0.00	0.000	1496.8	0.036	0.166
10.0	silica, Ludox	0.0	none	water	Silica ludox in p			2006/06/28 00:33:50	27.4	5.7	0.000	0.00	0.0	0.00	0.000	1496.4	0.035	0.163
10.0	silica, Ludox	0.0	none	water	Silica ludox in p			2006/06/28 00:43:29	27.5	5.7	0.000	0.00	0.0	0.00	0.000	1496.7	0.035	0.163
10.0	silica, Ludox	0.0	none	water	Silica ludox in p			2006/06/28 00:52:50	27.5	5.7	0.000	0.00	0.0	0.00	0.000	1497.0	0.035	0.162
10.0	silica, Ludox	0.0	none	water	Silica ludox in p			2006/06/28 01:02:14	27.6	5.7	0.000	0.00	0.0	0.00	0.000	1497.1	0.034	0.161
0.0	none	0.0	none	water	Distilled water ft			2006/06/28 01:51:15	25.9	5.7	0.000	0.00	0.0	0.00	0.000	1499.8	0.000	0.000
0.0	none	0.0	none	water	Distilled water ft			2006/06/28 02:07:24	26.4	5.8	0.000	0.00	0.0	0.00	0.000	1499.7	0.000	0.000
0.0	none	0.0	none	water	Distilled water ft			2006/06/28 02:14:27	26.9	5.8	0.000	0.00	0.0	0.00	0.000	1482.6	0.000	0.000
0.0	none	0.0	none	water	Distilled water ft			2006/06/28 02:35:49	27.2	5.8	0.000	0.00	0.0	0.00	0.000	1501.0	0.000	0.000
10.0	silica, Ludox	0.0	none	water	lg silica in polym			2006/06/28 02:40:08	27.4	5.9	0.000	0.00	0.0	0.00	0.000	1510.9	10.823	0.400
10.0	silica, Ludox	0.0	none	water	lg silica in polym			2006/06/28 02:58:44	27.7	5.9	0.000	0.00	0.0	0.00	0.000	1510.9	10.823	0.400
10.0	silica, Ludox	0.0	none	water	lg part in gel, 0.1			2006/06/28 03:16:45	27.7	5.9	0.000	0.00	0.0	0.00	0.000	1511.6	10.829	0.450
10.0	silica, Ludox	0.0	none	water	lg part in gel, 0.1			2006/06/28 03:36:18	27.9	5.9	0.000	0.00	0.0	0.00	0.000	1511.6	10.829	0.450
10.0	silica, Ludox	0.0	none	water	lg part in gel, 0.1			2006/06/28 03:49:52	28.0	5.9	0.000	0.00	0.0	0.00	0.000	1512.1	12.945	0.150
10.0	silica, Ludox	0.0	none	water	lg part in gel, 0.1			2006/06/28 04:02:32	28.0	6.0	0.000	0.00	0.0	0.00	0.000	1512.6	14.249	0.350
10.0	silica, Ludox	0.0	none	water	lg part in gel, 0.1			2006/06/28 04:15:34	28.1	6.1	0.000	0.00	0.0	0.00	0.000	1511.2	13.038	0.400
10.0	silica, Ludox	0.0	none	water	lg part in gel, 0.1			2006/06/28 04:28:24	28.1	6.2	0.000	0.00	0.0	0.00	0.000	1511.7	13.842	0.250

22 Experiments, involving altogether 191 measurements, are contained in this Query

If measurements were made today, that data will be displayed by default.

However, if no measurements were made today, by default the data window opens using the shortest time period (a week, a month, a year, whatever time period is required) to contain at least one measurement. In the example shown, the data is displayed for the past month as displayed in the caption at the top. The number of experiments found during this time period is displayed on the Status bar at the bottom of the form. In this case 22 experiments were found in the past month involving altogether 191 measurements.

The particular data item in each column is described in the heading at the top that column. The definition of the data in each column is given in the Status Bar at the bottom of the form when

any cell in that column is clicked with the mouse. Take a minute to examine the definition of several columns in the table in this way.

The data in the first six columns describe features of the measured sample.

The data in the next four columns describe the experiment performed on that sample.

The remaining columns contain measurement data and analyses of those data.

The width of any column can be changed by placing the cursor half way between headings in the first row of the table till a double arrow appears and then moving the cursor to provide the column width desired. These width settings are only temporary.

In the example shown, note that five measurements were performed in the last experiment at 03:20:28 (at the bottom of the form) , whereas in the previous experiment at 02:58:44 only one measurement was made. The nature of the Titration Protocol is displayed in the Titr Prot column, whereas any temperature titration experiment is described in the T Prot column.

The five measurements taken in the last experiment can be collapsed into a single row by clicking on the small box having a – inside located at the left of the Experiment Date column. The – changes to a + when the rows are collapsed, in much the same manner as for a directory tree if one uses Windows Explorer to select files.

wt%	Disperse Phase	xwt%	Ext Disp Phase	Medium	Mfr	Trade	ID	Experiment Date	Titr Prot	T Prot	N	Temp	pH	K1	Cvi	zp	CviPha	z
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox	10201CD	02/15/2006 10:19:45	none,1	none		21.0	7.0		2,172,965	-23.9	202.90	0.
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox	10201CD	02/15/2006 10:24:43	time,11	none								
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox	10201CD	02/15/2006 10:52:25	time,3	none		21.4	7.0					
												21.6	7.0					
												21.7	7.0					
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox	10201CD	02/15/2006 12:21:07	time,5	none,		22.9	7.0	4,392,738	-46.5	179.47	0.	
												22.9	7.0	4,394,320	-46.5	179.17	0.	
												22.9	7.0	4,400,473	-46.6	179.32	0.	
												22.9	7.0	4,376,673	-46.3	179.06	0.	
												22.9	7.0	4,375,140	-46.3	178.86	0.	
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox	10201CD	02/15/2006 12:25:14	none,	none,		23.0	7.0	4,395,132	-46.2	179.19	0.	
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox	10201CD	02/15/2006 12:25:59	none,	none,		23.0	7.0	3,628,048	-38.1	180.72	0.	
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox	10201CD	02/15/2006 13:03:56	none,	none,		22.7	9.3	3,496,654	-37.0	180.89	0.	
												22.8	9.4	3,536,273	-37.3	180.71	0.	
												22.8	9.4	3,514,448	-37.1	180.75	0.	
												23.0	8.9	3,394,018	-35.7	181.85	0.	
												23.1	8.5	3,200,717	-33.5	182.57	0.	
												23.2	8.1	2,965,980	-31.0	183.52	0.	
												23.2	7.6	2,660,383	-27.8	183.79	0.	
												23.2	7.3	2,367,910	-24.8	184.87	0.	
												23.2	6.8	1,990,700	-20.8	185.45	0.	
												23.2	6.4	1,621,382	-17.0	186.51	0.	
												23.2	6.0	1,261,080	-13.2	186.95	0.	
												23.2	5.6	950,862	-9.9	187.99	0.	

This collapsing can be reversed by clicking on the + box for the same experiment. This feature of collapsing and expanding data is particularly useful when large numbers of measurements are made as part of a single experiment. (For example a long time-series experiment or a very detailed titration experiment.

### Finding specific data

In day-to-day measurements the data shown by default will often be quite appropriate for the task at hand. However, when one wants to review historical records or see only specific results it is possible to query the database for specific data. To define a specific Query, click on the Query command button (or select **View – Query Definition** from the menu). The Query definition then appears near the top of the form as shown in the figure below.

wt%	Disperse Phase	xwt%	Ext Dis	Medium	ID	Experiment Date	Temp	pH	K1	Cvi	zp	CviPha	zSize	V1	PSM	PSW
10.0	silica_Ludox	0.0	none	water		2006/06/27 22:55:25										
10.0	silica_Ludox	0.0	none	water	Silica ludox in p	2006/06/27 23:16:24	time,1,11									
10.0	silica_Ludox	0.0	none	water	Silica ludox in p	2006/06/28 00:11:27	time,1,11									
0.0	none	0.0	none	water	Distilled water (r	2006/06/28 01:25:22	none									
0.0	none	0.0	none	water	Distilled water (r	2006/06/28 01:51:47	none									
0.0	none	0.0	none	water	Distilled water (r	2006/06/28 02:08:24	none									
0.0	none	0.0	none	water	Distilled water (r	2006/06/28 02:18:17	none									
10.0	silica_Ludox	0.0	none	water	lg silica in polym	2006/06/28 02:40:08	none									
10.0	silica_Ludox	0.0	none	water	lg silica in polym	2006/06/28 02:58:44	none									
10.0	silica_Ludox	0.0	none	water	lg part in gel, 01	2006/06/28 03:20:28	time,1,11									

The Query definition allows one to select data according to nine criteria as detailed in the nine combo boxes shown in the Query definition frame.  
 As an example of using the Query definition:

- Select a time period of **10 yrs** using the first combo box at the left.
- Select disperse phase **Alumina\_alpha**
- Click **GO**

The results of this query is shown in the figure below. The status bar at the bottom of the form shows that 56 experiments involving 129 measurements were found. Some of these experiments involved not one but several measurements. Your results will be different depending on what experiments have been performed on your system.

wt%	Disperse Phase	xwt%	Ext Dis	Medium	ID	Experiment Date	Titr Prot	T Prot	N	Measurement Date	Temp	pH	K1	Cvi	zp	CviPha	zSize	V1	PSM	PSW
3.5	alumina_alpha	0.0	none	water	ATK #1	2005/07/26 13:15:12				2005/07/26 13:22:54	24.8	6.0	0.091	1.153154	39.9	5.82	0.141	1492.5	0.646	0.593
3.5	alumina_alpha	0.0	none	water	ATK #1	2005/07/26 13:37:23				2005/07/26 13:44:58	24.3	1.4	1.283	1.407268	52.9	38.69	2.132	1492.1	0.776	0.579
3.5	alumina_alpha	0.0	none	water	ATK #1	2005/07/26 13:51:59				2005/07/26 13:59:29	24.5	1.4	1.264	1.441070	54.8	39.53	2.374	1492.4	0.803	0.590
4.1	alumina_alpha	0.0	none	water	ATK #2	2005/07/26 14:06:53				2005/07/26 14:16:30	24.1	5.0	0.093	2.199485	56.5	357.28	0.000	1491.3	0.505	0.389
4.1	alumina_alpha	0.0	none	water	ATK #2	2005/07/26 14:18:38				2005/07/26 14:26:48	24.3	5.0	0.090	2.221873	57.0	357.25	0.000	1492.0	0.507	0.386
4.1	alumina_alpha	0.0	none	water	ATK #2	2005/07/26 14:28:37				2005/07/26 14:42:59	24.5	3.6	0.100	2.243286	57.3	7.85	0.178	1491.1	0.522	0.461
4.1	alumina_alpha	0.0	none	water	ATK #2	2005/07/26 14:46:04				2005/07/26 14:54:25	24.5	4.0	0.102	2.150767	55.0	6.32	0.152	1492.0	0.525	0.461
4.1	alumina_alpha	0.0	none	water	ATK #2	2005/07/26 15:07:30				2005/07/26 15:15:50	24.8	4.3	0.104	2.098507	53.3	6.51	0.154	1492.4	0.504	0.391
1.4	alumina_alpha	0.0	none	water	ATK #3	2005/07/26 15:30:54				2005/07/26 15:37:47	26.9	4.8	0.088	822.067	52.6	368.38	0.000	1504.7	0.323	0.429
1.4	alumina_alpha	0.0	none	water	ATK #3	2005/07/26 15:40:21				2005/07/26 15:47:16	27.6	4.9	0.087	811.177	52.5	358.99	0.000	1501.9	0.315	0.428
1.4	alumina_alpha	0.0	none	water	ATK #3	2005/07/26 16:04:42				2005/07/26 16:15:09	26.4	4.2	0.095	841.337	55.3	358.62	0.000	1499.3	0.312	0.431
1.4	alumina_alpha	0.0	none	water	ATK #3	2005/07/26 16:33:20				2005/07/26 16:40:52	26.1	4.3	0.092	832.443	54.9	358.27	0.000	1498.7	0.312	0.424
3.3	alumina_alpha	0.0	none	water	ATK #4	2005/07/26 17:26:42				2005/07/26 16:48:07	26.0	4.2	0.096	837.498	55.4	358.23	0.000	1497.7	0.315	0.419
3.3	alumina_alpha	0.0	none	water	ATK #4	2005/07/26 17:36:09				2005/07/26 17:35:07	26.9	5.3	0.090	2.288469	54.5	350.41	0.000	1495.1	0.255	0.322
3.3	alumina_alpha	0.0	none	water	ATK #4	2005/07/26 17:51:23				2005/07/26 17:44:39	26.5	5.3	0.088	2.181334	52.0	351.10	0.000	1494.6	0.252	0.320
3.3	alumina_alpha	0.0	none	water	ATK #4	2005/07/26 18:05:11				2005/07/26 17:52:26	26.1	5.5	0.085	2.256726	47.2	351.42	0.000	1484.7	0.100	0.300
3.3	alumina_alpha	0.0	none	water	ATK #4	2005/07/26 18:05:48				2005/07/26 18:05:11	25.9	2.3	0.267	2.587033	63.3	13.36	0.268	1491.8	0.257	0.342
3.3	alumina_alpha	0.0	none	water	ATK #4	2005/07/26 18:05:59				2005/07/26 18:14:26	25.8	2.3	0.259	2.629166	64.6	12.78	0.256	1492.7	0.257	0.347
10.0	alumina_alpha	0.0	none	water	redgftku	2005/08/09 14:55:16				2005/08/09 15:03:13	21.2	5.4	0.211	11.002210	81.3	0.48	0.071	1425.8	0.034	0.217
10.0	alumina_alpha	0.0	none	water	redgftku	2005/08/10 11:44:45				2005/08/10 11:45:08	20.4	5.6	0.000	10.121740	77.0	0.64	0.044	1459.5	0.100	0.300
10.0	alumina_alpha	0.0	none	water	redgftku	2005/08/10 11:45:33				2005/08/10 11:45:55	20.5	5.6	0.000	10.152790	77.1	0.43	0.036	1459.5	0.100	0.300
10.0	alumina_alpha	0.0	none	water	redgftku	2005/08/10 11:46:10				2005/08/10 11:46:43	20.5	5.6	0.195	10.241070	77.8	0.33	0.064	1459.5	0.100	0.300
10.0	alumina_alpha	0.0	none	water	redgftku	2005/08/10 11:47:04				2005/08/10 11:56:24	20.5	5.6	0.232	10.475760	78.5	0.24	0.068	1425.2	0.036	0.178
10.0	alumina_alpha	0.0	none	water	redgftku	2005/08/10 12:20:17				2005/08/10 12:20:39	19.4	5.6	0.000	6.489407	50.5	358.30	0.000	1459.5	0.100	0.300
10.0	alumina_alpha	0.0	none	water	redgftku	2005/08/10 12:20:49				2005/08/10 12:21:11	19.4	5.6	0.000	6.574919	51.1	357.99	0.000	1459.5	0.100	0.300

Notice that the heading at the top of the form has changed to describe more accurately the data currently found in response to your query.  
 You may have noticed that the available selections for media and dispersed phase materials is quite a bit shorter than the total list of all materials supplied in the Material Table in the database.

This is because the materials enumerated in the Query box only include materials that have actually been used on your system for actual measurements. The same thing is true for all of the other query boxes, only manufacturers, trade names, sample ID, users, and projects that have been referenced in your experiments will be included in the available choices.

So far we have selected data using the Time period and the disperse phase. But you can use any of the nine query boxes to define your search criteria. For practice, try selecting data based on one or more of the other query boxes.

When you log on to the instrument you will have an opportunity to identify yourself and to note a particular project you are working on. You can later retrieve data using these two properties. The initials of the user making each experiment and the project being worked on are also available for querying the database using the User and Project Query boxes.

Each system has a unique serial number which is also recorded for each experiment. Normally all of the data stored in your database will be obtained on your own system. However, it is possible to exchange data between systems and in this case your database might include experiments from more than one system. If your instrument has data from more than one system, then the Query definition frame will automatically include a Query box labeled "System".

### Reading and revising comments in the data

wt%	Disperse Phase	xwt%	Ext Dis	Medium	ID	Experiment Date	Tit Prot	T Prot	N	Measurement Date	Temp	pH	K1	Cvi	zp	CvPpha	zSize	V1	PSM	PSW
4.8	alumina_alpha	0.0		ethanol		2000/05/19 13:54:42				2000/05/19 14:06:11	23.6	6.2	0.000	0.00	0.00	0.000	1137.3	0.332	0.488	
										2000/05/19 14:14:44	23.6	6.2	0.000	0.00	0.00	0.000	1139.3	0.334	0.495	
										2000/05/19 14:23:38	23.6	6.2	0.000	0.00	0.00	0.000	1137.2	0.330	0.495	
										2000/09/25 10:59:35	27.7	2.9	0.000	16.092973	91.4	45.77	0.000	1432.2	0.000	0.000
										2000/09/25 11:00:06	27.7	2.9	0.000	16.219102	92.1	46.93	0.000	1432.2	0.000	0.000
										2000/09/25 11:00:38	27.7	2.9	0.000	15.987193	90.8	46.01	0.000	1432.2	0.000	0.000
										2000/09/25 11:17:15	27.3	2.9	0.000	0.00	0.00	0.000	1484.7	0.623	0.239	
										2000/09/25 11:24:14	26.2	2.8	0.000	15.406332	83.2	41.86	0.000	1432.2	0.000	0.000
										2000/09/25 11:25:20	26.1	2.8	0.000	13.744859	74.3	29.95	0.000	1432.2	0.000	0.000
										2000/09/25 11:42:45	26.1	2.9	0.086	13.122098	72.9	29.94	0.000	1480.4	0.636	0.241
										2000/09/25 20:24:20	27.3	3.2	0.152	10.782740	57.8	24.90	0.775	1484.1	0.642	0.187
										2000/10/10 10:41:35	28.0	4.7	0.000	5.130253	0.0	51.96	0.000	1432.2	0.000	0.000
										2000/10/10 12:14:32	28.3	4.7	0.000	4.369753	0.0	30.50	0.000	1432.2	0.000	0.000
										2000/10/10 13:17:17	28.3	4.7	0.000	4.258926	0.0	29.12	0.000	1432.2	0.000	0.000
										2000/10/10 14:01:08	28.0	4.7	0.000	4.200571	0.0	28.16	0.000	1432.2	0.000	0.000
										2000/10/24 16:05:03	27.6	4.4	0.000	12.688054	72.2	62.73	0.000	1432.2	0.000	0.000
										2000/10/24 16:48:36	27.6	4.7	0.000	0.00	0.00	0.000	1490.7	0.675	0.258	
										2005/01/18 12:57:56	21.9	6.4	0.000	3.725032	-22.5	181.45	0.106	1452.0	0.030	0.200
										2005/01/18 13:02:38	21.8	6.4	0.000	3.717999	-9.0	181.62	0.159	1386.1	0.030	0.200
										2005/02/09 16:47:49	24.9	4.2	0.000	2.286402	23.5	34.55	0.931	1496.4	0.320	0.599
										2005/02/17 16:26:08	25.1	7.2	0.000	2.632172	23.7	15.57	0.368	1480.2	0.300	0.599
										2005/06/23 14:33:55	23.0	5.3	0.000	0.00	0.00	0.000	1501.7	0.135	0.599	
										2005/06/23 14:40:31	23.0	5.3	0.000	0.00	0.00	0.000	1501.5	0.159	0.599	
										2005/06/23 14:47:37	23.0	5.2	0.000	0.00	0.00	0.000	1505.3	0.114	0.599	
										2005/06/23 14:54:01	23.0	5.2	0.000	0.00	0.00	0.000	1504.6	0.099	0.599	

The third column to the right of the Experiment Date, labeled "N" for Notes, may contain an exclamation point! This indicates that a memo has been left for this particular experiment. If you left-click on this cell you can read the saved note, which would appear as in the figure below. If you right-click on this same cell, any existing text turns red and you can edit this text as you like. There is no limit to the size of the text that can be added. You can Copy and Paste information to and from this edit box as you like. To escape without saving any changes just press the Escape key. To save any comments press Cntrl-Enter.

DT1200 Data during 10 Yrs for Sigma-Aldrich in water , measured by P.JG for project Repro\_03 using system # -2

File View

wt%	Disperse Phase	xwt%	Ext Disp Phase	Medium	Mfr	Trade ID	Experiment Date	Titr Prot	T Prot	N	Temp	pH	K1	Cvi	zp	CviPha	z
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:15:56				13.9	7.0		2,606.737	-33.7	269.98	0.
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:16:46	none,	none		13.8	7.0		2,924.853	37.9	89.89	0.
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:19:15	none,	none		14.2	7.0		2,970.499	38.1	89.98	0.
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:19:44	none,	none		14.3	7.0		2,973.115	-38.0	179.85	0.
<p>This experiment was done to test the repeatability over a long time. The temperature was not controlled but increased gradually 14.8 to 23.1 C during the experiment.</p>																	
							11/24/2005 12:20:28	time,200	none		14.8	7.0		3,021.931	-38.2	180.29	0.
											15.2	7.0		3,032.309	-38.0	180.06	0.
											15.3	7.0		3,021.897	-37.8	180.74	0.
											15.3	7.0		3,015.318	-37.7	180.70	0.
											15.4	7.0		3,019.538	-37.7	180.36	0.
											15.5	7.0		3,022.518	-37.6	180.35	0.
											15.5	7.0		3,033.497	-37.7	180.17	0.
											15.5	7.0		3,042.939	-37.8	180.34	0.
											15.6	7.0		3,068.550	-38.1	180.25	0.
											15.6	7.0		3,030.200	-37.6	180.81	0.
											15.6	7.0		3,063.191	-38.0	180.58	0.
											15.7	7.0		3,045.680	-37.7	180.47	0.
											15.8	7.0		3,060.227	-37.8	180.86	0.
											15.9	7.0		3,102.487	-38.2	180.37	0.
											16.1	7.0		3,125.238	-38.3	180.28	0.
											16.3	7.0		3,124.351	-38.1	180.96	0.
											16.5	7.0		3,162.984	-38.3	180.89	0.
											16.8	7.0		3,183.358	-38.4	181.09	0.
											17.1	7.0		3,207.462	-38.4	181.20	0.
											17.4	7.0		3,188.210	-37.9	181.20	0.

Notes related to this experiment. Click on ! to view existing notes. Double click any record to add or edit notes.

### Getting a quick overview of the contents of a large set of saved data

To get a quick picture of all the data stored on your machine, or all the data during some extended period of time, it is convenient to use the collapse button (or menu command **View – Collapse**). This gives a quick overview of all the experiments contained in they the present query without any of the measurement details. This capability is illustrated in the figure below. This is a very convenient way to get an overview of all the data available or to find data of particular interest.

DT1200 Data during 10 Yrs for Sigma-Aldrich in water , measured by P.JG for project Repro\_03 using system # -2

File View

wt%	Disperse Phase	xwt%	Ext Disp Phase	Medium	Mfr	Trade ID	Experiment Date	Titr Prot	T Prot	N
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:15:56			
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:16:46	none,	none	
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:19:15	none,	none	
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:19:44	none,	none	
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:20:28	time,200	none	!

Material used for disperse phase of this sample

If you find an experiment of particular interest that contains for example several measurements, you can expand that experiment by clicking on the + contained in the Experiment date.

If you want to see all the measurements in great detail, use the Expand button (or menu **View – Expand**). Then every measurement collected occupies one line in the table.

### Selecting data fields to be displayed

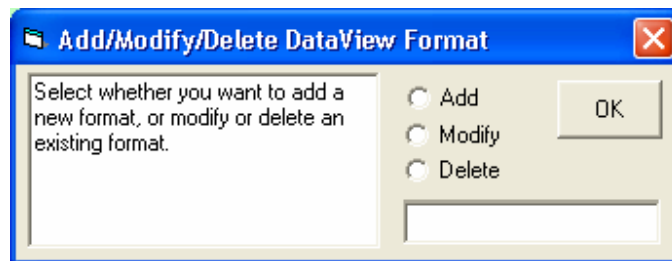
You can select what information you want to display in the DataView. By default, the Data View displays just Basic Information which includes information about the sample, the experiment that was performed, and some basic measurement data. If you click on the View menu you will see several other choices.

View – All fields	displays all fields in the data base
View – Basic measurements	by default, displays basic set of measured data
View – Particle size details	displays detailed information about particle size
View – Acoustic attenuation	displays acoustic attenuation for each frequency
View – Electroacoustic details	displays detailed information about zeta potential
View – Sample modifications	displays modifications to sample, e.g. added reagent
View – Analysis parameters	displays parameters used in analysis of data
View – Rheology	displays rheological data

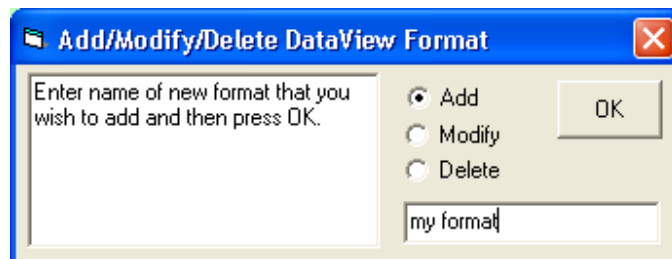
The various choices listed above can be used in any combination.

### Creating a custom view of the data

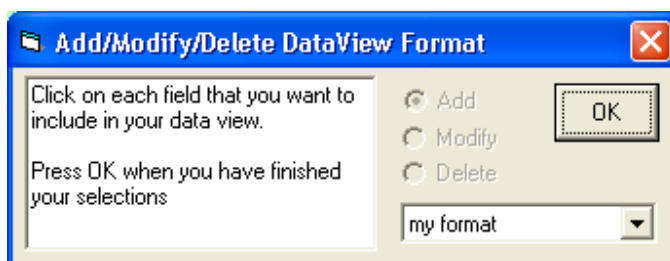
If you like, you can create a custom view of the data. On the Data View window, click on the menu item **Tools Add/modify/delete data view format**. The following window will appear.



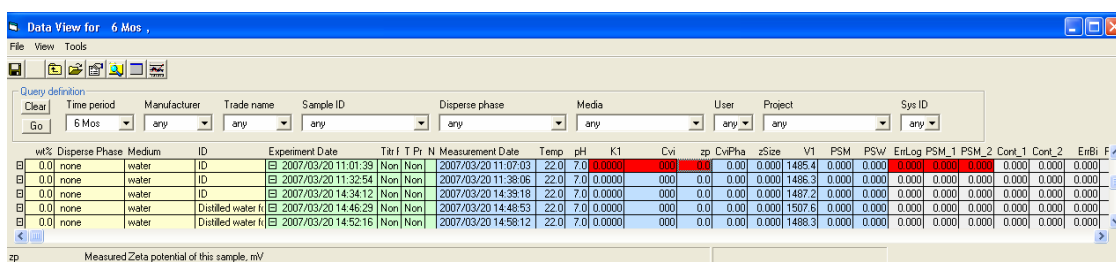
To create a custom format, click on the **Add** option and enter a name for your custom format, e.g. “my format”.



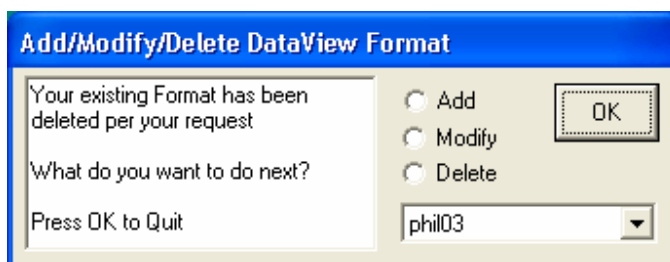
Click **OK** and the following instructions will appear.



On the Data View form, click on the heading for each field that you would like to include in your custom format. Press **OK** when you have finished adding fields to your custom format.



The Add/modify/delete window will then respond as follows. You can now **Add** yet another format, **Modify** or **Delete** an existing format, or press **OK** to quit.



### Editing your measured data using the Data View window

Your data is important. You want to be sure that important information saved to the database is not changed inadvertently or inappropriately. In order to safeguard the integrity of your data, the software takes certain precautions on your behalf.

In order to edit the saved ou must be logged on with User privileges. If you are logged on with only Guest or Reader Privileges then you will not be able to edit existing data.

If you do have User privileges, the menu item “**Allow Editing**” in the **View** menu will be enabled and you must check this box to do any editing of the data. Once “**Allow Editing**” is checked the cursor will change to a pointer with an attached question mark

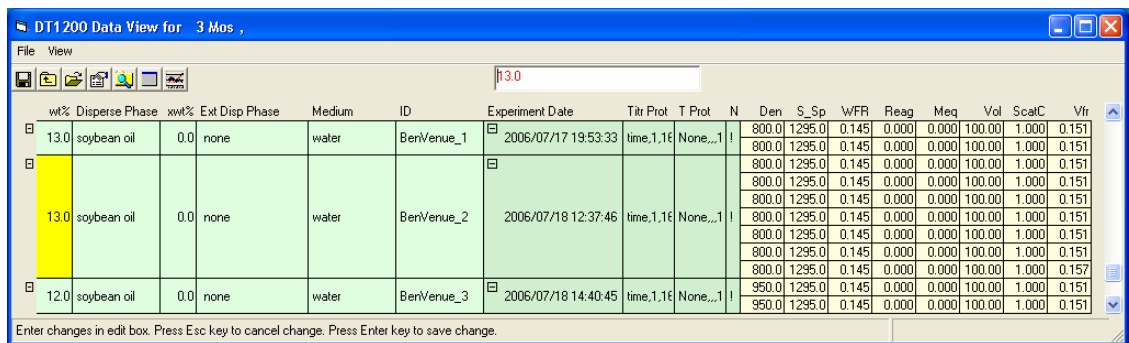
Once Allow Editing is checked, you will be able to edit the contents of only a few fields. You can edit the following:

<i>Field Name</i>	<i>Description</i>
wt%	stated weight % of disperse phase for this sample
Disperse Phase	name of the disperse phase
xWt%	wt% of any extra disperse phase
Ext Disperse Phase	name of any extra disperse phase



Medium ID name of medium  
 WFR text that was used to describe sample  
 weight fraction which is used in calculations for this measurement

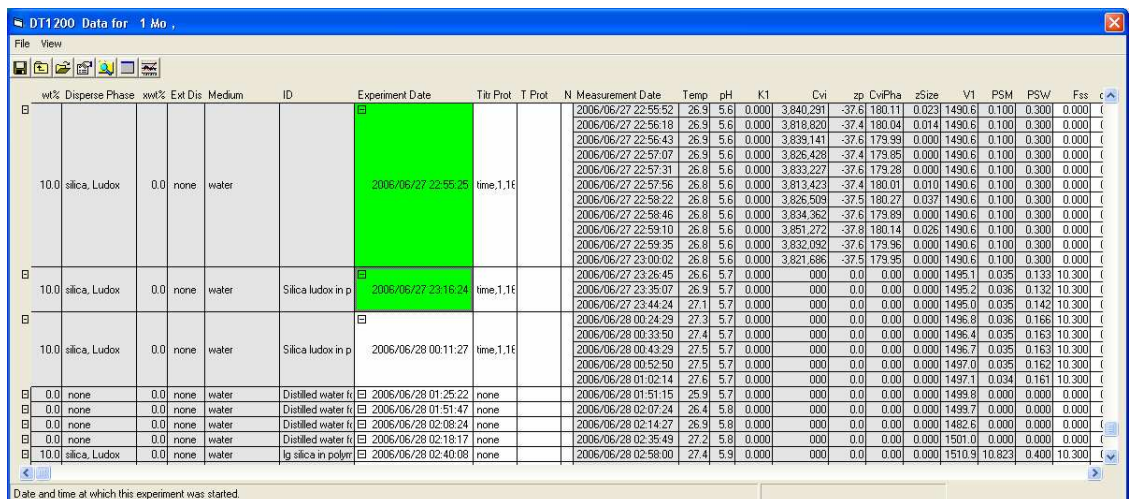
To edit one of these fields for a particular record, click on it. The selected record will become yellow and an edit box will appear for you to enter any changes, as shown in the figure below. Enter a new value if you wish. To save this new value to the database, press **Enter**. To forget making any change, press **Escape**.



If you need to change the name of the Disperse Phase, Medium, or Ext Disperse Phase it is convenient to do this in conjunction with the Define Materials form.

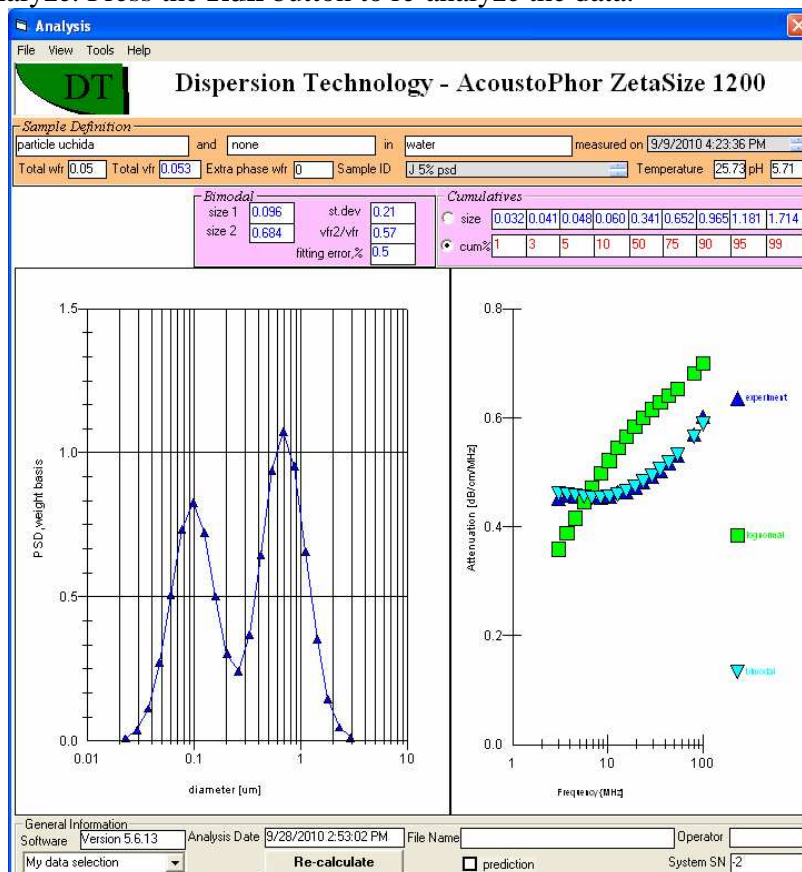
### Re-analyzing selected experiments

If you click on a specific Experiment Date or dates, the selected Experiment Date(s) will change color to green as shown in the figure below. You can click on as many experiments as you wish. Clicking on a selected experiment a second time un-selects an experiment and the green color disappears.



If you then click on the magnifying glass icon, the Analysis window will open automatically with “my data selection” listed as the data Query.

Click on **Measurement date** or **Chemical name** and then select the particular measurement that you wish to re-analyze. Press the **Run** button to re-analyze the data.



Note that Query created by DataView is called "my data selection".

### Exporting selected experiments to Export directory

There are several situations where you might like to export data for one or more experiments. If you have the DT1200 software installed on a second computer you may wish to export selected data to that machine. If you have questions about the measurement or analysis of a particular experiment you may want to export selected experiments and send them to DT, or our local agent, for comments or helpful suggestion.

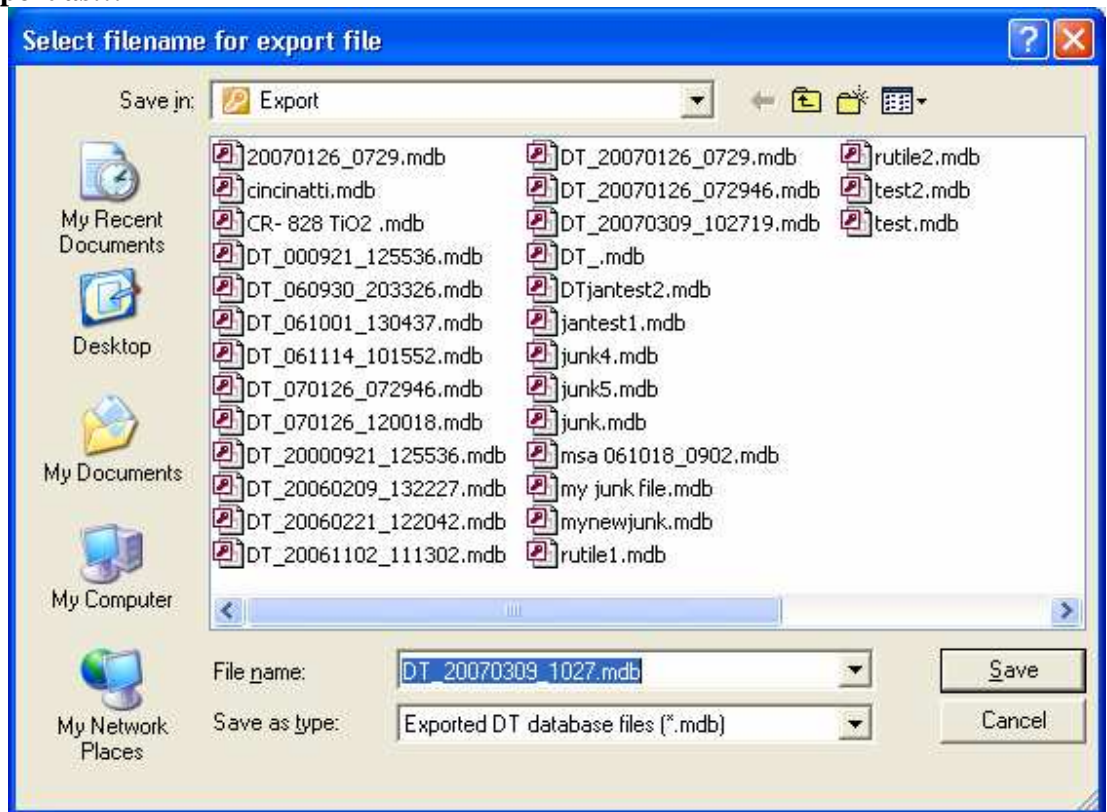
In either case it is a simple matter to export selected experiments. Simply click on each Experiment Date that you want to include in your Export File. As you select each new experiment, the Experiment Date background will change to green to indicate that it has been selected for export. If you click again, on an already selected Experiment Date, that experiment will be un-selected and the color will return to normal. When you have finished selecting experiments, just click on the Export icon on the toolbar (or click on menu **File – Export selected experiments**). The selected data will be saved to an export file with a filename defined by the first experiment date in your selection.

DT1 200 Data during 10 Yrs for Sigma-Aldrich in water , measured by P.JG for project Repro\_03 using system # - 2

wt%	Disperse Phase	xwt%	Ext Disp Phase	Medium	Mfr	Trade ID	Experiment Date	Titr Prot	T Prot	N	Temp	pH	K1	Cvi	zp	CviPha	z'
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:15:56				13.9	7.0	2,606,737	-33.7	263.98	0.	
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:16:46	none,	none		13.8	7.0	2,924,853	-37.9	89.89	0.	
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:19:15	none,	none		14.2	7.0	2,970,499	-38.1	89.98	0.	
10.0	silica, Ludox	0.0	none	water	Sigma	Ludox Lot 2880	11/24/2005 12:19:44	none,	none		14.3	7.0	2,973,115	-38.0	179.85	0.	
											14.8	7.0	3,021,931	-38.2	180.29	0.	
											15.2	7.0	3,032,309	-38.0	180.06	0.	
											15.3	7.0	3,021,897	-37.8	180.74	0.	
											15.3	7.0	3,015,318	-37.7	180.70	0.	
											15.4	7.0	3,019,538	-37.7	180.36	0.	
											15.5	7.0	3,022,518	-37.6	180.35	0.	
											15.5	7.0	3,033,497	-37.7	180.17	0.	
											15.5	7.0	3,042,939	-37.8	180.34	0.	
											15.6	7.0	3,068,550	-38.1	180.25	0.	
											15.6	7.0	3,030,200	-37.6	180.81	0.	
											15.6	7.0	3,063,191	-38.0	180.58	0.	
											15.7	7.0	3,045,680	-37.7	180.47	0.	
											15.8	7.0	3,060,227	-37.8	180.86	0.	
											15.9	7.0	3,102,487	-38.2	180.37	0.	
											16.1	7.0	3,125,238	-38.3	180.28	0.	
											16.3	7.0	3,124,351	-38.1	180.96	0.	
											16.5	7.0	3,162,984	-38.3	180.89	0.	
											16.8	7.0	3,183,358	-38.4	181.09	0.	
											17.1	7.0	3,207,462	-38.4	181.20	0.	
											17.4	7.0	3,188,210	-37.9	181.20	0.	

Date and time at which this experiment was started.

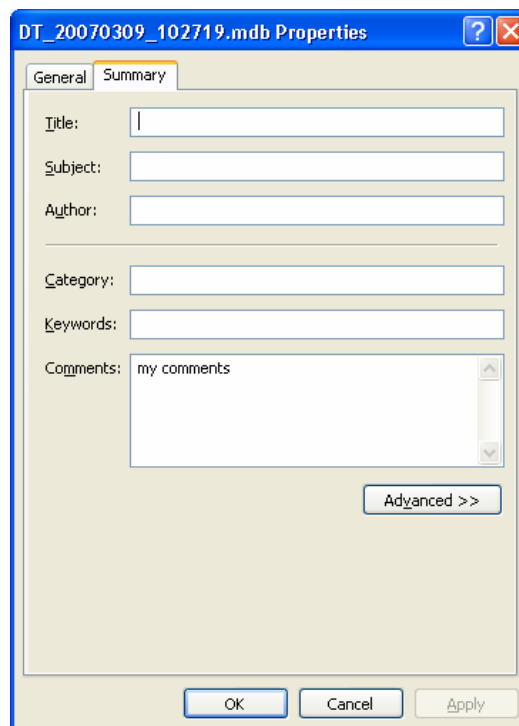
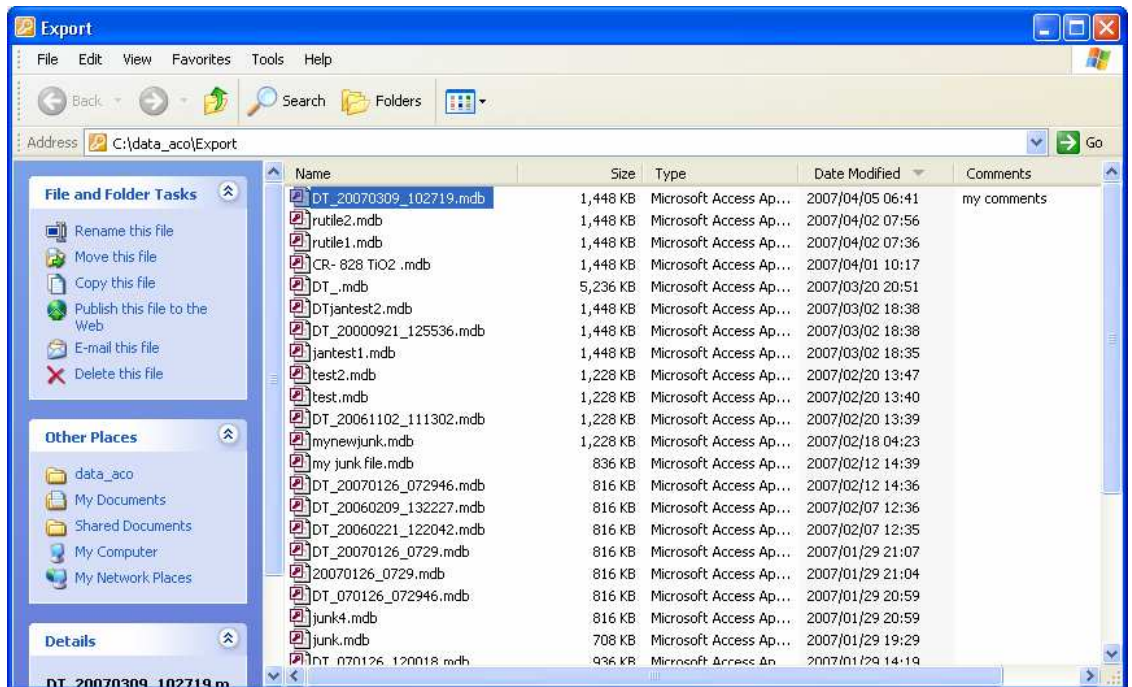
Alternatively, you can save the file using a name that you select by clicking on the menu item **File > Export as...**



Replace the suggested name with any name of your choosing and press the **Save** button.

## Reviewing exported files

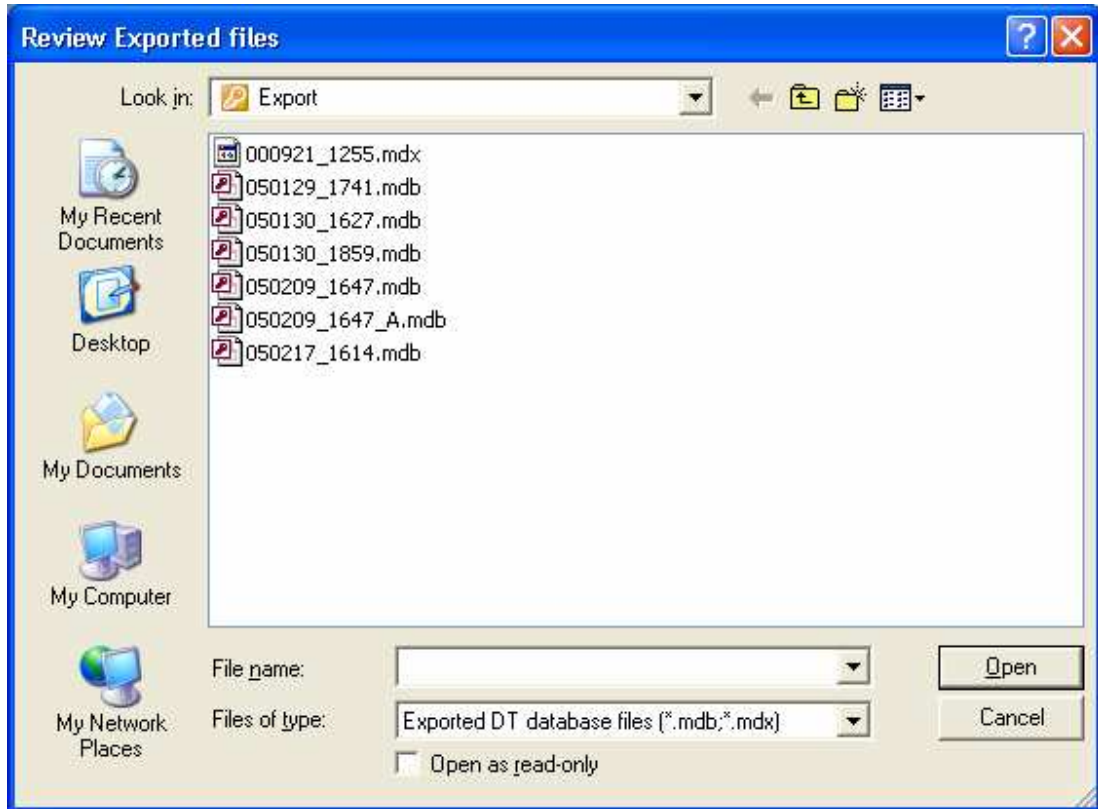
To review all of your exported files, click on menu item File View Export file directory.



Some users may find it convenient to add comments to a particular exported file. To do this right click on the desired file and select Properties. Select the Summary tab in the Properties window. Add any text that you like in the Comment box.

## Sending an export file by email

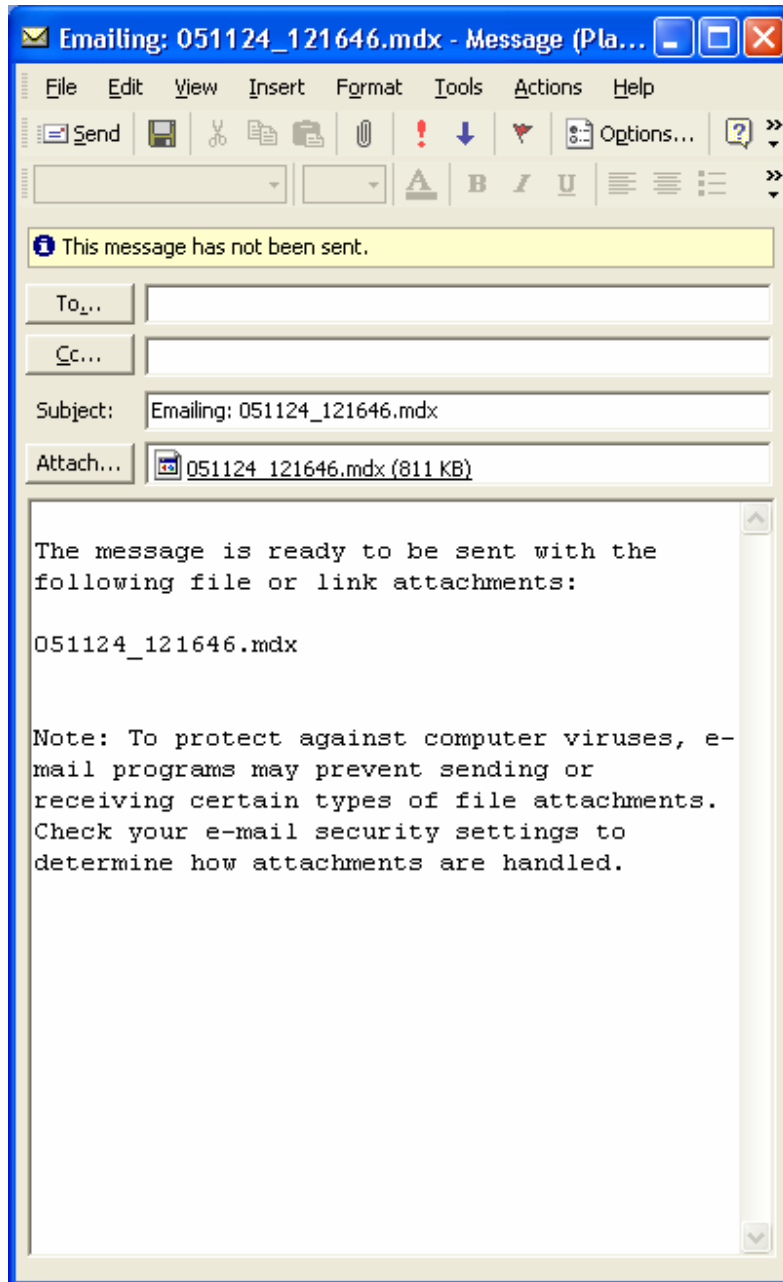
Click on **File – View exported files** to see the files that you have already exported. The form will appear similar to that shown below.



A word of caution is in order. Some email services will not send or receive files with suspicious file name extensions. It may be prudent to rename your export .mdb file before trying to email it. This is simple. Just right click on the file and select rename. Change the file name extension from mdb to mdx. By default, the Review Exported files form will show files with both mdb and mdx suffix.

To send a data file by email, right click on the renamed file and select **Send to – Mail Recipient**.

The email will be automatically composed with the file attached. If your instrument is connected to the internet, simply fill in the email address of your local DT service provider or other desired recipient and press **Send**.



### Saving all Experiments in a Query to an Excel Spreadsheet

It may happen that you want to save the results on the Data View form to an Excel worksheet. This is very easy. To save the data using a default filename just click on **File Save Query to Excel**. The default filename is based on the Experiment date and time of the first experiment in your Query. In this example the first experiment was on 2006/10/18 at 13:47:29.

DT1200 Data View for 2 Mos, Sample defined as alumina\_gamma in water,

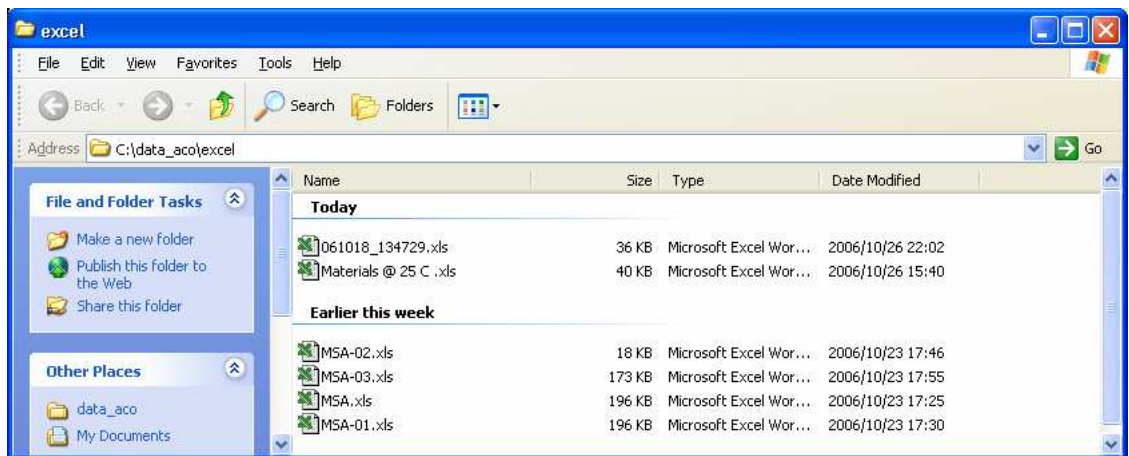
Query definition

Time period: 2 Mos, Manufacturer: any, Trade name: any, Sample ID: any, Disperse phase: alumina\_gamma, Media: water, User: any, Project: any, Sys ID: any

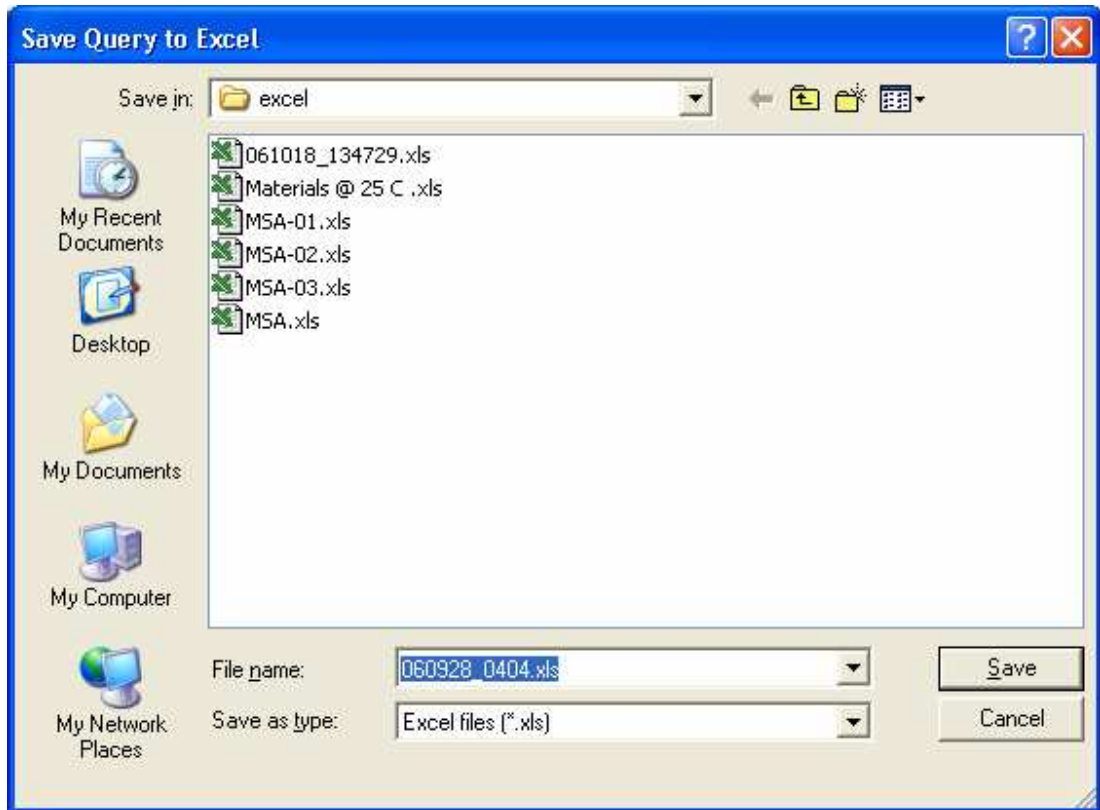
wt%	Disperse Phase	xwt%	Ext Dis	Medium	ID	Experiment Date	Titr Prot	T Prot	N	Measurement Date	Temp	pH	K1	Cvi	zp	CviPha	zSize	V1	PSM	PSW
3.6	alumina_gamma	0.0	none	water	6130	2006/10/18 13:47:29	time,1,16	None...	1	2006/10/18 13:53:39	26.5	6.4	0.000	0.000	0.0	0.000	0.000	1533.2	0.061	0.243
										2006/10/18 13:58:46	26.5	6.4	0.000	0.000	0.0	0.000	0.000	1534.1	0.062	0.245
										2006/10/18 14:03:36	26.5	6.4	0.000	0.000	0.0	0.000	0.000	1533.7	0.062	0.246
3.6	alumina_gamma	0.0	none	water	6130	2006/10/18 14:31:15	time,1,16	None...	1	2006/10/18 14:32:01	26.4	6.4	0.000	215.431	0.0	189.70	0.000	1485.3	0.000	0.000
										2006/10/18 14:34:38	26.4	6.4	0.000	161.501	-3.2	129.61	0.000	1485.3	0.100	0.300
										2006/10/18 14:36:39	26.4	6.4	0.000	192.459	-3.8	115.68	0.000	1485.3	0.100	0.300
										2006/10/18 14:38:38	26.4	6.3	0.000	210.359	-4.2	110.33	0.000	1485.3	0.100	0.300
										2006/10/18 14:40:36	26.4	6.3	0.000	225.988	-4.5	107.10	0.000	1485.3	0.100	0.300
										2006/10/18 14:41:47	26.4	6.3	0.000	255.268	-5.1	198.70	0.448	1485.3	0.100	0.300
3.6	alumina_gamma	0.0	none	water	6135	2006/10/18 14:50:48	time,1,16	None...	1	2006/10/18 14:43:45	26.4	6.3	0.000	153.354	-3.1	149.30	0.000	1485.3	0.100	0.300
										2006/10/18 14:45:44	26.4	6.3	0.000	190.135	-3.8	124.36	0.000	1485.3	0.100	0.300
										2006/10/18 14:47:45	26.4	6.3	0.000	84.344	1.7	295.45	0.000	1485.3	0.100	0.300
										2006/10/18 14:56:53	26.5	6.3	0.000	0.000	0.0	0.00	0.000	1534.2	9.360	0.611
3.6	alumina_gamma	0.0	none	water	6135	2006/10/18 15:00:48	time,1,16	None...	1	2006/10/18 15:01:33	26.5	6.3	0.000	0.000	0.0	0.00	0.000	1534.2	9.360	0.651
										2006/10/18 15:06:31	26.5	6.3	0.000	0.000	0.0	0.00	0.000	1533.5	9.360	0.651
										2006/10/18 15:11:24	26.5	6.3	0.000	0.000	0.0	0.00	0.000	1533.7	9.360	0.651
										2006/10/18 15:16:17	26.5	6.3	0.000	0.000	0.0	0.00	0.000	1533.2	9.036	0.700
										2006/10/18 15:21:09	26.5	6.3	0.000	0.000	0.0	0.00	0.000	1533.4	9.360	0.651
										2006/10/18 15:25:57	26.6	6.3	0.000	0.000	0.0	0.00	0.000	1533.3	9.360	0.611
3.6	alumina_gamma	0.0	none	water	6131	2006/10/18 15:30:57	time,1,16	None...	1	2006/10/18 15:37:32	26.9	6.3	0.000	0.000	0.0	0.00	0.000	1534.5	0.060	0.241
										2006/10/18 15:43:44	26.8	6.3	0.000	0.000	0.0	0.00	0.000	1535.1	0.060	0.241
										2006/10/18 15:48:43	26.9	6.3	0.000	0.000	0.0	0.00	0.000	1534.6	0.060	0.241
3.6	alumina_gamma	0.0	none	water	6133	2006/10/18 15:53:13	time,1,16	None...	1	2006/10/18 16:00:00	27.0	6.3	0.000	0.000	0.0	0.00	0.000	1535.3	0.051	0.213
										2006/10/18 16:05:00	27.0	6.3	0.000	0.000	0.0	0.00	0.000	1533.9	0.051	0.214
										2006/10/18 16:10:04	27.0	6.3	0.000	0.000	0.0	0.00	0.000	1534.2	0.051	0.214

5 Experiment(s), involving altogether 25 measurement(s), are contained in this Query

To look at all the Excel files you have created simply click on **File View Excel Queries**. A window such as that shown below will appear. The file created in this instance, based on the date and time, is named appropriately “061018\_134729.xls”



If you wish, you can instead name the file anything you like. To do this, click on **File -Save Query to Excel as...** A window such as that shown below will appear. The usual default filename will appear in the File name box, but you can change this as you like. You can also change the directory in which the file is to be saved.





## Viewing graphical presentation of data

There are multiple ways to see collected results in graphical presentation. These programs can be accessed through Home page menu **View**.

**Analysis** presents results of particle sizing, rheology or zeta potential for a single measurement. There is a special section on Analysis program due to its importance.

**Graph** program can generate overlays of various curves for different measurements. It is especially useful for overlaying attenuation spectra and particle size distributions.

**Titration Control** presents results of titration. It shows not only the last report but demonstrates how measured parameters evolve in time due to change in chemical composition of the sample.

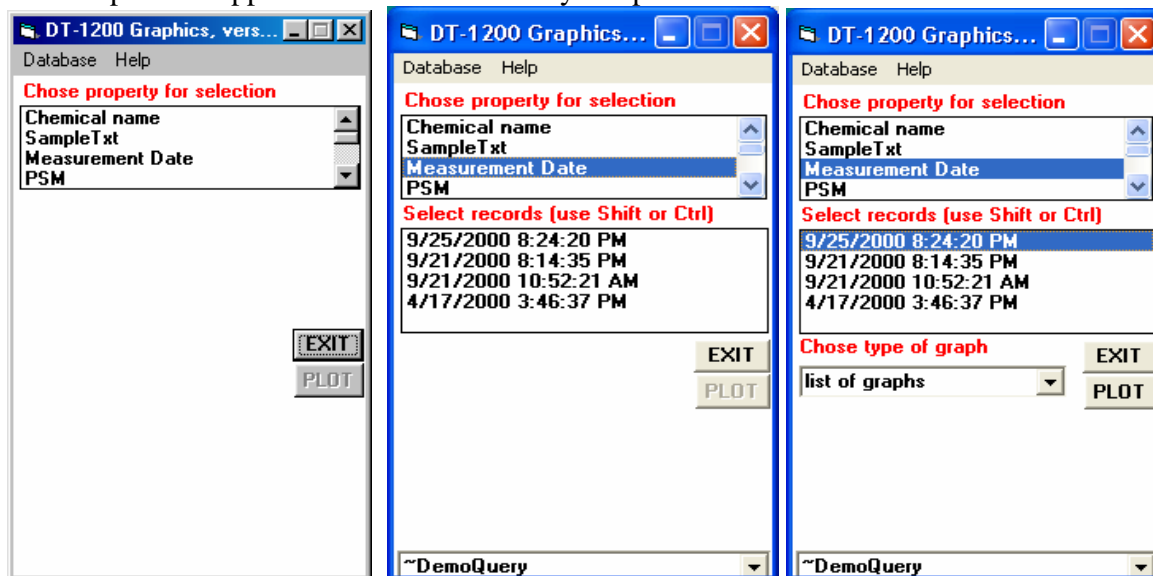
**On-line Continuous data** is useful for plotting results evolving in time. It makes possible to plot any parameter that is being saved to the database versus time.

**Grid raw data** program presents results of the last single measurement. It contains graphs required for troubleshooting procedures.

### *Viewing overlays with Graph program*

The Graph program allows you to create various graphs of the data already obtained and stored in the database. This program prepares set of excel files for selection of records chosen by user and then runs script file that initiates graphical program **PlotIT**.

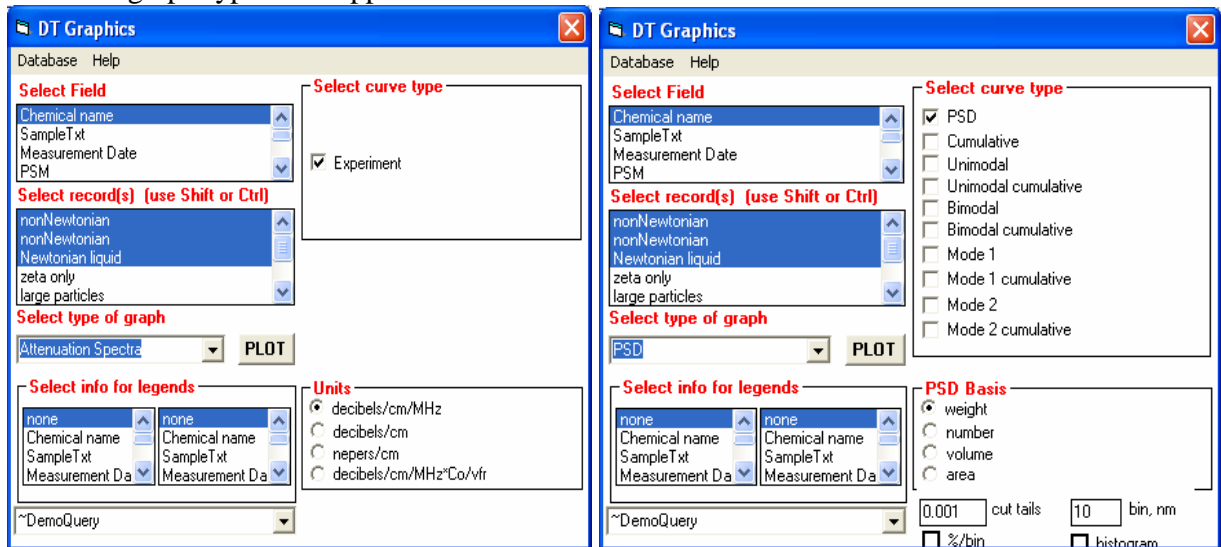
To start the Graph program from the Home page: Click **View > Overlay Graph Data**. The Graph form appears as shown at the very left picture below.



Choose the property by that you would like to use in selecting the particular measurements to be plotted. For example, click on the measurement date. The list of records available for plotting then appears, as shown at center picture.

The records that appear are those that are contained in the current "Query". The name of the current Query is shown in the box at the bottom of the form. In this case it is "~DemoQuery". The Query "Today" always contains all of the measurements made on the current date. Tomorrow, new measurements will be shown corresponding to the measurements made on that day.

Select one of the records. Click on the “list of graphs” pulldown box and a list of the available graph types will appear.



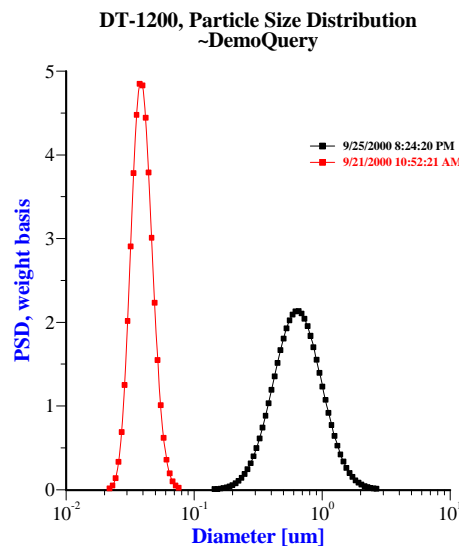
Select the Attenuation spectra graph. As shown on the left Figure, the form expands to allow more choices concerning the items in the legend, the curves to be plotted, and the units for plotting.

Select the PSD graph. As shown on the right Figure, the form expands to allow more choices concerning the items in the legend, the curves to be plotted, and the units for plotting.

The legend can contain two items of information, as defined by your choices in the left-hand and right-hand entries in the Legend frame. For now select Measurement Date in the left-hand legend item, and none for the right-hand legend item.

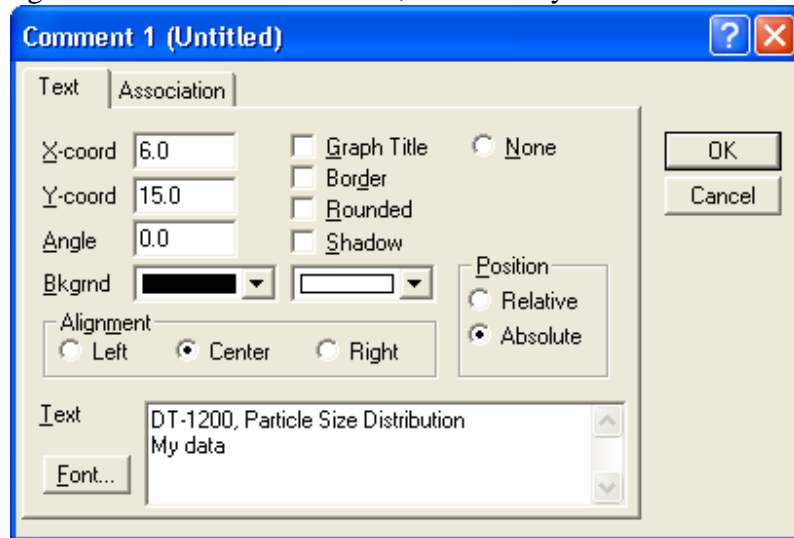
To plot more than one record, select the desired records by clicking on them. Use the control key to select individual records. Alternatively, use the Shift key to select a range of adjacent records.

To generate the plots, click on the Plot button. Plot appears as shown below.



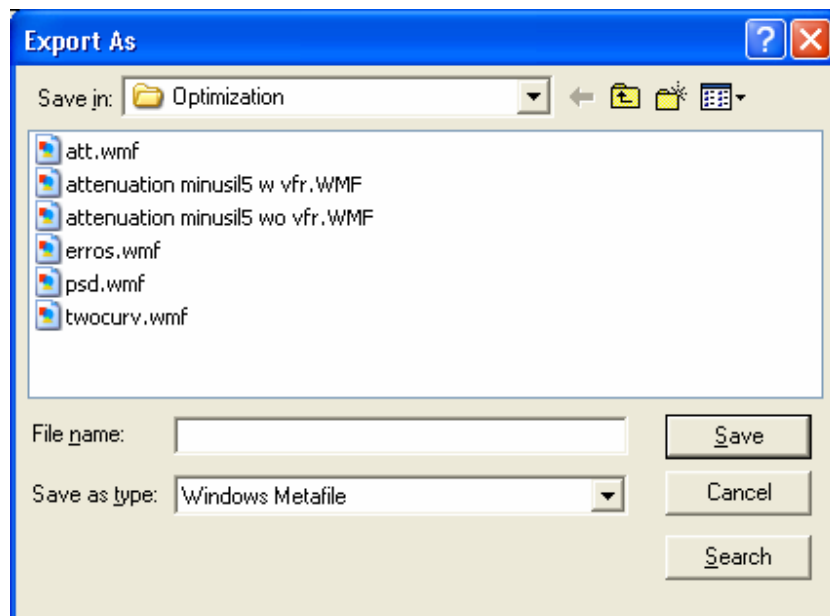
If you like, you can edit many of the features of any plot. Simply click on the feature you would like to edit. For example, click anywhere on the title at the top of the plot. The form depicted

in **Error! Reference source not found.** appears. You can for example edit the text that appears in the title by simply typing new text in the text window, such as “my data”.



### Saving graphs to metafiles

Sometimes it is very convenient to take a graph created with the Graph program and simply copy it to the clipboard and then paste it into a document. Select one of the graphs you made earlier by clicking anywhere on that graph. Click on **File- Export**. The form shown in **Error! Reference source not found.** will then appear. Select the Windows Metafile type (the default) , type in a filename where you would like to save the graph, and click on Save.

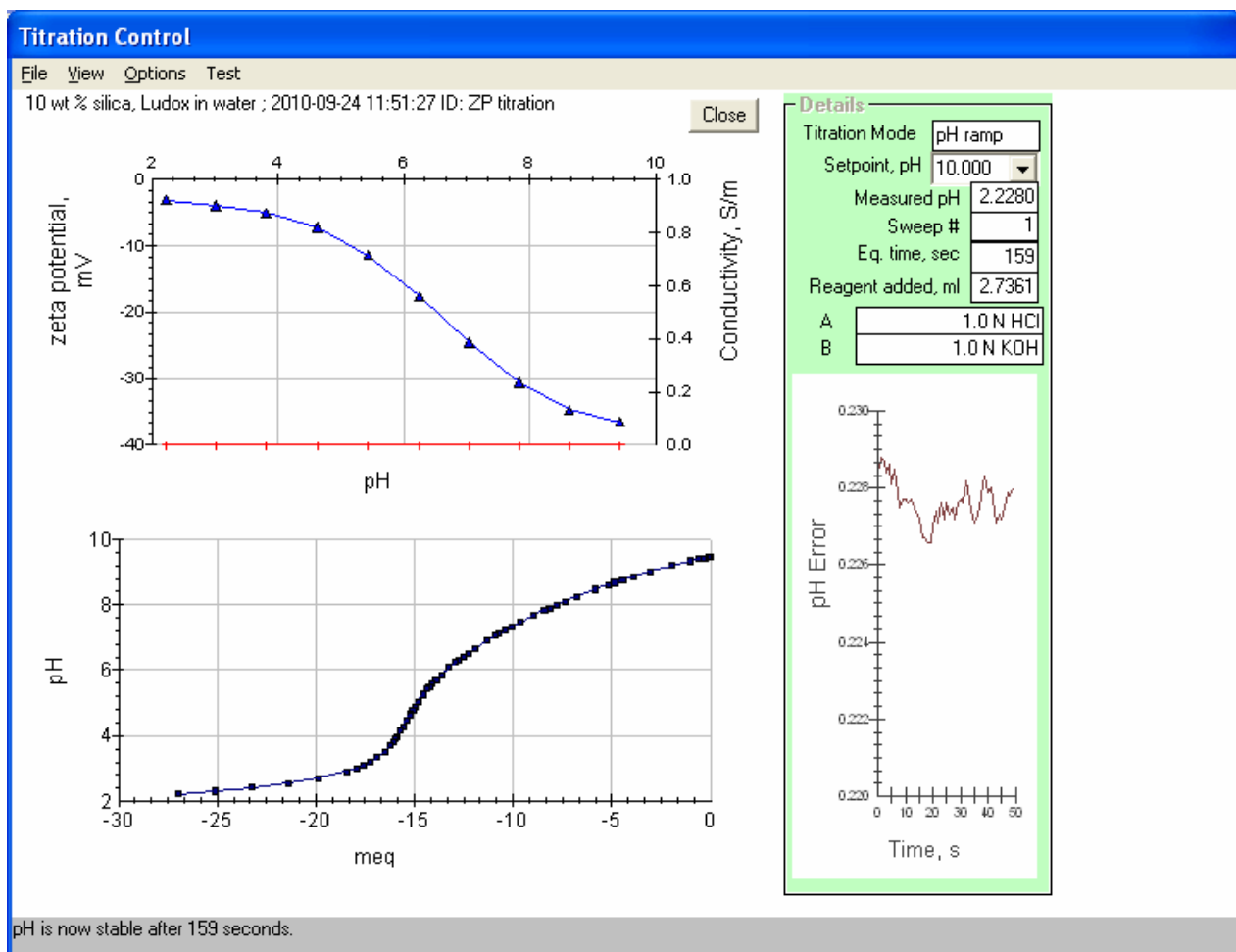


### Adding a saved graph to a document

It is very easy to paste a graph that you have saved to a Word document. Open the word document. Then from the Word menu click on **Insert – Picture – From file**. A file menu appears. Select the filename you saved earlier and click on Save.

## Viewing titrations data with Titration control

Titration control has two graphs that show how Zeta potential, Conductivity and pH change with addition of chemicals. In the case of pH titration typical Titration Control is shown in Figure below. It was measured for silica Ludox.



Top graph shows Zeta potential versus pH. It would also show Conductivity versus pH if this parameter would be measured.

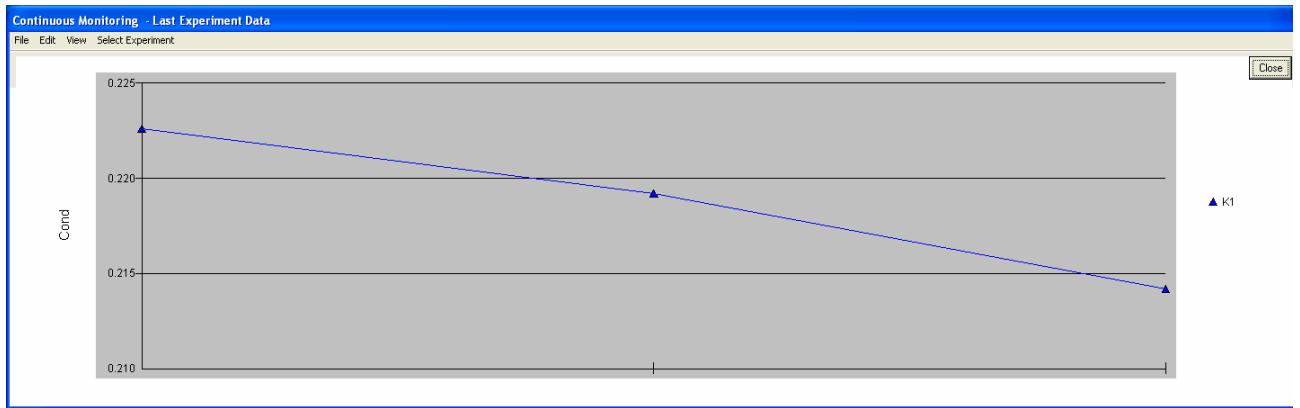
The bottom graph shows pH versus meq of the added acid and base.

This picture could be printed using File menu item. It can be saved to Clipboard using Alt+PrintSc keys and then Paste into the Word file.

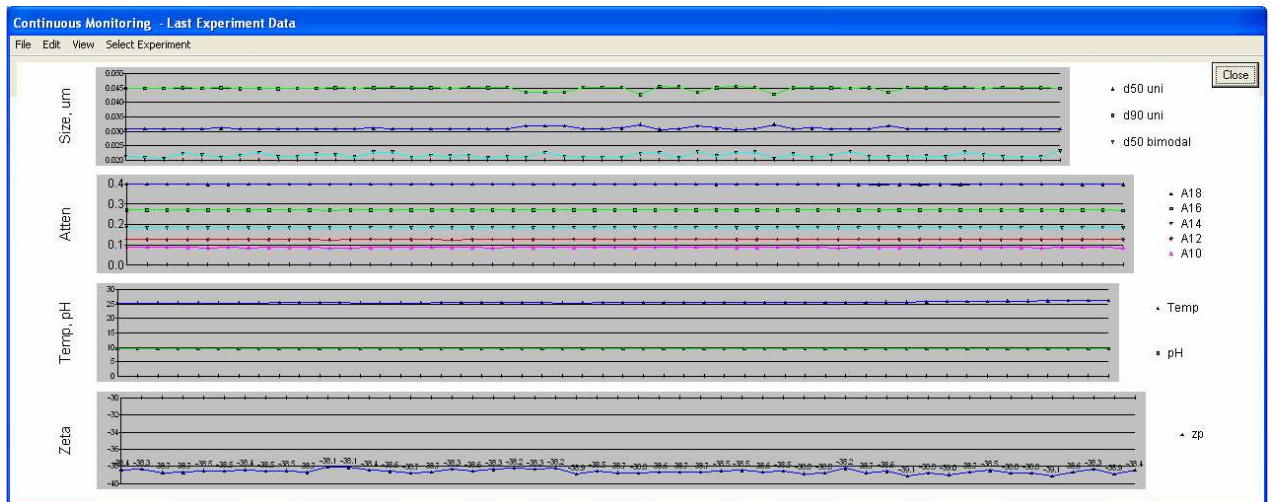
## Viewing time dependences with On-Line Continuous data control

On the Home page: Click **View > On-line/Continuous data**.

A form with the caption "Online Monitoring" appears. The graph for your data would appear similar to that shown below.

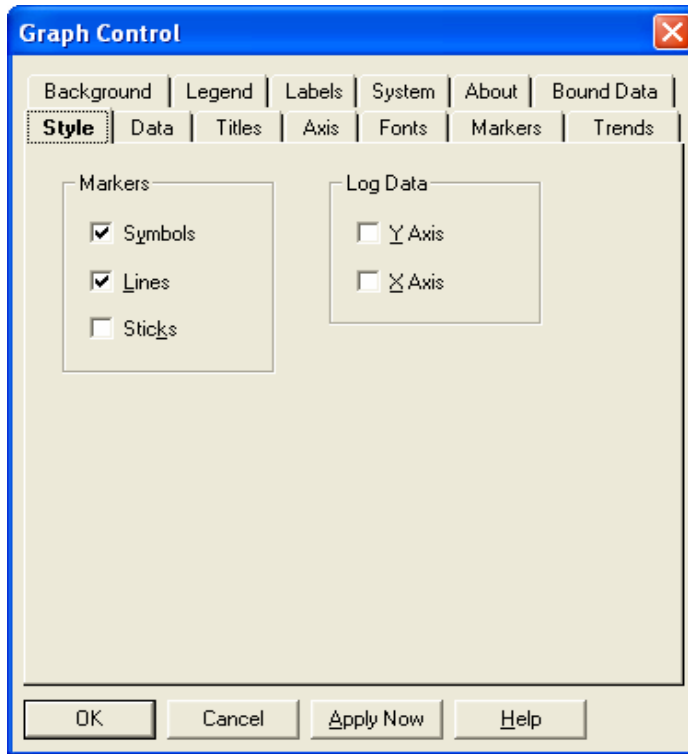


This form would show changes in particular parameter, or set of parameters. Every new measurement would be shown on the graph. User can display graphs for various parameters by checking option on the View menu. Graph below shows such multipurpose presentation.

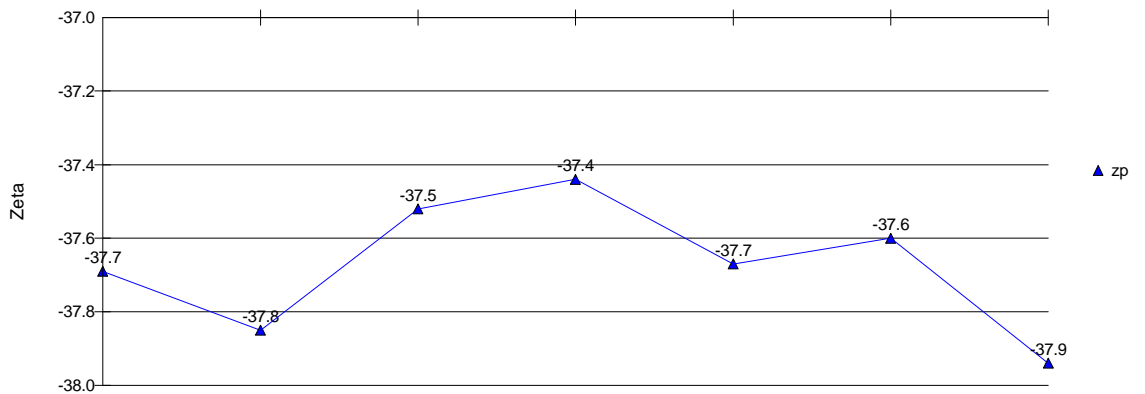


User can save this form as a picture using Alt+PrintSc.

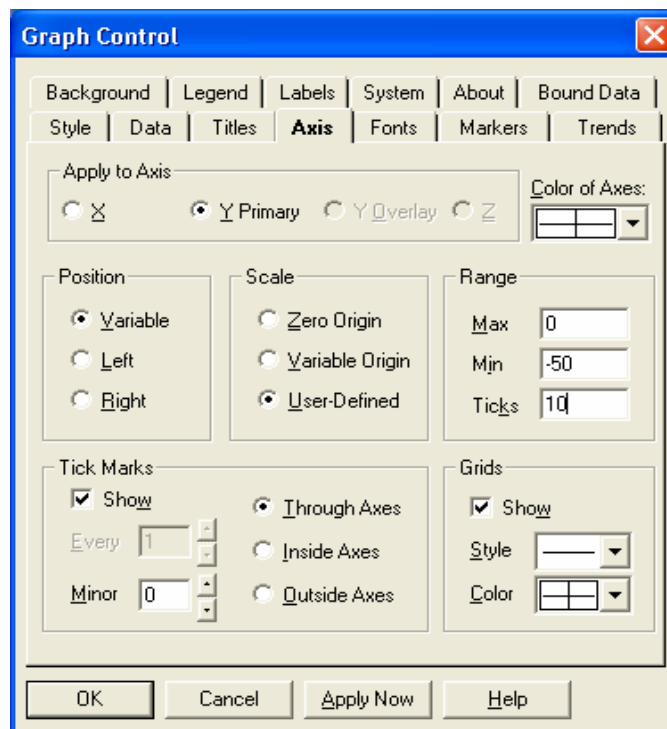
There is possibility of reformatting and saving graphs only. For this purpose Click right mouse button on particular graph. Following dialog box appears.



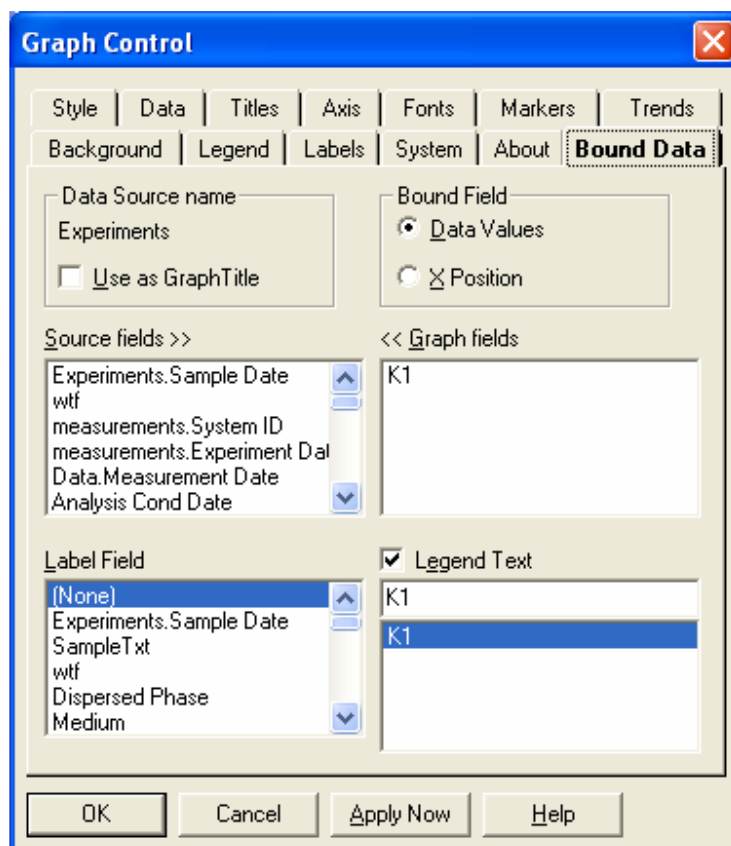
If you go to the System option, you would be able to save just this particular graph to Windows metafile or bitmap in desirable directory. Such picture of multiple measurements of zeta potential is shown below.



User can do many modification to the graph using this dialog box. For instance he can use axis, as shown below.



Using the same dialog box user can change data property shown on particular graph. He should click on the option Bound Data as shown below.



Boxes on the left hand side demonstrate list of database field. Any of this field can be could to the graph. In this particular case it is field K1 – conductivity. If you click on the field name on the right hand side box, it will disappear. Then you can click on the any field on the left hand side boxes and graph would become bound to this new field.

### ***Viewing raw data with Grid***

In an earlier lesson we used the Raw Data Grid form to display the attenuation of water for verification and troubleshooting purposes. In this lesson we will look at some of the other features of the Raw Data Grid. This lesson is of interest for those who want to get some better understanding of the details of the attenuation measurement. Some features will also be used in some of the advanced topics described later.

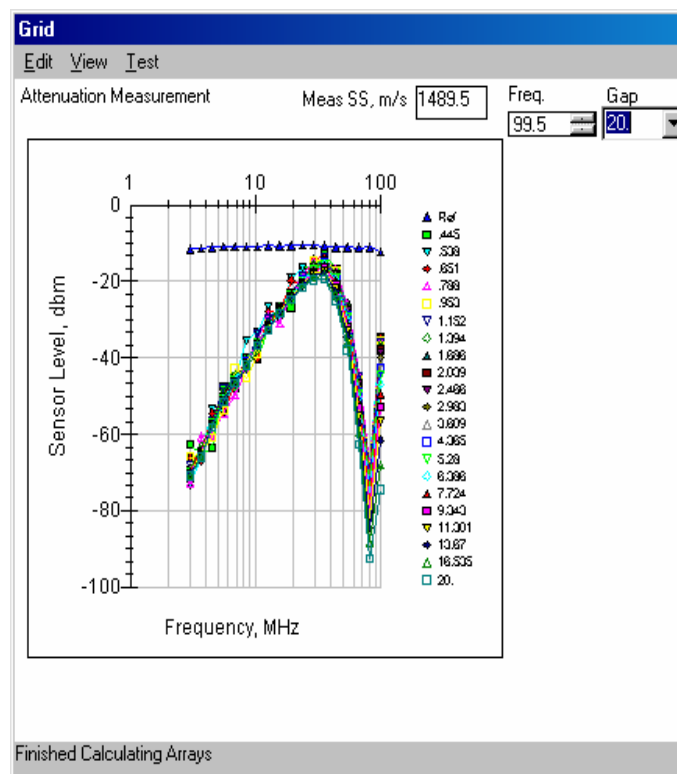
You can proceed from here in two ways. You can simply read the following text, or you can measure the attenuation of water as described earlier and continue with “live data”. The following text will assume that you have just finished a measurement of the attenuation of water and already have the Grid Raw Data form displayed on the screen.

The acoustic attenuation measurement is made at several frequencies, and for each frequency the signal level is measured at several gaps between the transmitting and receiving transducers. In default, the system measures the acoustic signal at 18 frequencies and 21 gaps. Information on this 18 by 21 grid is depicted in several ways on the Grid form, which are represented with various graphs accessible through **Grid, View**.

We present here all these graphs for attenuation measurement.

#### ***Sensor Level***

On the Grid form, click on the menu item View Sensor Level. The Sensor Level graph will appear as shown below.

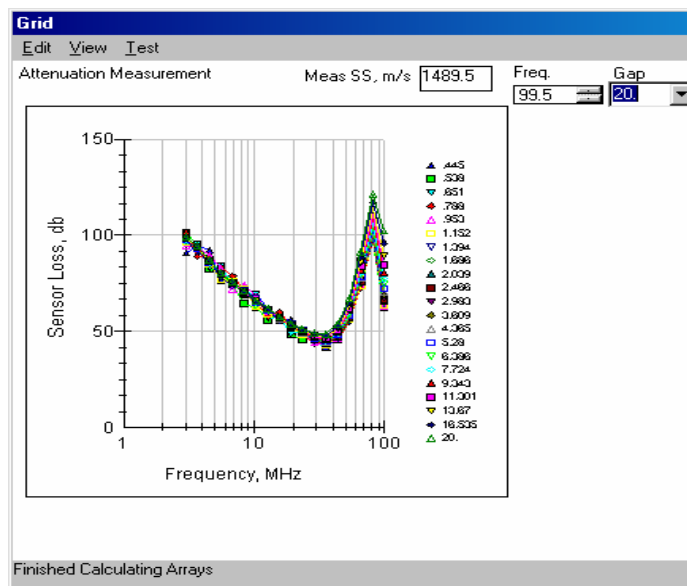




The top curve corresponds to the signal level at the output of the reference channel. If all the electronics and software related to the attenuation measurement are working normally, this curve should be more or less constant with frequency with a value of approximately -10 dbm. The only item not checked by the reference channel level is the acoustic sensor itself. The signal level at the output of the acoustic sensor is shown in the set of 21 curves corresponding to 21 different gaps between the transmitting and receiving transducers.

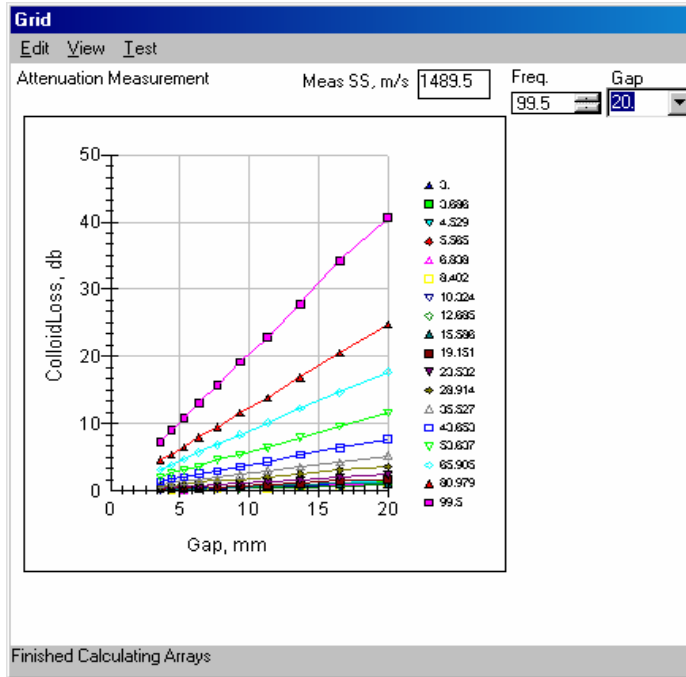
### ***Sensor Loss***

On the Grid form, click on the menu item View Sensor Loss. The Sensor Loss graph will appear as shown below



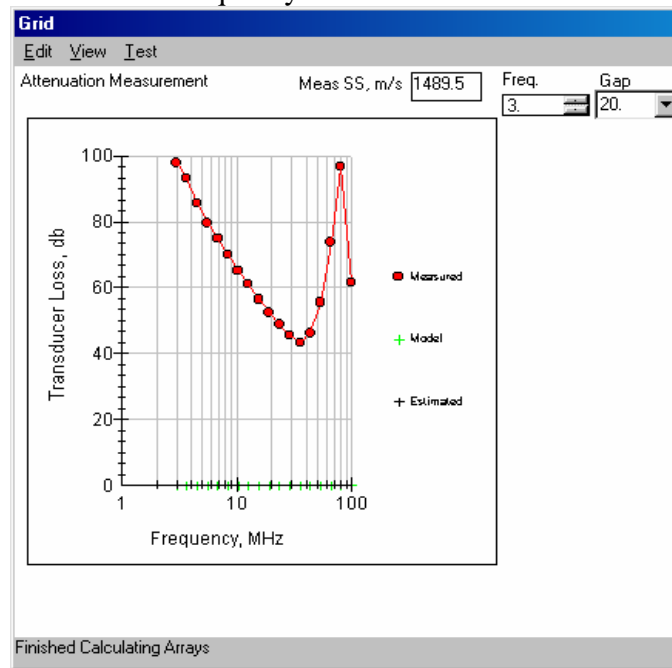
### ***Colloid Loss***

Click on the menu item View Colloid Loss. The Colloid Loss graph will appear as shown below. Colloid loss is very informative. Measurement of attenuation can be considered reliable only when colloid losses versus gap are straight line, as shown below. Otherwise, sample changes during the measurement.



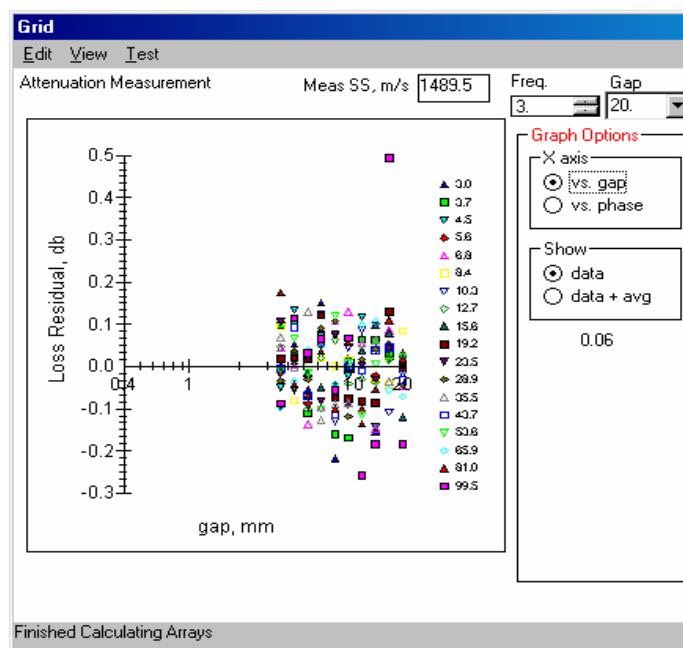
### ***Transducer Loss***

Click on the menu item View Transducer Loss. The Transducer Loss graph will appear as shown below. Minimum on this curve corresponds to the maximum transducer efficiency. Modern transducer have this resonance frequency at 50 MHz. It is done for eliminating blind spot, as for transducer shown below with resonance frequency around 35 MHz.



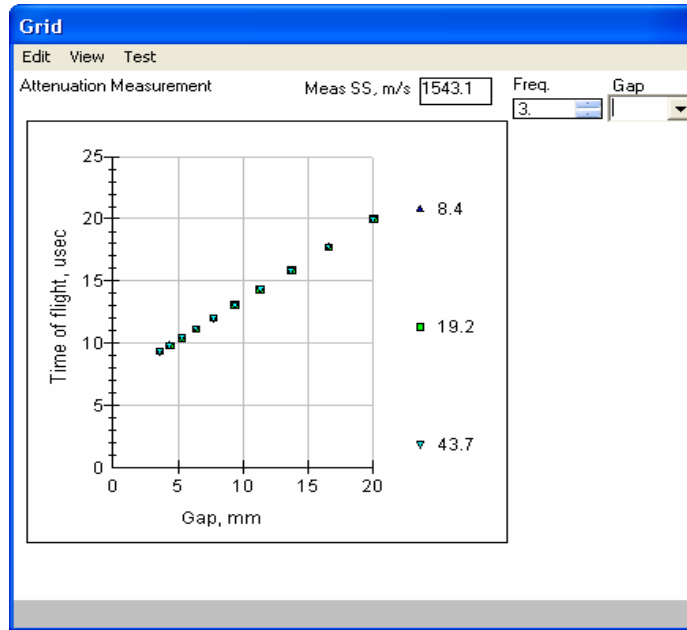
### ***Residual Loss***

Click on the menu item View Loss Residual. The Loss Residual graph will appear as shown below. .



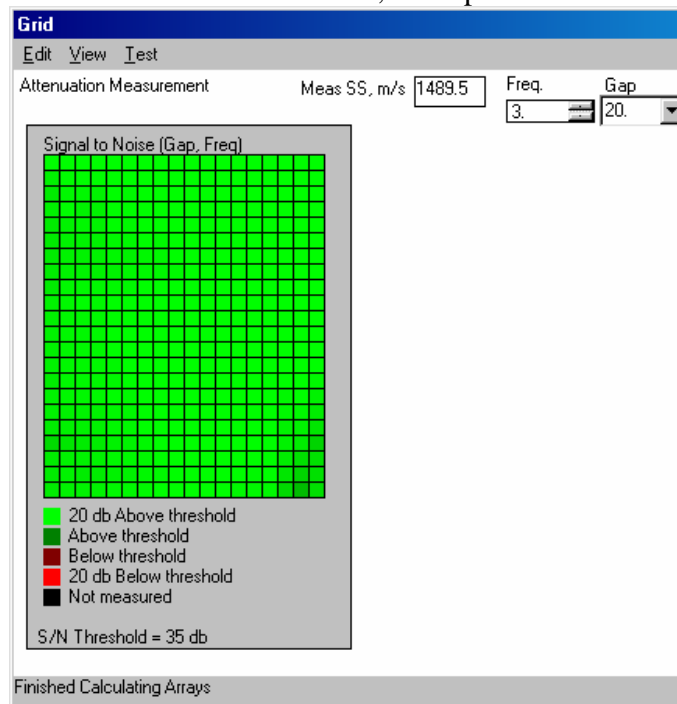
### *Time of Flight*

Click on the menu item View Time of Flight. The Time of Flight graph will appear as shown below. This graph is used for calculating sound speed. Slope of the linear regression to the time of flight versus gap is sound speed. Time of flight is measured only for rather large gaps above 2 mm. This measurement is necessary for correcting time of the pulse arrival, that is unknown a priori.



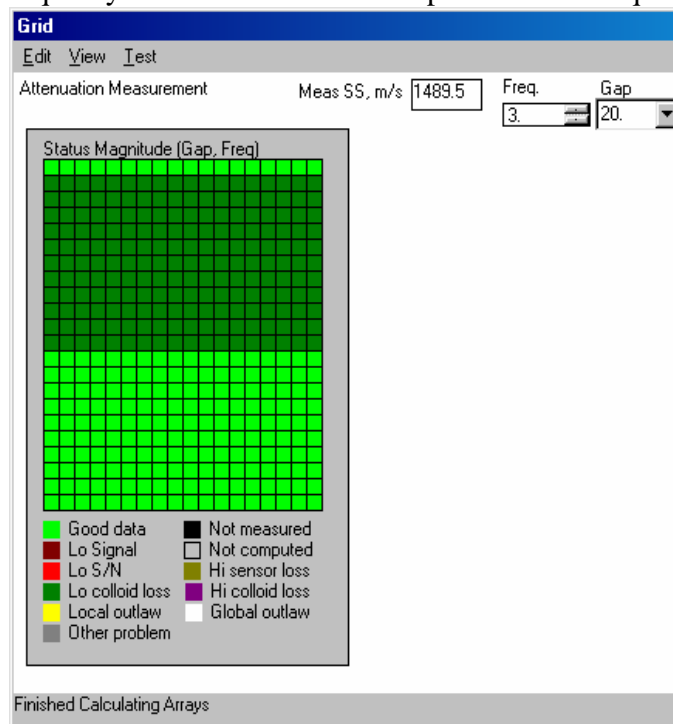
### *S/N Ratio*

This graph shows signal-to-noise ratio. Instrument transmits at least 800 pulses for the each point on the frequency-gap grid. Averaging of the magnitude of the received pulses yields information on S/N ratio. If it is below certain threshold, more pulses are accumulated.



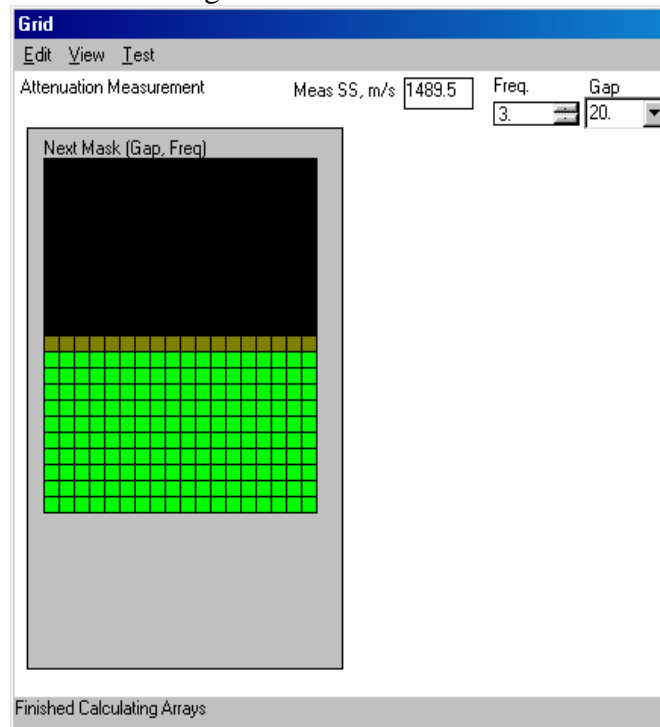
### Status Magnitude

This graph illustrates quality of the data for different points of the frequency-gap grid.



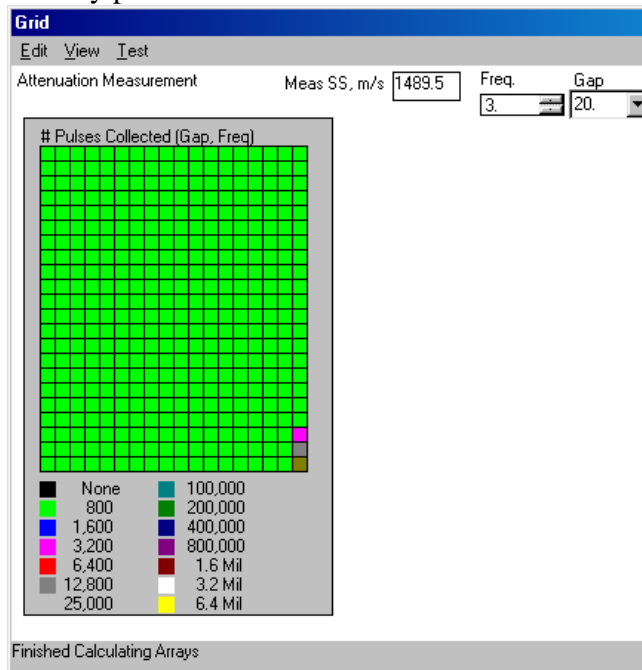
### Mask for the next measurement

Statistical analysis of the measured pulses for the complete grid allows determination of the optimum combination of frequencies and gaps for particular sample. This “mask” would be applied to every next measurement if more than single measurement has been defined in the Protocol.



***Number of pulses collected.***

This graph shows how many pulses were collected for the last measurement.



# Titration

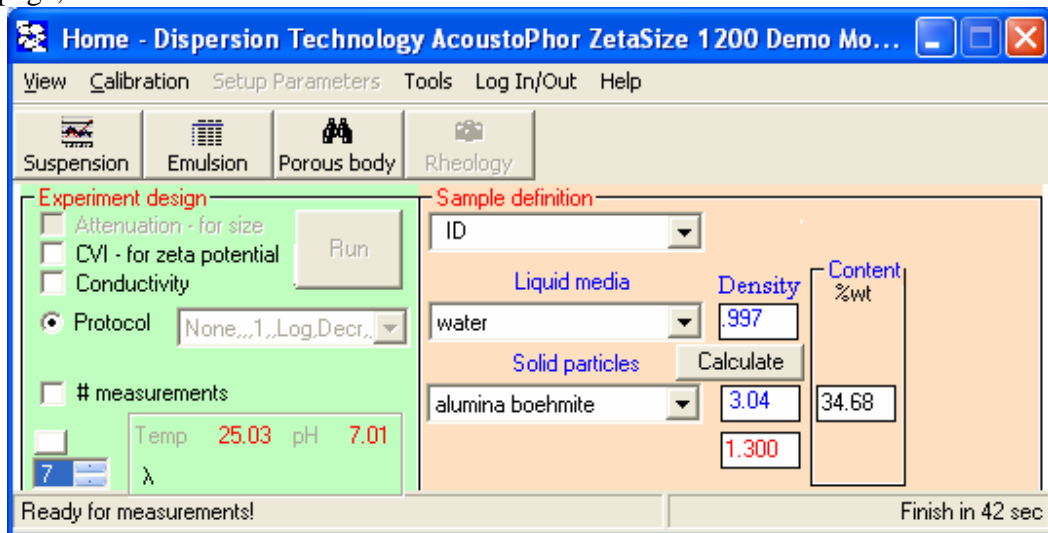
There are 5 different types of titrations that are available with DTI software. This includes 1) pH ramp; 2) pH stat; 3) ml; 4) meq; 5) time. There is also Temperature titration that is setup with a different software object on Home page.

Time titration is the simplest. That is why we start with it. Other titrations require burettes. Their function is discussed next.

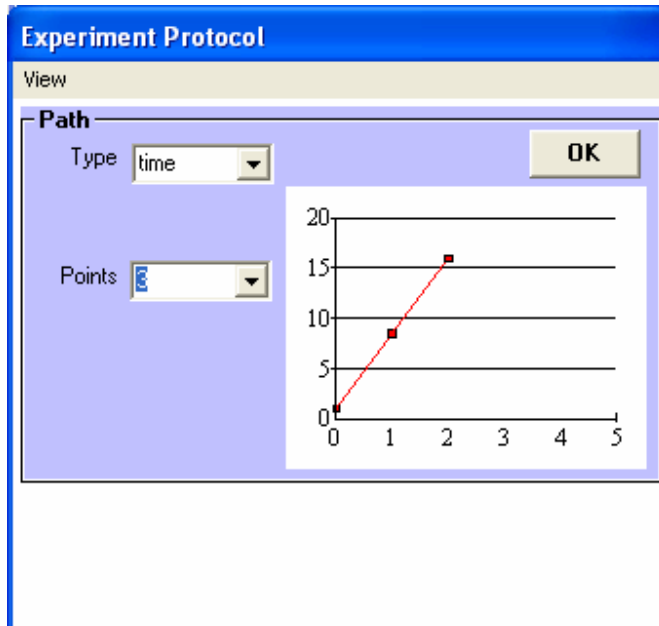
## *Time titration.*

Often times you may want to repeat a given measurement several times as part of a single experiment. Perhaps you just want to see how reproducible the measurements are. Monitoring kinetics of the sample evolution is another important application of this titration.

The simplest way to setup multiple measurement is using box “# measurements” on the Home page, see below.



If you check this box the following form appears:

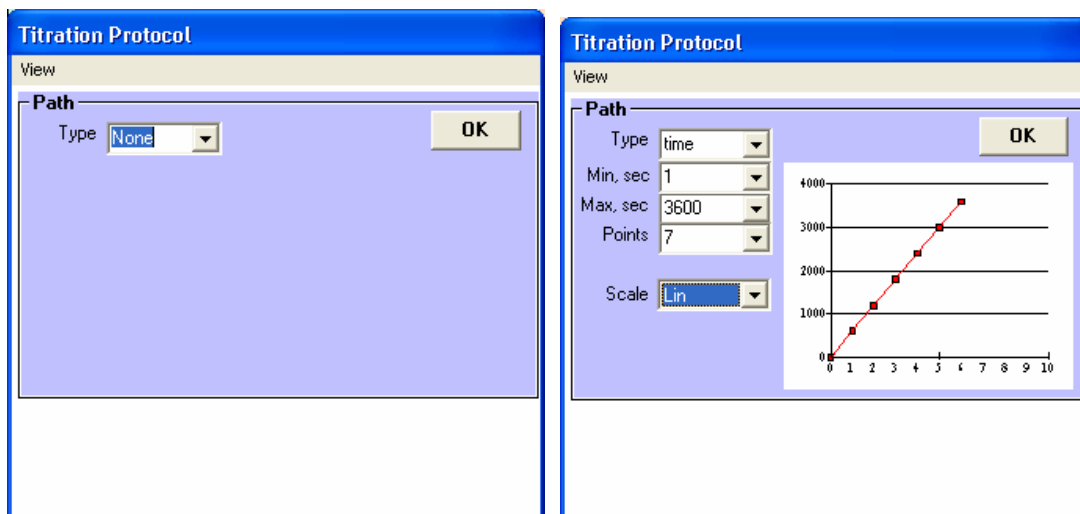


You can either change number of points from 3 to different number, or select it as 1 for single measurements. If you just click 3, then Home page would contain a string with 3 consecutive measurements:

The 'Home' page of the software shows the 'Experiment design' and 'Sample definition' sections. The 'Experiment design' section includes checkboxes for 'Attenuation - for size', 'CVI - for zeta potential', and 'Conductivity', and a 'Run' button. The 'Protocol' is set to 'time,1,16,3,1,Lin,Inc' and '# measurements' is checked. The 'Sample definition' section includes fields for 'Liquid media' (water), 'Density' (.997), 'Solid particles' (alumina boehmite), and 'Content %wt' (34.68). A 'Calculate' button is also present. The status bar at the bottom indicates 'Ready for measurements!' and 'Finish in 2 min, 6 sec'.

You can achieve the same result by clicking on Protocol on the Home page. The form on the left would appear. Then in the combo box Type, you should select "time". The form on the right hand side would appear. Again you can select number of measurements. Box "# measurements" simply provides short cut to this form.

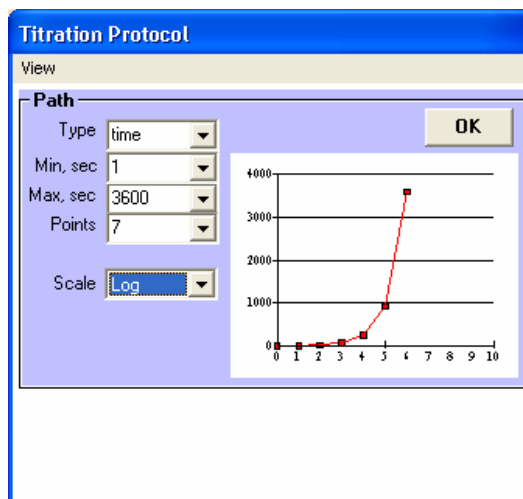




If you just specify the number of points in a time protocol, the measurements will be made as quickly as possible, depending on the complexity of the individual measurements (zp, psd, sc or some combination of each). Sometimes it is desirable to make measurements on some fixed schedule that spreads the measurements out over some period of time. This is done as follows:

- On the Home page:
- Double click **Titration**
- On the Titration
- Click **View > Details**

You will note that several additional items appear now on the screen, as shown in the figure at right.



You can spread the measurements over a fixed time period by specifying the value of “**Max, sec**”. For example, if you set this value to 3600, the measurements will be spread over 3600 seconds or 1 hour.

In addition, you can specify that the first measurement will not be started for a given time interval by specifying the value of “**Min, sec**”. For example, if you set this value to 60, the first measurement will not start until 1 minute after you click on Run.

Sometimes it may be desirable to make measurements more frequently at first and then to make them less often as the experiment proceeds. We can accomplish this by selecting Log from the Scale pull down list. The protocol form would then appear as shown at right.

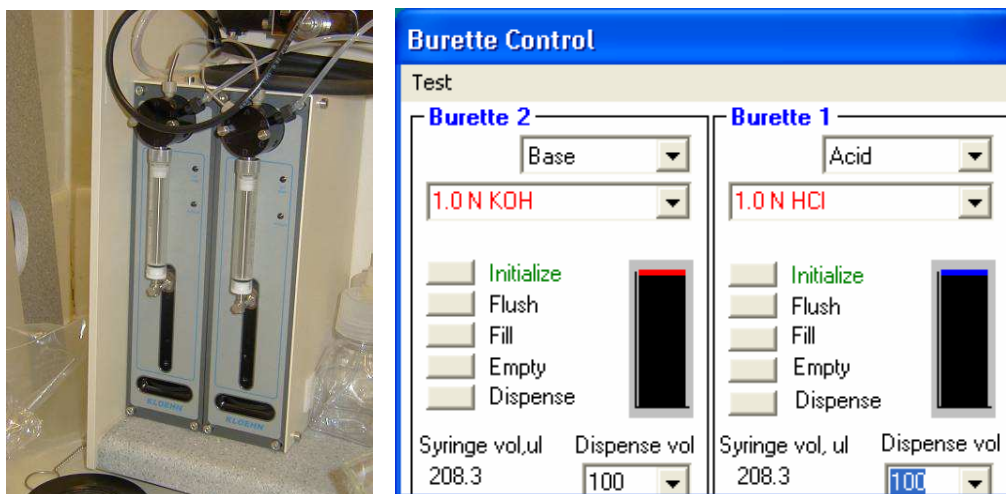
In all of these cases, the selected titration protocol is saved by clicking on **OK**.

If you are following these tutorials in sequence, then you already have defined the sample as 10 wt% colloidal silica and have checked only the cvi measurement. In this case all you need to do now is click RUN and watch the results.

### ***Initializing the burettes***

Each burette must be initialized before it can be used to dispense reagent. After the burette is initialized, it then knows the position of the syringe plunger and how much reagent is left in it that can be dispensed. If you are not planning to use the burette to dispense reagents you can skip this step. You can always initialize the burette later, but you will have to empty any sample before doing so, or some unspecified amount of both reagents will be added to the sample.

Some of the following lessons do use the burettes, so let's initialize it now. The two burettes are shown in the figure at right.



On the Home page:

Click **View > Burette control**.

The Burette Control form will appear as shown at right. Some instruments may only have one burette installed in which case Burette 2 will not be shown. By default, it is assumed that 1 N HCl is installed in Burette 1 on the right and 1 N KOH is installed in Burette 2 on the left, but this can be changed as will be shown later.

On Burette 1:

Click **Initialize**

The plunger moves to empty the contents of the syringe into the sample, and then only partially fills it with fresh reagent. Notice that after initialization the Initialize text changes color to green. Next initialize the second burette in the same way.

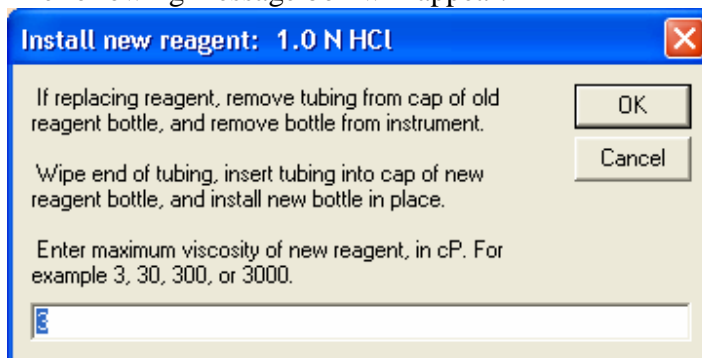
## ***Setting up burettes with 1 Normal Acid and Base Reagents***

If your burettes are already set up with 1N acid and 1 N base reagent, then you can skip this lesson if you are in a hurry. However, if you plan to use other reagents in your work and you want to understand how to load the burette with fresh reagents, proceed as follows.

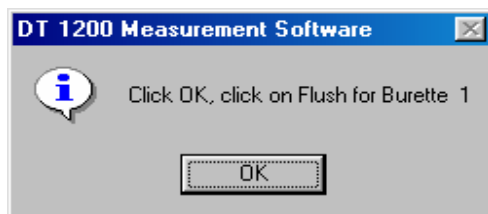
Click on the **down arrow** on first pull down box at the top of the form for Burette 1.  
Select **Acid**.

Click on **down arrow** on second pull down box.  
Select **1.0 N HCl**

The following message box will appear:



Follow instructions concerning adding new reagent.  
Most reagents will have a viscosity of less than 3 cP, so then just click **OK**.



The message shown at right then appears.

Click **OK**.

Click **Flush**.

Notice that the syringe first pulls in a small amount of air, then some fresh reagent from the bottle. Then it dispenses this volume of air and reagent to the sample via the dispensing probe. This sequence of partially filling the syringe with air and reagent is repeated three times. The purpose of this operation is to remove any trace of the previous reagent. At the end of the flush cycle the burette is empty of any air and contains only some new reagent.

Click the fill button for burette 1. Note that the syringe moves all the way down, filling the syringe with 5 ml of reagent.

You can also empty the syringe by clicking of the Empty button, but we will not do that just now. Instead we will manually dispense a specified dose of reagent.

Click on the pull down box for the dispense volume for Burette 1. Select a dose of 100 ul. Click on the Dispense button to dispense this volume of reagent. Note that even this small dose is

not dispensed suddenly, but instead dispensed slowly over the next 10 seconds or so. This is done so as not to shock the system by adding reagent too quickly.

Repeat the same process for Burette 2 using 1.0 N KOH. Then empty the Chamber and refill with distilled water.

### ***Setting up burette to dispense any acid, base, salt, polymer, or surfactant***

The burettes can be used to dispense any concentration of acid, base, salt, polymer, surfactant, or virtually any other liquid reagent.

Click on the **down arrow** on first pull-down box at the top of the form for Burette 1.

Select **Acid, Base Salt Polymer** or **Surfactant** as desired

Click on **down arrow** on second pull down box.

Select one of displayed choices

Alternatively, you can enter a description of the reagent you want to use. The reagent description must be entered in the following format:

**Value Units Reagent name**

Examples of reagent description:

For an Acid:

**0.3 N H2SO4**

**0.2 N Citric acid**

For a Base:

**0.5 N KOH**

**0.3 N Nitric Acid**

For a Salt:

**1.0 M KCl**

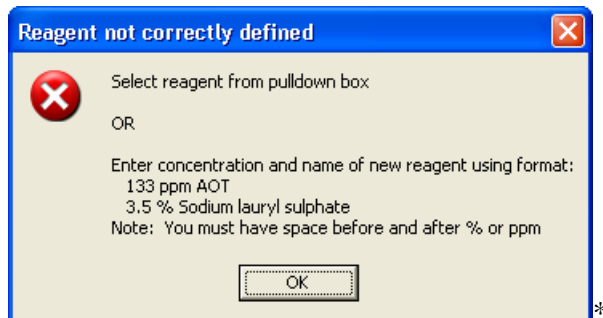
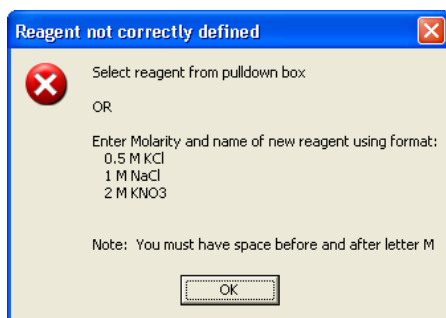
**0.5 N Silver Nitrate**

For a polymer, surfactant, or other material:

**100 ppm Polymin P**

**3 % Aerosol OT**

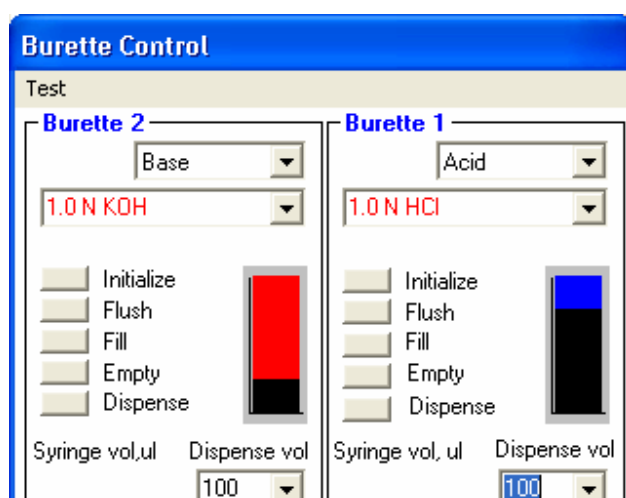
You must type a space before and after the Units (N, M, ppm, %). If the reagent description is not formatted correctly, you will see a message such as one of those below.



## ***Getting familiar with Burette Control in manual mode***

Most of the time you will be using the automatic titration software to control the dispensing of reagents from the burettes into the sample. However, there will be times when it is convenient to dispense reagents manually. You can dispense reagent directly into your sample using the Burette Control as is described here. We assume that you have 10% wt silica Ludox in the sample chamber that is being stirred by the stir bar.

1 N Acid and base reagents have been loaded in the burette, and the pH probe should also be in place. Note the initial pH reading. It should be between 9.0 and 9.3.



Let's dispense a small amount of reagent and test the response of the pH probe. The Burette form is shown above. Set the Dispense volume for Burette 2, containing the 1 N KOH base, to 100 ul. Click on the Dispense command button. You will hear the burette dispense this small volume of reagent in small mini-doses over the next 10 seconds. The reagent is added slowly to allow proper mixing and to prevent shocking the system with a locally high concentrations of reagent.

Observe the increase in pH value. The pH should increase a small amount above the previously noted value.

Now set the dispense volume for Burette 1, containing the 1 N HCL acid, to 100 ul and again click on the Dispense command button.

Observe now the decrease in pH value back to the original value noted before.

## ***Titration – pH ramp, measuring $pI$ of 10 wt% colloidal silica***

In many cases it will be desirable to control the chemistry of your colloid by automatically by adding reagents programmatically to achieve some specific purpose. For example, perhaps we will want to do a pH titration to determine the isoelectric pH of a particular sample.

Let's demonstrate this capability by doing a pH titration of our 10 wt% colloidal silica sample that is already in place. We will plan to titrate over the range of pH 10 to pH 2.

**Titration Protocol**

View

**Path**

Type: pH ramp

Min, pH: 4.

Max, pH: 10.

Points: 7

Sweeps: 2

Ini Dir: Incr

Ini point: 4.

OK

**Reagents**

A Acid 1.0 N HCl in buret # 1

B Base 1.0 N KOH in buret # 2

We can set this up as follows. On the Home page, double click on “Titration”  
Set the parameters inside the Path frame as follows:

Type	<b>pH ramp</b>
Min, pH	<b>2</b>
Max, pH	<b>10</b>
Points	<b>9</b>
Sweeps	<b>2</b>
Ini Dir	<b>Incr</b>

The graph on the Titration Control form, shown in figure to the right, shows the pH path that we have defined. Our demonstration colloid has a pH of about 9.3. We have specified that the initial direction should be increasing and that the maximum pH should be 10, so the sample will be titrated from the initial pH of 9.3 to 10, at which point it will change direction and titrate back down to pH 2 where it will stop.

Click **OK** to save the desired titration protocol.

On the Home page, click **Run** if you wish to start the titration.

### *Using some details in pH ramp Titration in special situations*

Most of the time you can simply specify a pH ramp titration as we have just illustrated. However, it is possible to make some adjustments to the titration protocol that might provide some benefit in special situations. If this is of interest continue reading, otherwise skip to the next lesson.

**Titration Protocol**

View

**Path**

Type: pH ramp

Min. pH: 4.

Max. pH: 10.

Points: 7

Sweeps: 2

Ini Dir: Incr

Ini point: 4.

OK

**Reagents**

A Acid 1.0 N HCl in buret # 1

B Base 1.0 N KOH in buret # 2

**Equilibrium criteria**

Tolerance: 0.25

Drift, pH/s: 0.001

pH/s/s: 0.002

Min Eq. s: 30

Max Eq. s: 300

**Constraints**

Min meq: -300

Max meq: 300

Max Reagent: 30

On the Titration Protocol form:

Click **View > Details**.

This will expand the Titration Protocol to include details of Equilibrium Criteria and Constraints, as shown in the figure to the right.

The Equilibrium criteria describe the conditions that must be met before the sample is judged “stable”. Each time the program adds a dose of reagent, it waits until the sample is stable before calculating whether the sample is at the next setpoint, or if not, how much additional reagent is necessary to get it closer to that setpoint.

The “Tolerance” value specifies how closely the pH must approach each setpoint before the measurement data is obtained.

The “Drift” value specifies the maximum rate of change of pH with respect to time for the sample to be judged stable.

The value of “pH/s/s” specifies the maximum second derivative of the pH value for the sample to be judged stable.

The Min Eq value specifies the minimum time interval, in seconds that must elapse after adding reagent, before the sample can be judged stable. This provides time for the pH probe to show an initial response and time for some initial mixing of the reagent with the sample.

The “Max Eq.” value specifies a maximum time interval, in seconds. If the time after the last reagent dose exceeds this limit, the sample is judged stable whether the other conditions are met or not.

### *pH Stat titration protocol*

In some applications it is of interest to determine how much continued addition of reagent is necessary to maintain the pH at some fixed value. For example it may be desirable to determine the end point in a milling operation. As the material is ground down, new surface is exposed that then requires that additional acid or base be added to keep the pH constant. The end of any chemical demand can indicate the end of grinding effectiveness.

Reagents		
<input checked="" type="checkbox"/>	A Acid	1.0 N HCl in buret # 1
<input checked="" type="checkbox"/>	B Base	1.0 N KOH in buret # 2

On the Titration Protocol form, select pH stat mode. As shown in figure to the right, you can now specify the pH that you would like to hold during measurements. You can also set the number of measurements that you would like to take while holding the pH at this value.

We will not actually do this pH stat titration now, but instead move on to the next lesson.

### *ml titration protocol, 10 wt% colloidal silica*

In some applications you may just want to add a specified amount of a given reagent to the sample in well defined increments. This can be done quite easily with the ml titration protocol.

On the Home page:

Double click **Titration**

On the Titration Protocol screen:

Select Type **ml**

Set the minimum reagent volume as

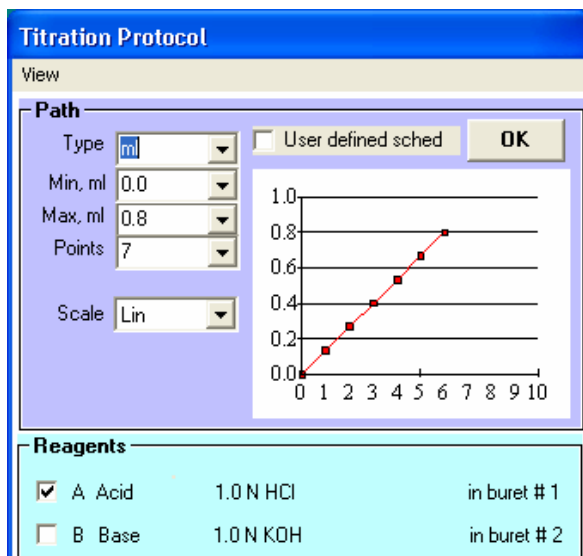
**Min, ml** (Typically this is zero)

Set the maximum reagent volume as\

**Max, ml**

Set the number of reagent additions as **Points**





### *Simple colloid titration without measurements*

Important surface properties of the particles in a dispersion can be gleaned from a simple colloid titration without even measuring properties such as zeta potential, conductivity, or particle size. We can learn a lot from a simple plot of pH vs. the number of milliequivalents (Meq) of added acid or base. The following procedure describes how to do such a colloid titration using for example a 10 wt% alumina slurry ( Sumitomo AKP-30).

Calculating the added Meq requires knowledge of the sample volume, the normality of the acid/base reagents and the volume of added acid/base reagent.

The normal sample volume when using the magnetic stir bar is about 110 ml. Assuming you are using this configuration for this test, you want to make sure that the sample volume stored in the Instrument Constants is this same value. On the home page:

Click **Calibration > Constants**.

Scroll down till you reach Sample volume and click on that line. Enter your sample volume in the edit box at the top of the form. Click on the words "Calibration Constant" directly below edit box. Click on **Save** box to the right of the Edit box to save your new value.

It is assumed that you have already set up the burette with the correct acid/base reagents and have defined the normality of the reagents.

Next we need to set up the Titration protocol for this colloid titration. On the main form, double click on the Titration option to open the Titration Protocol form. On the Titration Protocol form:

Click **View Details**.  
Select type **pH ramp**

The Titration Protocol form will then appear as shown at right. For this example, set the Path as follows:

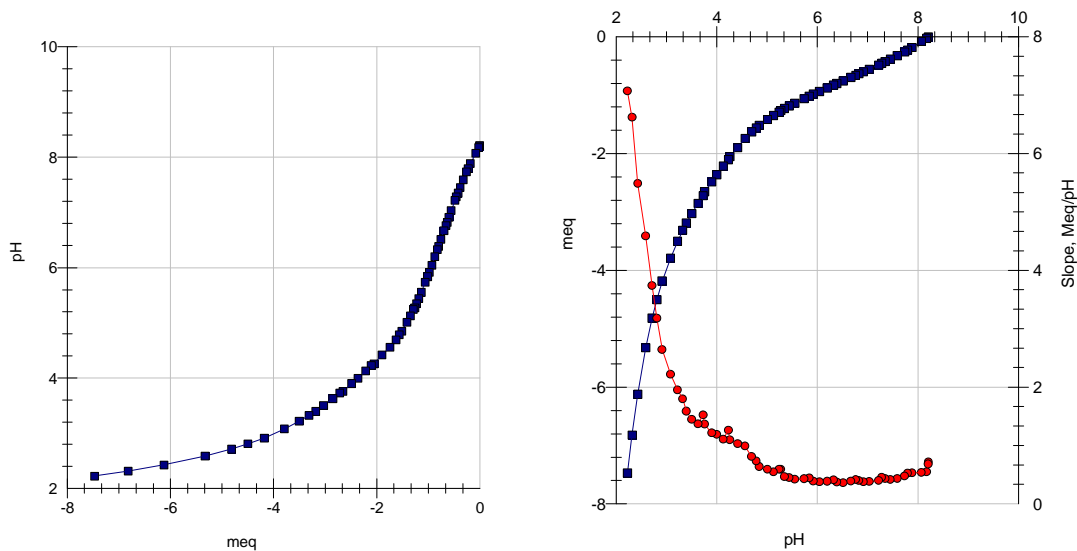
Min, pH        2.0  
 Max, pH        10  
 Points         17  
 Sweeps         1

The reagents installed in the burette are shown in the Reagents frame.

Set the Equilibrium criteria as shown below. These are all default values, except for the value for Drift which is set so as to require a better equilibrium condition at each addition point. The improved equilibrium yields somewhat smoother titration curves, which is important when doing this colloid titration experiment. ??

Next we need to define the experiment protocol on the Home page. In this simple colloid titration we do not measure PSD, zeta potential or conductivity, so these boxes are unchecked. Define the Sample in the normal manner. Uncheck Run Time Analysis since we do not need to do any analysis. The Home page should now appear as below.

Content				Density	SSspeed
%wt	%vl	gm	ml		
90.02	97.29	106.7	107.02	997	1497.
9.98	2.71	11.83	2.98	3.97	6000.
		118.53	110.	1.078	1459.5



Now we are ready to do the titration. Click on the **Run** button. After the experiment starts, the Titration form will display the results of the titration experiment. Click on Titration on the status bar at the bottom of the screen to make the Titration form visible.

When the experiment is complete, the plot of pH vs Meq will appear as shown at right.??

For such colloid titrations it is more useful to display the data somewhat differently, as meq vs pH. To switch the axes, click on **Options – Plot ml or Meq vs pH**. The display will then appear as shown in **Error! Reference source not found.** . When displaying data in this way, it is also useful to see a plot of the derivative of pH/meq which is shown by the red overlay curve. If you do not want to see the derivative curve, click on the menu item Options - Overlay Meq/pH slope. ??

When the experiment is complete, the titration data shown in these plots, along with other related data, can be permanently saved to a file by clicking on **File - Save last experiment to Excel file**. The saved files can be inspected by clicking on **File Inspect Excel files**. The filename for these pH titration files consist of date/time stamp followed by the letter “H”. These files are in csv format (comma separated values) which can be directly viewed by Excel by simply double clicking on the file name. The file contains a header which contains information about the sample and the experimental protocol.

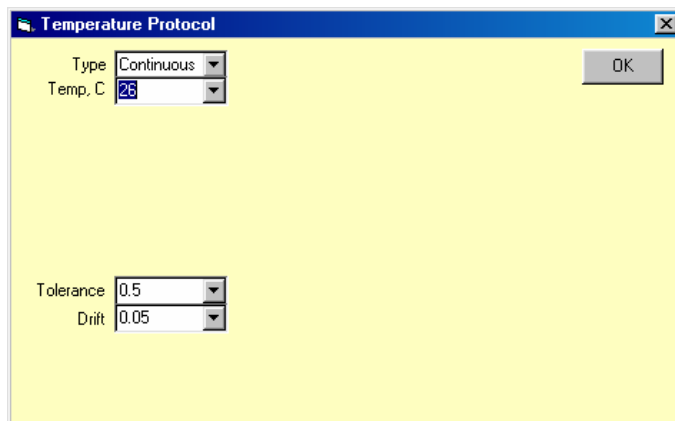
### ***Continuous temperature control of sample***

If your system is equipped with temperature control option, you can program the instrument to maintain a constant sample temperature.



The temperature control is achieved using a special heater probe that is inserted through the top sample cap such that when the plug is installed the heating element is immersed in the sample. The heater probe installed in the top cap is shown at right.

As a demonstration we will just use water for our sample. Fill the chamber with water in the normal manner. Then place the top plug with the heater in place.



On the Home page, double click on the Temperature option. The Temperature Protocol will open as illustrated at right.

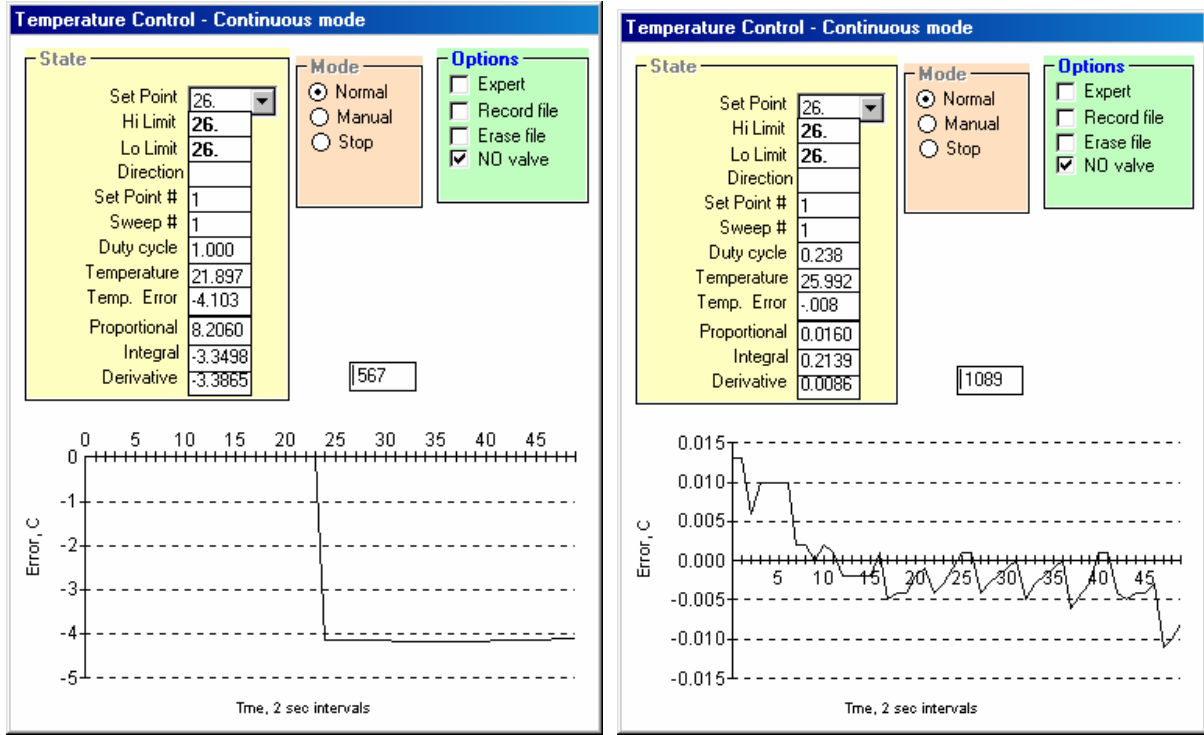
Select type **Continuous**

Since the temperature is controlled using only a heating element, the temperature can only be maintained at some temperature somewhat above room temperature, typically at least three degrees above room temperature. Accordingly, set the Temp value to three degrees above ambient, for example

Set temp,C **26**

Leave the Tolerance and Drift values set to default values.

Click **OK** to close the Temperature Protocol form.



The Temperature Control form will open automatically. The graph at the bottom of the Temperature Control form shows the error between the present temperature and the setpoint that you previously selected on the Temperature Protocol form. A typical plot of the temperature error after a short time of operation is shown in figure to right. In this case the set point temperature is 26 C, the actual temperature is 21.897 and the Temperature error is thus -4.103 which is the value plotted on the graph.

The temperature is adjusted by controlling the duty ratio of the voltage applied to the heater. A duty ratio of 1.0 means the heater is on continuously. A duty cycle of 0.1 means that the voltage is on only 10% of the time.

After several minutes the temperature of the sample will reach a steady state condition. The temperature will fluctuate somewhat about the setpoint value, typically no more than +/- 0.05 degrees C. A typical plot of the fluctuation after the temperature controller has been operating for some time is shown at right.

When you no longer want to maintain temperature you must again double click on the Temp option on the home page, and click on Type **None** on the Temperature Protocol.

**Warning: The heater must always be immersed in a sample when the Temperature Control is in the Continuous mode, else the heater will overheat. The heater has a built in thermal fuse that will permanently shut off the heating element if it overheats due to being removed from sample without shutting down the control.**

### ***Ramp Temperature Protocol using 10 wt% Silica Ludox sample***

Whereas the Continuous type temperature control works constantly, whether an experiment is in progress or not, the Ramp and Stat mode controls temperature only if an experiment is running.

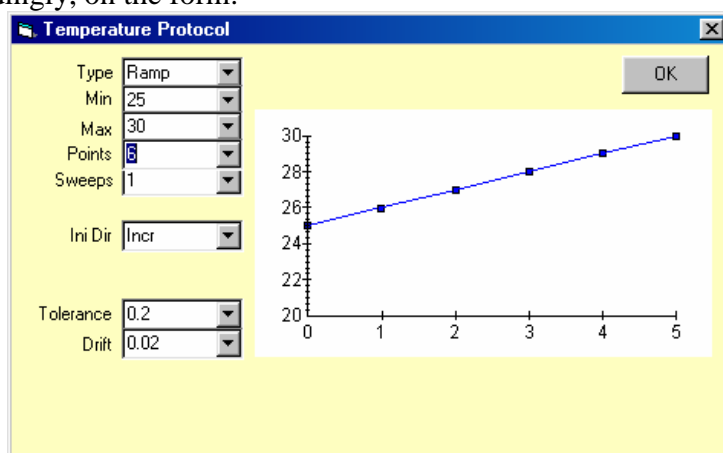
**Caution: It is safer to use the Ramp or Stat mode, rather than Continuous mode, since one is guaranteed that after the experiment is complete the heater will automatically be turned off.**

As a demonstration we will use a 10 wt% silica Ludox dispersion. Fill the chamber with a sample in the normal manner. Place the top plug with the heater in place.

On the Home page, double click on the Temp option. The Temperature Protocol will open. Select the Ramp Type

Since the temperature controller uses a heating element, the temperature can only be set to temperatures somewhat above room temperature, typically at least 2 degrees above room ambient.

For this example we will make measurements from 25 to 30 degrees C in steps of 1 degree. Accordingly, on the form:



Select Type    **Ramp**  
Min            **25**  
Max            **30**  
Points        **6**  
Sweeps        **1**  
IniDir        **Incr**

The Temperature Protocol for this Ramp experiment would then appear as shown at right.

Leave the Tolerance and Drift values set to default values. The Tolerance value of 0.2 specifies that the measurement at each temperature set point will not start till the temperature is within 0.2 degrees C of the selected set point. The Drift value of 0.02 degrees C/sec specifies further that the measurement will not start at any setpoint till the rate of change of temperature is less than that amount.\*

Click **OK** to close the Temperature Protocol form.

The Temperature Control form will not appear immediately as before with Continuous type control, and in fact will not open until you begin the experiment by clicking the **Run** button on the Home page.

For this experiment we will measure the attenuation, zeta potential and conductivity of a 10 wt% silica Ludox sample. (Depending on your system configuration you may only be able to measure some subset of these properties)

When you are ready to proceed with the experiment, click the Run button. Shortly thereafter the Temperature Controller can be made visible by clicking on its icon on the status bar at the bottom of the screen.

The heater is controlled by varying the duty ratio of the voltage applied to it. A duty ratio of 1.0 means the heater is on continuously. A duty cycle of 0.1 means that it is on just 10% of the time.

When the experiment is complete, the heater is turned off and the temperature is therefore no longer controlled.

## Calibrations

There are several menu items in the Home page menu Calibrations. This section describes purpose and function of all these items.

### *Temperature calibration*

The temperature probe is calibrated against an external standard precision thermometer. To perform this calibration, click on Calibration – Temperature on the Home page. Then simply follow the directions shown on the screen.

The screenshot shows a software window titled "Temperature Probe Calibration". It contains the following text: "Empty chamber. Fill chamber with hot water having measured temperature of 40 to 50 C. Make sure stirrer is on and working properly. Insert thermometer having 0.1 precision into sample. Make sure to immerse thermometer to correct depth, typically 76 mm. Click on Next when ready." To the right of this text are "Next" and "Cancel" buttons. Below the text is a table with two columns: "New" and "Old". The rows are "Temperature Probe Response" and "Temperature Probe Offset". The "New" column has two empty input boxes, and the "Old" column has the values "1.060" and "-2.544". Below the table is a section titled "Data" with a table with three columns: "Test sample", "Measured", and "Actual". The "Test sample" column has the value "Hot", the "Measured" column has an input box containing "26.295", and the "Actual" column is empty.

Temperature Probe Calibration		
Empty chamber. Fill chamber with hot water having measured temperature of 40 to 50 C. Make sure stirrer is on and working properly. Insert thermometer having 0.1 precision into sample. Make sure to immerse thermometer to correct depth, typically 76 mm. Click on Next when ready.		
<input type="button" value="Next"/> <input type="button" value="Cancel"/>		
	<b>New</b>	Old
Temperature Probe Response	<input type="text"/>	1.060
Temperature Probe Offset	<input type="text"/>	-2.544
<b>Data</b>		
Test sample	Measured	Actual
Hot	<input type="text" value="26.295"/>	

### *pH calibration*

The pH probe is calibrated against one or more pH buffer solutions. The characteristics of the 13 buffers recognized by the US National Institute of Standards Technology (NIST) are stored in this calibration software. In default, the commonly used pH 4-7-10 buffers are used. However the user can use any combination of as many of the 13 buffers as needed.

To perform this pH calibration, click on the menu item Calibrate- pH on the Home page.

The Buffer Temperature form shown below will appear. Accurate pH calibration depends on knowing the temperature of the buffer solution. Typically you will be calibrating the pH by dipping the pH probe into a small beaker of buffer solution. In this case check the temperature of the buffer, probably the same as the room temperature, enter this temperature in the box, and click OK. ( If the temperature probe is also in the buffer solution, you can simply click Cancel and the measured temperature will be used instead of your manual input).



**Buffer Temperature**

Enter temperature of pH buffer if you do not use measured temperature in chamber and then click OK. Click Cancel to use measured temperature in chamber

22

OK Cancel

After the Buffer Temperature form closes, the pH calibration form appears, as shown below. Three commonly used buffers are pre-selected. If you want to use a different buffer in place of one of the default buffers, then click on the row containing the buffer you want to delete and press the delete buffer button. Then select a replacement buffer from the “Select another buffer?” list as shown in Figure below.

If you want to add an additional buffer, just click on an empty row and then select the additional buffer from the pull-down list. You can use as many buffers as you like.

**pH Calibration**

Calibration raw data Select another buffer? ▾

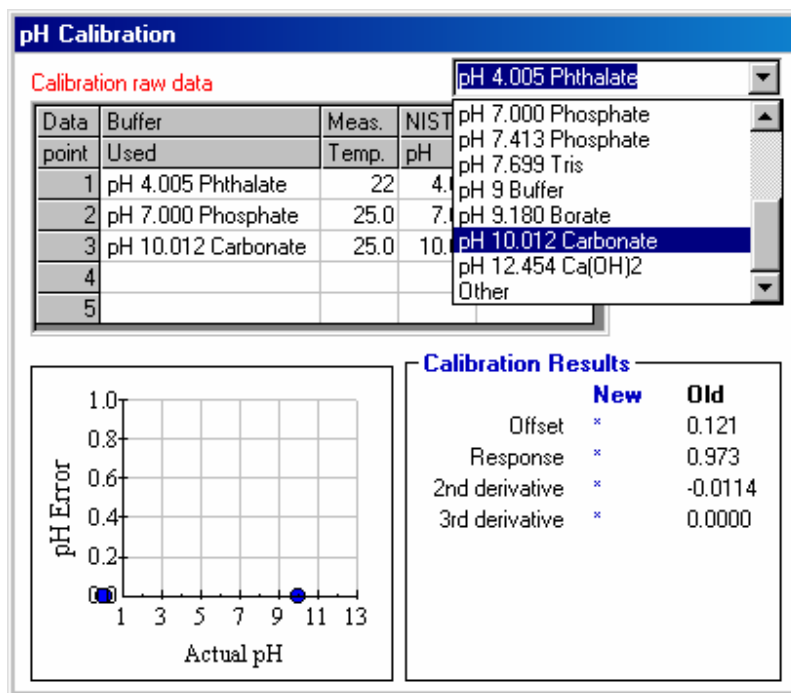
Data point	Buffer Used	Meas. Temp.	NIST pH	Meas. pH
1	pH 4.005 Phthalate	25.0	4.01	
2	pH 7.000 Phosphate	25.0	7.00	
3	pH 10.012 Carbonate	25.0	10.01	
4				
5				

Cancel  
Measure pH  
Delete buffer

Actual pH

**Calibration Results**

	New	Old
Offset	*	0.121
Response	*	0.973
2nd derivative	*	-0.0114
3rd derivative	*	0.0000



Prepare a small sample cup of each of the selected buffers. Fill each cup to a depth of at least 3 cm. It is important that the buffer is stirred during calibration in order that the value reaches an equilibrium value. Although the buffer can be stirred with the probe itself, some prefer to use a small stir bar in the sample cup. If you stir the sample with the probe, be careful not to break the probe by hitting the sides of the cup.

To start calibration, click on the row containing the buffer you want to use first. Dip the pH probe in this buffer. Make sure the buffer is stirred, one way or the other, while you measure the pH. Depress the Measure pH button to measure the pH. Press again every few seconds until the value is no longer changing.

When a stable value has been reached for one buffer, move on to the next. Click on the row containing the buffer you want to use next. You can do the buffers in any sequence. Rinse the pH probe between buffers with water and wipe dry with a soft tissue.

When you are though measuring the pH of each of the buffers, click on the “Calibrate” button. The deviation of the probe from a theoretically perfect probe is shown in the plot of pH-Error vs. actual pH shown at the bottom left corner of the form. The old and new values of the calibration constants related to the pH probe are shown to the right.

Click on the Save button to permanently save these calibration constants.

### ***Aqueous Conductivity calibration***

To calibrate the aqueous conductivity probe, first click on the menu item Calibrate – Conductivity. The Conductivity Probe Calibration form will appear as shown in Figure below. Follow the step by step instructions on the form.

### Conductivity Probe Calibration

Step 1) Empty chamber. Fill chamber with 0.01 M KCl standard and stir 10 sec. Empty chamber, again fill with 0.01 MKCl, and stir. Check that stirrer is working properly. Click on OK when ready to measure

OK

Cancel

#### Measured Data

Standard	Temp.	Actual		Measured K
		K	$\epsilon$	
0.01 M KCl	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
0.1 M KCl	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>

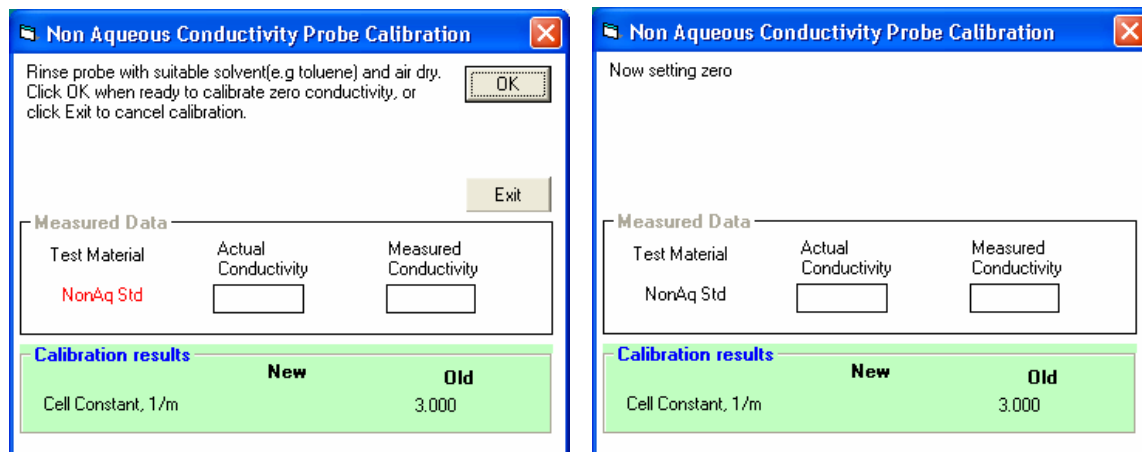
#### Calibration results

	New	Old
Response		7.136E+04
Linearity		0.4698

## Non Aqueous Conductivity Calibration

On the Homepage: Click >> **Calibration** >> **Non aqueous conductivity**

The window shown at right will appear.



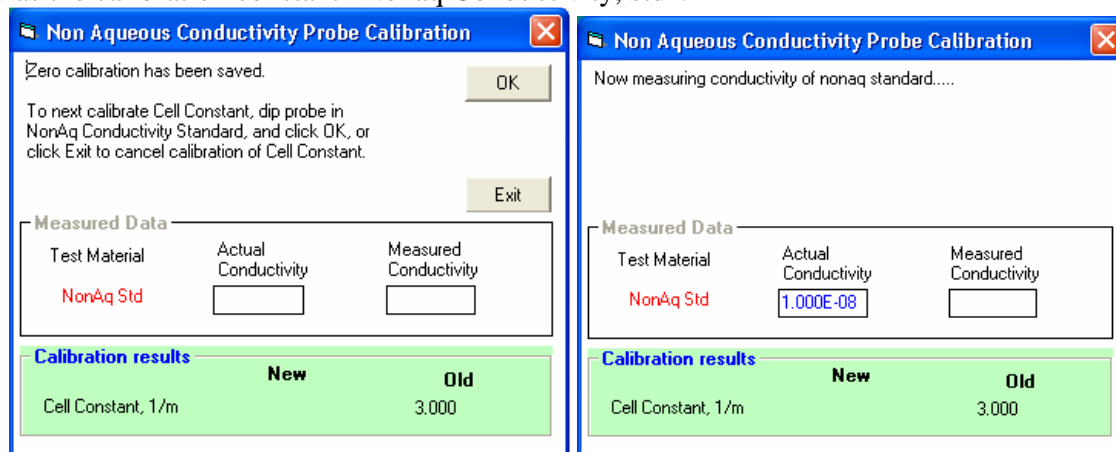
Follow instructions concerning rinsing the probe and click **OK** when ready.

The window shown at right will appear.

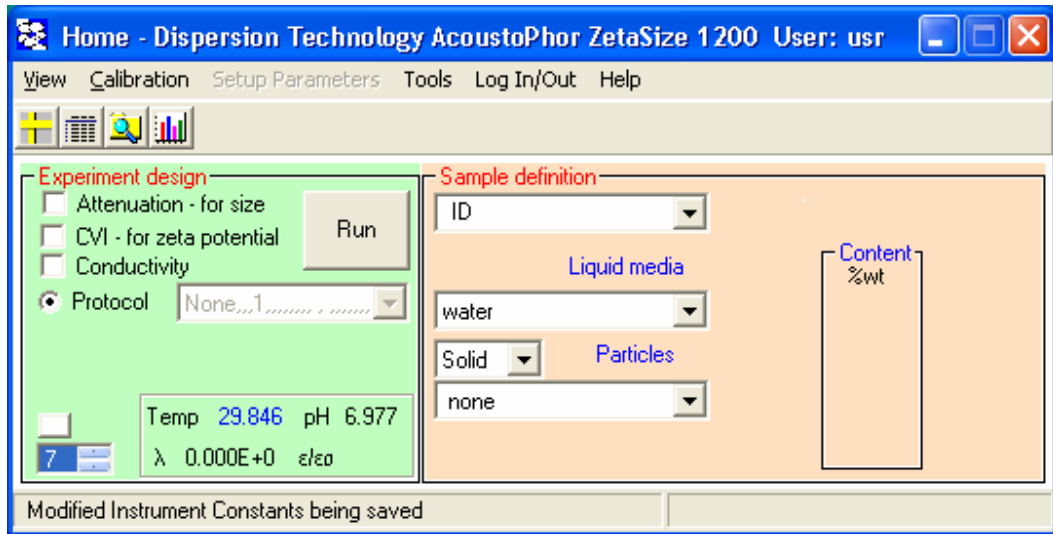
When the measurement is complete, the window shown at right will appear.

If you just want to zero the probe, then click **Exit**

However, in order to continue with a calibration of the cell constant you must have a suitable calibration fluid of known conductivity and the conductivity value must have been previously entered as the calibration constant "NonAq Conductivity, std".



If a standard fluid is available, and the constant recorded, you can continue to measure the Cell Constant for the conductivity probe by clicking **OK**. The window shown at right will appear.

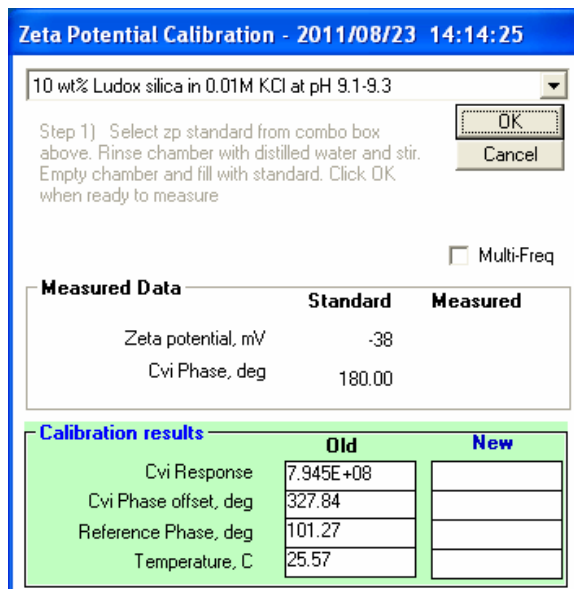


### *Zeta potential calibration*

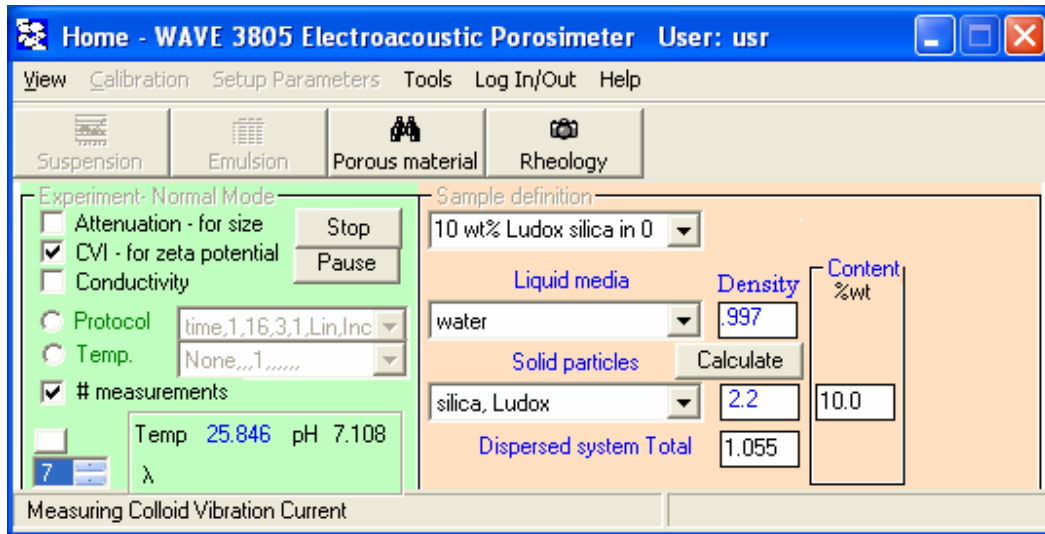
The zeta potential is calibrated using a standard colloid having known zeta potential. The DT-1200 employs a standard 10 wt% colloidal silica that is prepared from a concentrated 50 wt% slurry by diluting it with 0.01 M KCl media.

The calibration procedure assumes that the last measurement was of such a zp standard, and that only zp was measured ( i.e. not in combination with attenuation or conductivity).

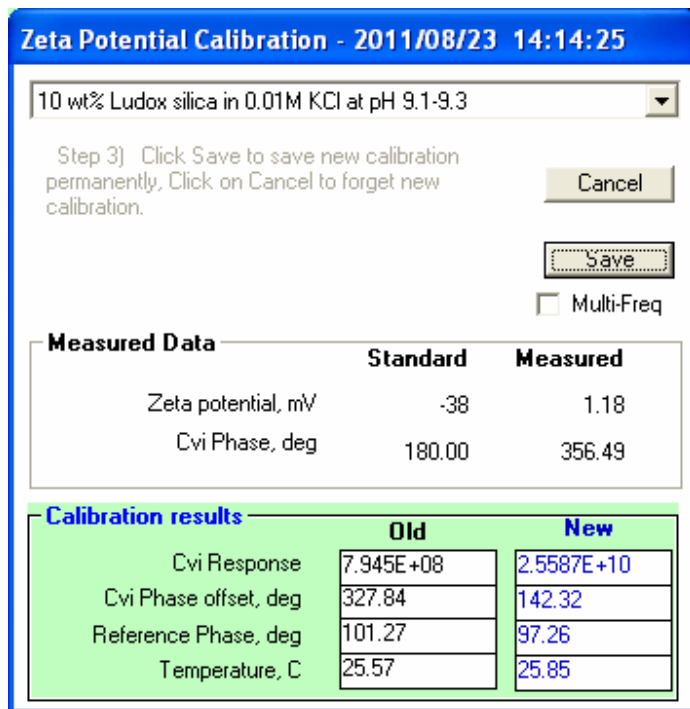
Following this measurement, on the home page click on Calibrate- Zeta potential. The Zeta Calibration form will appear as shown in Figure below. The measured value will normally be fairly close to the calibration value if the probe has not been replaced.



When you Click OK, Home Page would change Setup as shown below:



Then, after measurement finished, Calibration form would show results of measurement.



Click on the Calculate calibration button to compute the new calibration constants. If the calibration is successful, the Save button will appear, as shown in Figure below. Click on the Save button to permanently save the new constants and exit this calibration form

## ***Porous body***

Characterization of the porous body includes measurement of porosity with aqueous conductivity probe and pore size plus zeta potential with zeta probe. This calibration function allows user introduction of multiplier that would eventually match measured values with independently known.

This calibration procedure is less useful for porosity measurement, which is absolute when performed with high frequency conductivity.

In the case of pore size it is absolutely required. Calculation of pore size from the measured electroacoustic signal completely relies on independently measured value. This independent value can be gained by mercury porosimetry or adsorption methods.

Zeta potential calibration for porous body is also essential because of unknown visco-elastic properties of the porous body skeleton. Unfortunately independent measurement of zeta potential in porous body is practically unavailable.

After clicking on the Calibration menu item the following form appears. User should select parameter for calibration.

Calibration Porous body

Select what type of porous body characteristics you want to calibrate.

Porosity from Conductivity

Pore size for known porosity from electroacoustics

Zeta potential for known porosity

New constant  = Expected Value

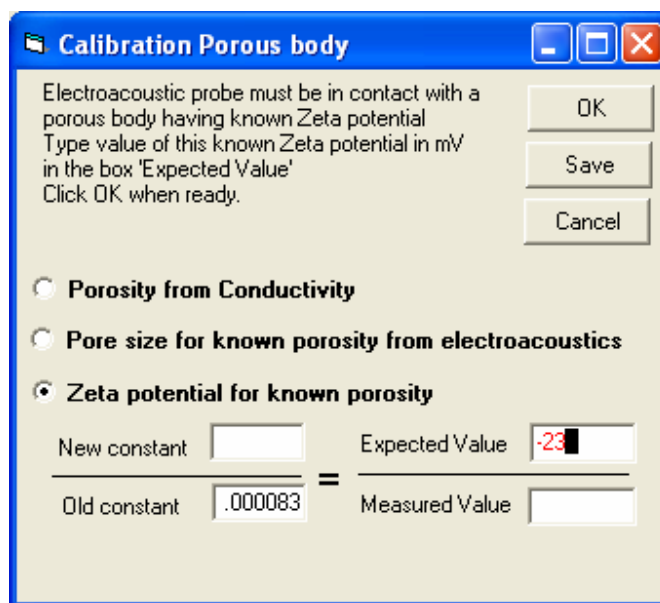
Old constant  = Measured Value

OK

Save

Cancel

Next step is placing independently known value of this parameter in the box Expected Value as shown on Figure below. Click OK runs measurement and the measured value would be displayed in the box Measured Value, as well as the new value of calibration constant.

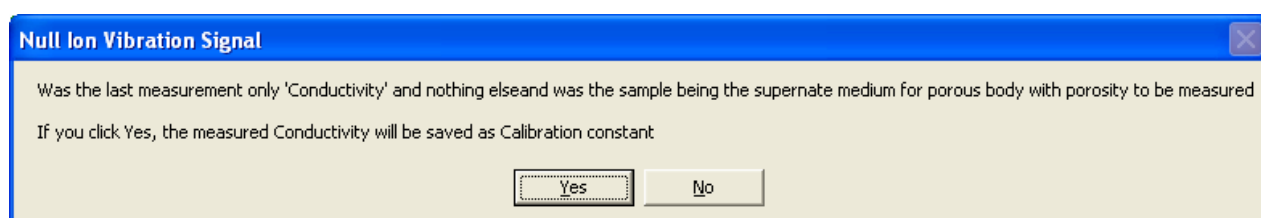


Click Save for saving the new constant value.

We recommend making a measurement with this new constant value for verifying that this procedure has worked.

### ***Save conductivity of supernate***

This function is required for porosity measurement. Conductivity of supernate is being saved as calibration constant. This number is used as reference for measured conductivity of the porous body when porosity is calculated. Porosity is ratio of porous body conductivity to the supernate conductivity with Maxwell – Wagner correction, which might cause about 10% correction.



Click Yes for saving measured conductivity as supernate reference value.

### ***Save last CVI as Ion Vibration Current***

This calibration item is required for measuring zeta potential at the conditions when ions contribute to the measured electroacoustic signal comparably with particles or pores. This might occur either at high ionic strength or at rather low surface area. Procedure associated with this calibration item allows measurement of Ion Vibration Current (IVI) and saving it as calibration constant for further subtraction from the measured signal for dispersion.

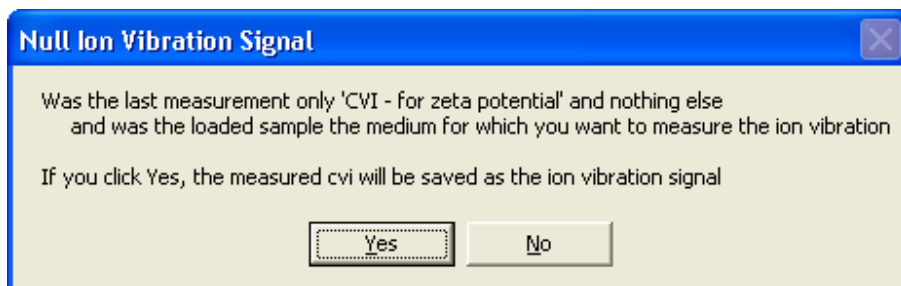
**WARNING:** This calibration should not be mixed with the following item – background electroacoustic noise subtraction. That noise is created by electronics, not by ions.

In order to save IVI, probe must be placed in the equilibrium supernate with no particles and electroacoustic signal must be measured.

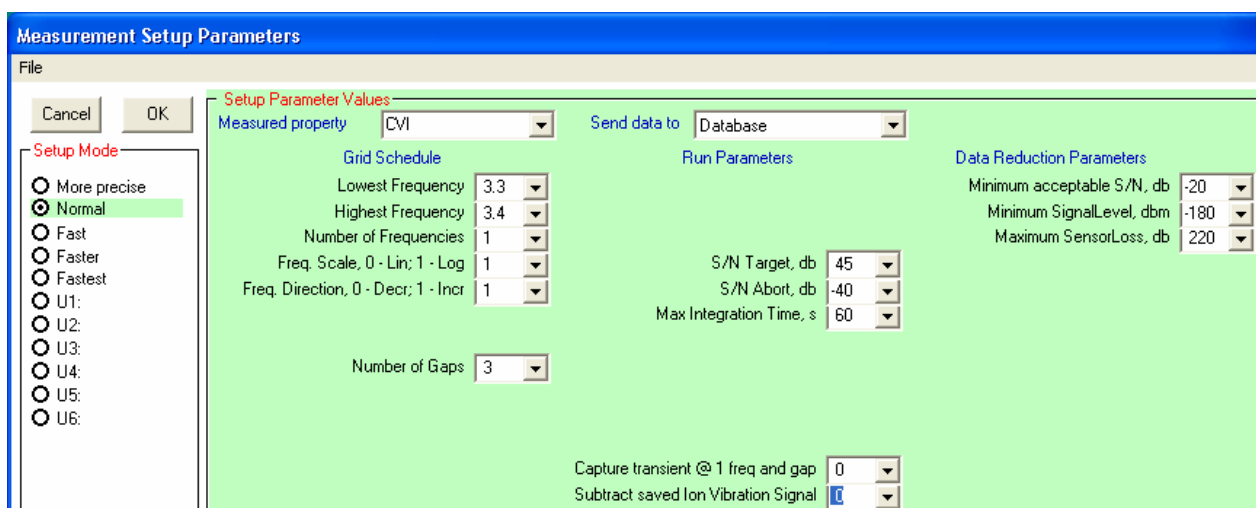
Then user goto Home page, Calibrations and click on this menu item.

Following message appears.





If all conditions stated in the message are valid, then click Yes and IVI would be saved. In order to subtract this IVI from the coming zeta potential measurements user should goto Home page, Setup parameters. The following form appears, IF previously user checked CVI on the Home page.



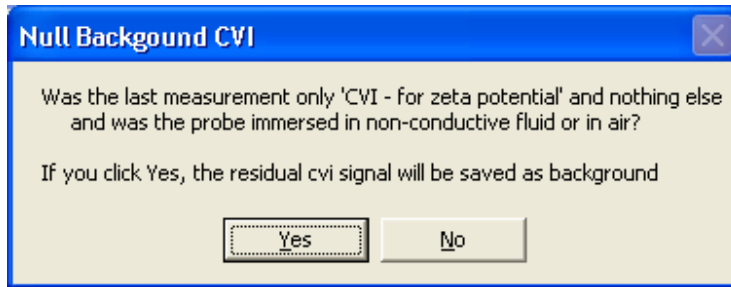
There is a box "Subtract saved Ion Vibration Signal" on the very bottom of this form. If one select value 0 for this box, IVI will NOT be subtracted. If one select value 1 for this box, IVI will be subtracted.

**WARNING:** this subtraction is subtraction of vectors, which is quite different than scalars. It might lead to increasing of the magnitude.

### ***Save last CVI as background***

There is background electroacoustic signal created by electronics and the probe. It is rather small, on scale of 20000 CVI units, which is fraction of % of the signal generated by 10% wt silica Ludox. This signal is negligible in aqueous samples, but becomes important in low conducting media.

In order to measure this signal the probe must be placed in the air or non-polar liquid. After measurement done, click on this item in Calibration menu. Following form appears.



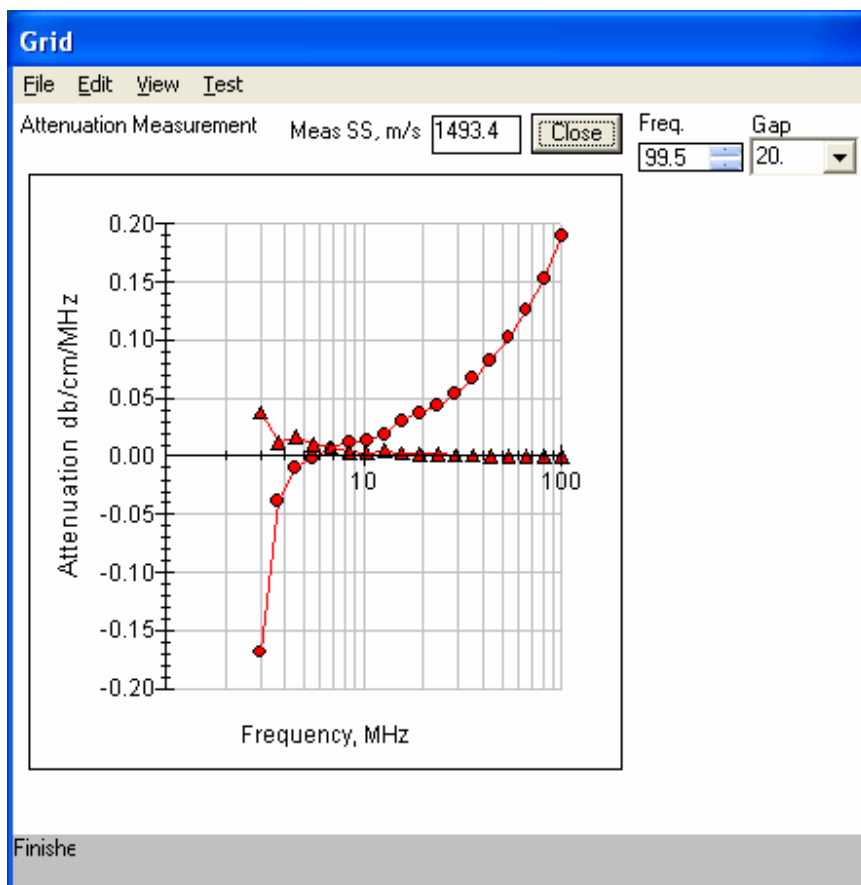
If all conditions specified in this message are satisfied, click Yes.

When this signal measured and saved it would be subtracted from all further measurements.  
**WARNING:** Do not mix this signal with Ion Vibration Current.

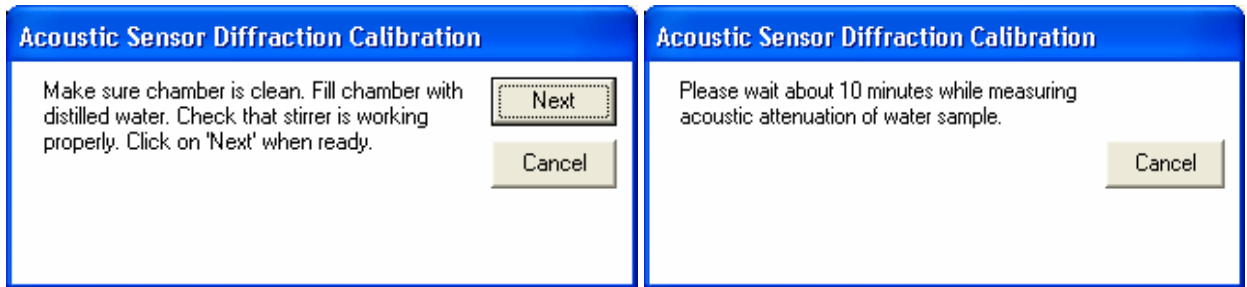
### *Acoustic geometry*

Low frequency attenuation measurement is affected by diffraction effects. This calibration procedure allows us to null them out.

This procedure is important when attenuation of water looks like shown below. Noisy and/or negative values of attenuation at low frequency is result of diffraction effect.




In order to calibrate out this erroneous attenuation at low frequency, goto Home page, Calibration and click on this menu item. You will see a set of instructions. Follow them.



After measurement of water finished, the following list of the new calibration constants appears.

**Details of Acoustic Sensor Diffraction** ✖

 09-23-10 10:48  
Data used for linear regression

p	f	Freq(p)	Atten(p)
1	0	00.0	000.000
2	0	00.0	000.000
3	0	00.0	000.000
5	13	35.5	000.066
6	14	43.7	000.080
7	15	53.6	000.098
8	16	65.9	000.119
9	17	81.0	000.146
10	18	99.5	000.181

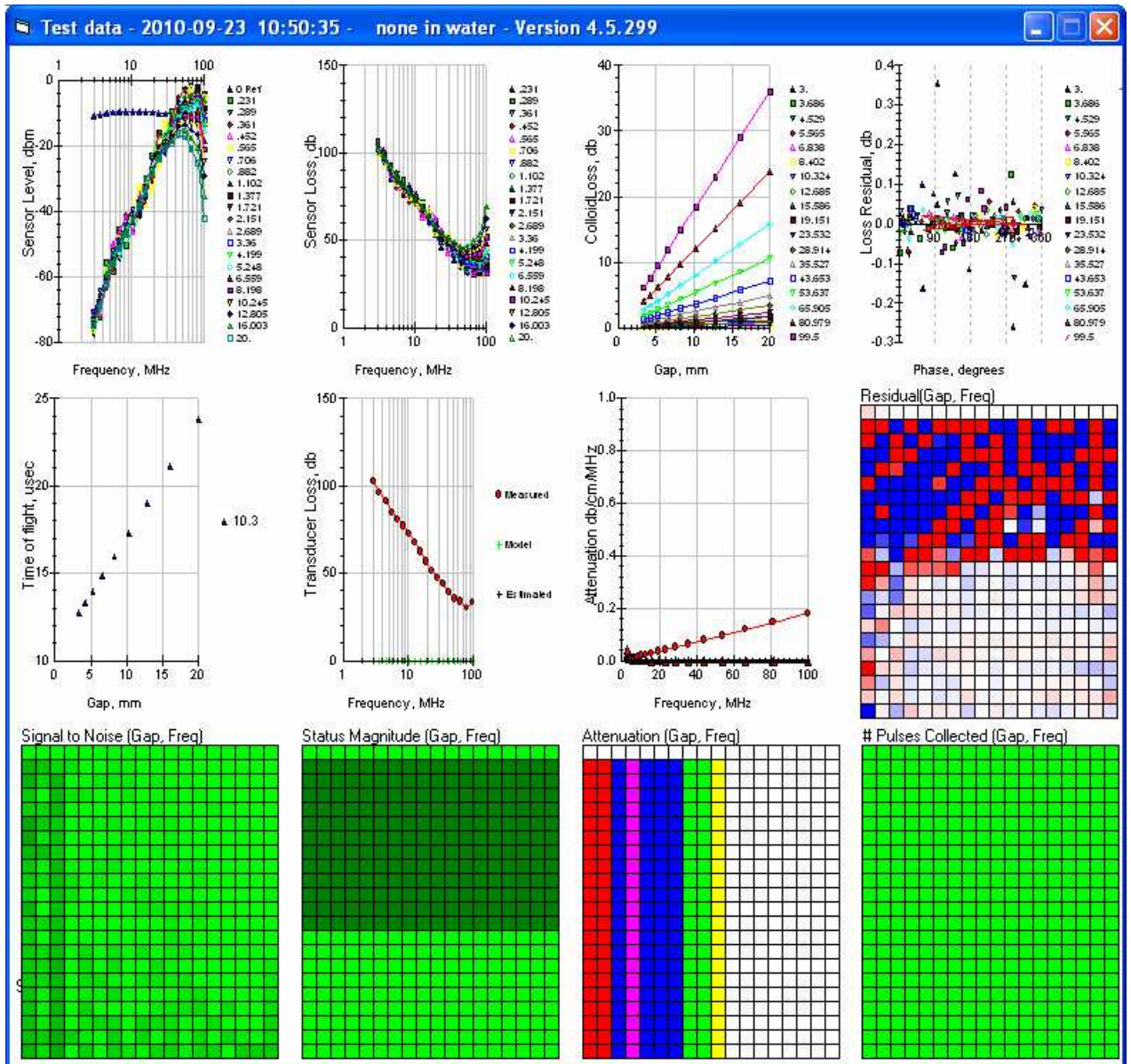
Diffraction Calibration Constants

f	Freq(f)	Atten(f)	Resid(f)
1	03.0	.001	-0.0051
2	03.7	.087	+0.0805
3	04.5	.001	-0.0076
4	05.6	.030	+0.0192
5	06.8	.010	-0.0024
6	08.4	-.006	-0.0219
7	10.3	-.003	-0.0221
8	12.7	.020	-0.0033
9	15.6	.043	+0.0141
10	19.2	.040	+0.0050
11	23.5	.046	+0.0030
12	28.9	.055	+0.0026
13	35.5	.066	+0.0017
14	43.7	.080	+0.0009
15	53.6	.098	+0.0002
16	65.9	.119	-0.0004
17	81.0	.146	-0.0010
18	99.5	.181	+0.0000

Atten, db/(cm x Mhz^2)	0.00181
Offset, db/cm/Mhz	0.000
Avg Abs Resid < 18 Mhz	0.0196
Avg Abs Resid > 18 Mhz	0.0015
Avg Abs Resid all freq	0.0106

If you click OK, the new values will be saved to Calibration table.

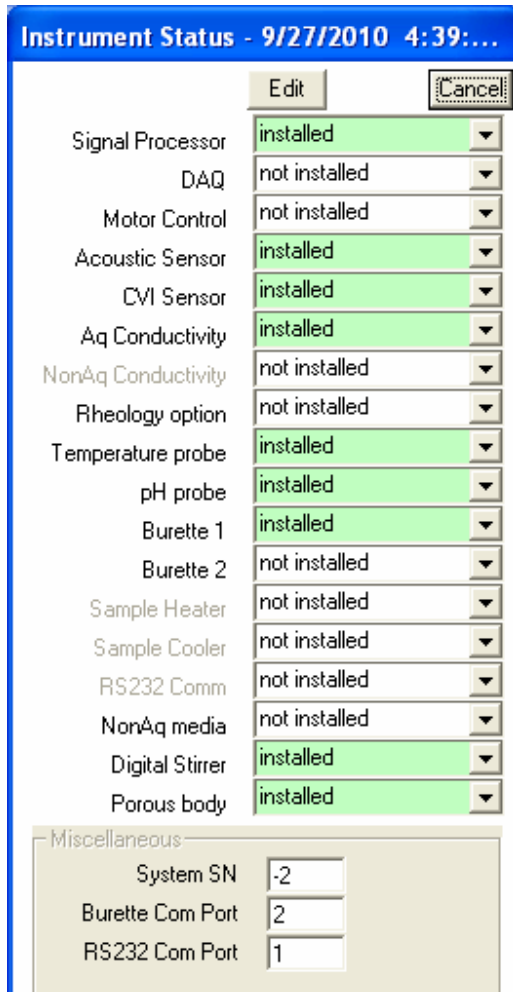
For double check one can look at the complete measurement report on Grid raw data for. Goto: Home page, View, Grid raw data, View, All graphs. You will see the picture as below.



Attenuation is a straight line. This is confirmation that Acoustic geometry calibration has succeeded.

### ***Instrument status***

Instrument status form demonstrates what options and sensors are installed in particular instrument. DTI agents can install or uninstall specific functions using this form.



### ***Single gap acoustic sensor calibration***

It is possible to make Attenuation measurements using an acoustic sensor that has a fixed gap between the two sensors, as opposed to the normal variable gap sensor. Making such measurements requires three calibration steps before we can make a measurement at a fixed gap. It is useful for samples that are affected by the gap motion.

You should change open Setup parameters when only Attenuation check box is checked on the Home page. Then you change “Number of Gaps” to 1 and “Smallest gap” to the gap of your choice. “Largest gap” must be a bit larger, as shown on Figure below.

Measurement will use parameters of the Acoustic sensor from the previous measurement. That is why previous measurement must be full gap sweep with water.

Selection of the optimum gap for shear sensitive sample is not simple. We recommend that you perform initially full gap sweep measurement. Then you open Grid and look at the signal-to-noise grad table. You should select the gap that provides data for most frequencies.

**Measurement Setup Parameters**

File

Cancel OK

Setup Parameter Values

Measured property: Attenuation Send data to: Database

**Setup Mode**

- More precise
- Normal
- Fast
- Faster
- Fastest
- U1:
- U2:
- U3:
- U4:
- U5:
- U6:

**Grid Schedule**

Lowest Frequency: 3

Highest Frequency: 99.5

Number of Frequencies: 18

Freq. Scale, 0 - Lin; 1 - Log: 1

Freq. Direction, 0 - Decr; 1 - Incr: 1

Smallest Gap, mm: .5

Largest Gap, mm: .51

Number of Gaps: 1

Gap Scale, 0 - Lin; 1 - Log: 1

Gap Direction, 0 - Decr; 1 - Incr: 1

**Run Parameters**

RunLevel: 1

Gap Equilibrate Time, s: .1

S/N Target, db: 40

S/N Abort, db: 10

Max Integration Time, s: 5

GapMode, 0-Norm, 1-Single, 2-Fixed: 0

Target # gaps each freq: 5

Capture transient @ 1 freq and gap: 0

**Data Reduction Parameters**

Minimum acceptable S/N, db: 35

Minimum SignalLevel, dbm: -120

Maximum SensorLoss, db: 180

Maximum ColloidLoss, db: 60

Max ColloidAtten Err, Db/Cm: 100

Max ColloidAtten Err, Db/Cm/MHz: 1

Max ColloidAtten Err, %: 20

Max SdDev in gap regression, db: .9

Minimum # of Gaps: 3

Bimodal Threshold: 3

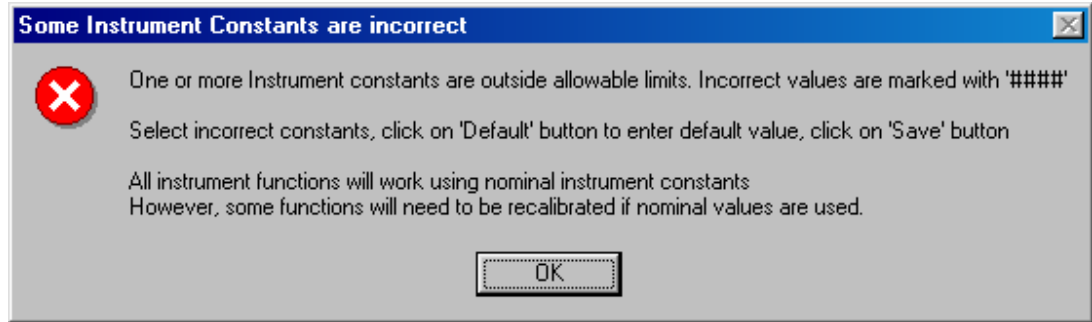
### *Viewing Calibration Constants*

The various calibration procedures, described in the above sections, calculate various calibration constants that are then saved to disk memory so that they will be available each time the machine is started. Normally you will not need to look at these calibration constants, but you can if you wish. On the home page, click on Calibration- Constants. A form similar to that shown in Figure will appear. The constants are ordered alphabetically. Three columns display the present value, the minimum and maximum value for each constant.

Calibration Constants				
1E+10	Cancel	Save	Default	Print
Calibration Constant	Value	Min Value	Max Value	Error
Acoustic sensor PiezoLoss f 1	9.302E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f 2	8.868E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f 3	8.053E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f 4	7.394E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f 5	6.968E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f 6	6.509E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f 7	6.057E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f 8	5.512E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f 9	5.158E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f10	4.781E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f11	4.355E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f12	4.030E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f13	3.858E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f14	4.127E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f15	4.974E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f16	6.897E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f17	9.281E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f18	5.654E+01	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f19	0.000E+00	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f20	0.000E+00	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f21	0.000E+00	0.000E+00	1.500E+02	
Conductivity probe extra dielectric	0.000E+00	-1.000E+03	1.000E+04	
Conductivity probe Linearity	4.698E-01	-1.000E+01	1.000E+01	
Conductivity probe Phase offset, deg	4.000E+00	-2.000E+01	2.000E+01	
Conductivity probe Response	7.136E+04	1.000E+02	3.000E+06	
ElectroAcoustic Buffer rod Delay, s	4.600E-06	0.000E+00	2.500E-05	
ElectroAcoustic Buffer rod Impedance, ?	5.630E-06	5.000E-06	6.000E-06	
ElectroAcoustic Buffer rod Length, m	0.000E+00	0.000E+00	2.000E-02	
ElectroAcoustic Buffer rod loss, db	0.000E+00	0.000E+00	3.000E+00	
ElectroAcoustic Buffer rod loss, db/cm/Mhz	0.000E+00	0.000E+00	3.000E-02	
ElectroAcoustic Buffer rod Loss, db/cm/MHz^2	0.000E+00	0.000E+00	2.000E+00	
ElectroAcoustic Buffer rod loss, db/cm/MHz^2	0.000E+00	0.000E+00	3.000E-03	
ElectroAcoustic Gap	0.000E+00	0.000E+00	1.000E-02	
Electroacoustic offset cosine	0.000E+00	-1.000E+00	1.000E+00	
Electroacoustic offset sine	0.000E+00	-1.000E+00	1.000E+00	
ElectroAcoustic Phase 1, deg	2.664E+02	0.000E+00	3.600E+02	
ElectroAcoustic Phase 2, deg	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic Response	1.000E+10	1.000E+09	1.000E+12	

### *Error in one or more Calibration constants*

One of the tasks that the instrument performs at start-up is to check that all of the instrument constants are within nominal limits. If one or more are outside nominal limits then the form shown in Figure below will be displayed.



Click OK. Since a problem has been detected, all of the text will be red, and the offending constant or constants will be marked with “#####” at the right hand edge of the form as shown in Figure below.

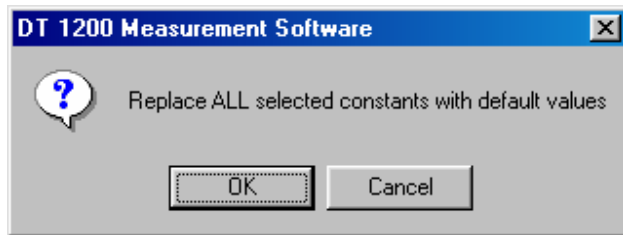
Calibration Constant	Value	Min Value	Max Value	Error
Acoustic sensor PiezoLoss f20	0.000E+00	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f21	0.000E+00	0.000E+00	1.500E+02	
Conductivity probe extra dielectric	0.000E+00	-1.000E+03	1.000E+04	
Conductivity probe Linearity	4.698E-01	-1.000E+01	1.000E+01	
Conductivity probe Phase offset, deg	4.000E+00	-2.000E+01	2.000E+01	
Conductivity probe Response	7.136E+04	1.000E+02	3.000E+06	
ElectroAcoustic Buffer rod Delay, s	4.600E-06	0.000E+00	2.500E-05	
ElectroAcoustic Buffer rod Impedance, ?	5.630E-06	5.000E-06	6.000E-06	
ElectroAcoustic Buffer rod Length, m	0.000E+00	0.000E+00	2.000E-02	
ElectroAcoustic Buffer rod loss, db	0.000E+00	0.000E+00	3.000E+00	
ElectroAcoustic Buffer rod loss, db/cm/Mhz	0.000E+00	0.000E+00	3.000E-02	
ElectroAcoustic Buffer rod Loss, db/cm/MHz^2	0.000E+00	0.000E+00	2.000E+00	
ElectroAcoustic Buffer rod loss, db/cm/MHz^2	0.000E+00	0.000E+00	3.000E-03	
ElectroAcoustic Gap	0.000E+00	0.000E+00	1.000E-02	
Electroacoustic offset cosine	0.000E+00	-1.000E+00	1.000E+00	
Electroacoustic offset sine	0.000E+00	-1.000E+00	1.000E+00	
ElectroAcoustic Phase 1, deg	2.664E+02	0.000E+00	3.600E+02	
ElectroAcoustic Phase 2, deg	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic Response	1.669E+18	1.000E+09	1.000E+12	#####
ElectroAcoustic Transmit delay, s	2.200E-06	1.800E-06	5.000E-06	
ElectroAcoustic phase f 1	2.362E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 2	3.122E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 3	1.192E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 4	2.627E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 5	8.522E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f 6	3.171E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 7	2.179E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 8	1.875E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 9	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f10	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f11	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f12	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f13	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f14	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f15	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f16	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f17	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f18	0.000E+00	0.000E+00	3.600E+02	



If necessary, scroll down till you find the offending constant. Click on the line containing the offending constant. If more than one constant is in error use the control key in combination with the left mouse click to select all the offending constants. Then click on the Default button.

Calibration Constants				
1.669E+18	Cancel	Save	Default	Print
Calibration Constant	Value	Min Value	Max Value	Error
Acoustic sensor PiezoLoss f20	0.000E+00	0.000E+00	1.500E+02	
Acoustic sensor PiezoLoss f21	0.000E+00	0.000E+00	1.500E+02	
Conductivity probe extra dielectric	0.000E+00	-1.000E+03	1.000E+04	
Conductivity probe Linearity	4.698E-01	-1.000E+01	1.000E+01	
Conductivity probe Phase offset, deg	4.000E+00	-2.000E+01	2.000E+01	
Conductivity probe Response	7.136E+04	1.000E+02	3.000E+06	
ElectroAcoustic Buffer rod Delay, s	4.600E-06	0.000E+00	2.500E-05	
ElectroAcoustic Buffer rod Impedance, ?	5.630E-06	5.000E-06	6.000E-06	
ElectroAcoustic Buffer rod Length, m	0.000E+00	0.000E+00	2.000E-02	
ElectroAcoustic Buffer rod loss, db	0.000E+00	0.000E+00	3.000E+00	
ElectroAcoustic Buffer rod loss, db/cm/Mhz	0.000E+00	0.000E+00	3.000E-02	
ElectroAcoustic Buffer rod Loss, db/cm/MHz^2	0.000E+00	0.000E+00	2.000E+00	
ElectroAcoustic Buffer rod loss, db/cm/MHz^2	0.000E+00	0.000E+00	3.000E-03	
ElectroAcoustic Gap	0.000E+00	0.000E+00	1.000E-02	
Electroacoustic offset cosine	0.000E+00	-1.000E+00	1.000E+00	
Electroacoustic offset sine	0.000E+00	-1.000E+00	1.000E+00	
ElectroAcoustic Phase 1, deg	2.664E+02	0.000E+00	3.600E+02	
ElectroAcoustic Phase 2, deg	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic Response	1.669E+18	1.000E+09	1.000E+12	#####
ElectroAcoustic Transmit delay, s	2.200E-06	1.800E-06	5.000E-06	
ElectroAcoustic phase f 1	2.362E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 2	3.122E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 3	1.192E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 4	2.627E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 5	8.522E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f 6	3.171E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 7	2.179E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 8	1.875E+02	0.000E+00	3.600E+02	
ElectroAcoustic phase f 9	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f10	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f11	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f12	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f13	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f14	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f15	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f16	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f17	0.000E+00	0.000E+00	3.600E+02	
ElectroAcoustic phase f18	0.000E+00	0.000E+00	3.600E+02	

The message shown in Figure below will appear. Click OK.



All of the selected constants will be replaced by default values. The form will then appear with normal black text as was shown above.

Using the default values will enable you to continue operation. However, the function that had an incorrect constant must be re-calibrated before the results will be highly accurate. For example, if you replaced a constant related to some “Electroacoustic” constant, then you will have to calibrate zeta potential.

### ***Definition of instrument Constants***

The Instrument constants are ordered alphabetically by name. The first part of name refers to the object that is being calibrated.

- Some are set by calibration procedures that user can do
- Some are set by procedures done by service
- Some are done only when instrument is built
- Some are no longer used

#### **Acoustic Sensor**

- Acoustic Sensor Diffraction Exponent
- Acoustic Sensor Diffraction Loss, db
- Acoustic Sensor Fixed Gap, m
- Acoustic Sensor Gap Compliance, m
- Acoustic Sensor Gap Stop, m
- Acoustic Sensor Recalibration freq.
- Acoustic Sensor Receive delay, s
- Acoustic Sensor Residual limit
- Acoustic Sensor Transmit delay, s
- Acoustic Sensor ZeroGap delay – FixedGap, s
- Acoustic Sensor ZeroGap delay – SingleGap, s
- Acoustic Sensor Diffraction f1 – f18
- Acoustic Sensor Gapscale
- Acoustic Sensor PiezoLoss f1 – f21

#### **Conductivity NonAq**

- Conductivity NonAq FullScale
- Conductivity NonnAq Response

#### **Conductivity probe**

- Conductivity probe Extra

Conductivity probe extra dielectric  
Conductivity probe Linearity  
Conductivity probe Phase offset, deg  
Conductivity probe Response

### **ElectroAcoustic**

ElectroAcoustic Buffer rod Delay, s  
ElectroAcoustic Buffer rod Impedance, ?  
ElectroAcoustic Buffer rod Length, m  
ElectroAcoustic Buffer rod loss, db  
ElectroAcoustic Buffer rod loss, db/cm/MHz  
ElectroAcoustic Buffer rod loss, db/cm/MHz<sup>2</sup>  
ElectroAcoustic Buffer rod Loss, db/cm/MHz<sup>2</sup>  
ElectroAcoustic Gap  
ElectroAcoustic offset cosine  
ElectroAcoustic offset sine  
ElectroAcoustic Phase 1, deg  
ElectroAcoustic Phase 2, deg  
ElectroAcoustic reflection, deg  
ElectroAcoustic temp coef, deg/C  
ElectroAcoustic Response  
ElectroAcoustic Transmit delay,s  
ElectroAcoustic calibration temp, C  
ElectroAcoustic phase f1 - f21  
ElectroAcoustic response f1 -f21  
ElectroAcoustic  
ElectroAcoustic  
ElectroAcoustic

### **Impedance probe**

Impedance probe

### **Interface**

Interface Digital Attenuator

### **pH probe**

pH probe Offset  
pH probe Response  
pH probe Second derive  
pH probe Third derive

### **Pyncnometer**

Pyncnometer volume, ml

### **Sample**

Sample Volume, ml

### **Signal Processor**

SP cable delays, s  
SP Digital Attenuator K1 – K8  
SP Offset Cos, V  
SP Offset Sin, V  
SP Quad Gain balance  
SP Quad Phase balance, deg  
SP rf delay c0, s  
SP rf delay c1, s  
SP rf gain, dB  
SP video delay, s

**Temperature probe**

Temperature probe Offset, C  
Temperature probe Response

**Wfr calibration**

Wfr calibration, conductivity reference  
Wfr calibration, standard deviation reference  
Wfr calibration, zp reference, mV

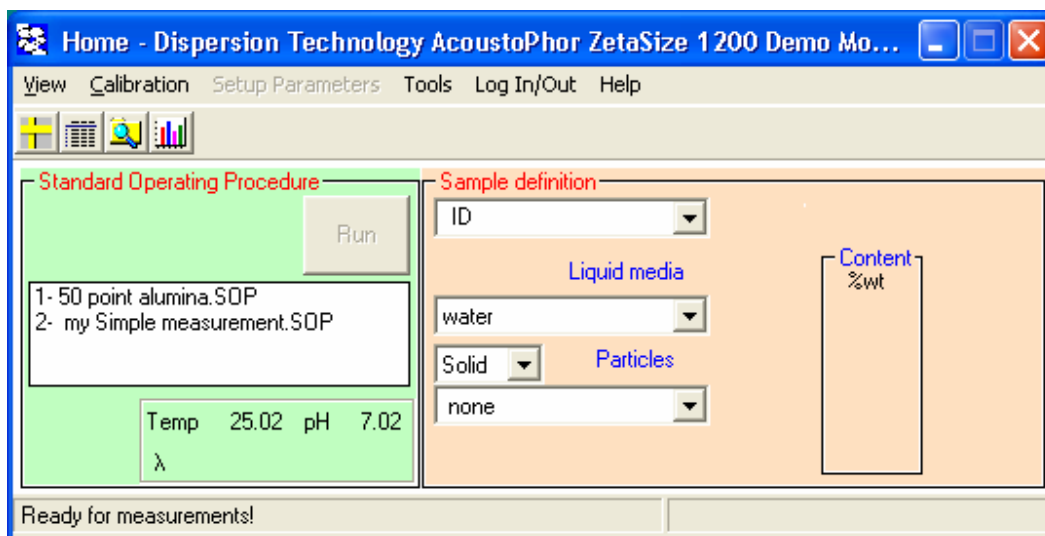
## Standard operating procedures SOP

The instrument can be used in two formats: Professional and Standard Operating Procedure (SOP).

Professional format provides a lot of flexibility and power, which in a research setting provides tremendous capability for exploring many areas of colloid science.

On the other hand, the SOP format is designed to simplify the operation of the instrument for an already defined application. Typically the SOP is developed in Professional format by the R&D group or perhaps by DT for a specific need.

The Home page appears much simpler in SOP format compared to Professional, as shown below.



The Home page consists of two frames: the Standard Operating Procedure and Sample Definition.

The Standard Operating Procedure frame contains a list of SOP's from which the user simply selects the desired procedure.

The Sample definition frame appears identical to that in Professional format except that the various sample properties are those defined in the SOP and it is not possible for the technician to change this information. The weight fraction might be a special case since perhaps the weight fraction might change from one test to another. So we might consider making the weight% a parameter that can be changed by the technician at run time. The ID is not saved in the SOP but can be used by the technician at run time to define a particular lot number or other information that might change from one experiment to the next. (Similarly, the Mfr and Trade name boxes are optionally available for special use by technician at run time.)

In summary, in order to do an experiment the technician would simply select the appropriate SOP from the pull down list, enter whatever information is highlighted in yellow, and then push the RUN button.

A Standard Operating Procedure is defined exactly the way one would make an experiment using the Professional format. One would define the sample, define any titration or temperature protocol, select the measurements to be performed, perhaps even change the Setup Parameters.

Analysis form should be also setup on the desirable way. Analysis File has item Save Analysis Protocol. It creates a string that would be saved as part of particular SOP.

However, instead of making a measurement under these conditions, one would simply click on the menu item

**Tools > Save SOP**

And then give this SOP a name.

There is a special directory in C/data\_aco directory where all SOP files are stored.

If you want to have the user enter certain information at run time, then you would enter a ? in place of a specified value.

As an example, suppose we want to generate an SOP to do a pH titration of a 22 wt% alpha alumina pigment in ethanol but we want the user to enter an appropriate lot number for the particular pigment being tested.

## Sample handling systems

Our decade long experience dealing with concentrated dispersions and emulsions has taught us that there is no universal sample handling system that will work for all systems of this kind. This is the major difference between instruments that target concentrated system relative to the instruments for dilute samples. Consequently, we have implemented several different sample handling methods for the DTI instruments. This file provides photographs of these setups with short explanations.

### ***DT-300, Electroacoustic Zeta Potential Probe.***

The main purpose of this instrument is characterization of the  $\zeta$ -potential. We show here various setups just for this sensor. This instrument could contain, as options, conductivity probe, temperature probe, pH probe, dielectric permittivity probe and 2 burettes titration. Some of the setups allow to add these probes next to  $\zeta$ -potential probe.

#### **Zeta Probe, manually**

This is the simplest setup. It could work for a quick single point test. Measurement time for dispersions of heavy rigid particles (rutile, alumina, silica, etc) takes about 30 seconds.



#### **Zeta Probe supported with the holder plate**

This setup is suitable for stable systems when measurements take longer time or multiple measurements.

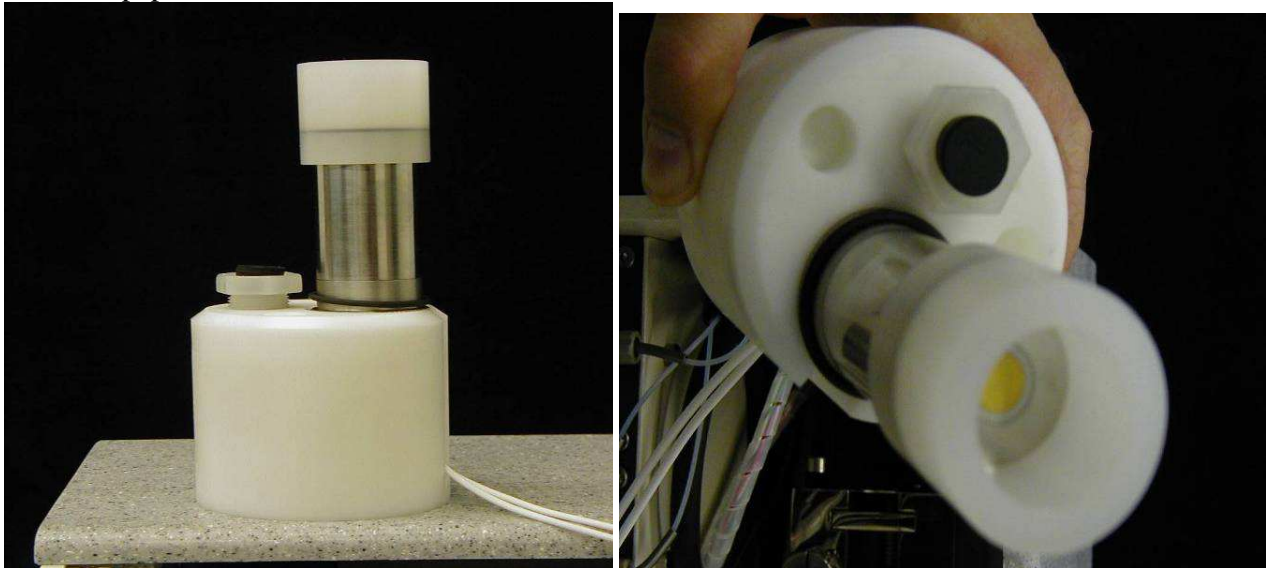


### **Zeta Probe up side down with small volume sample cup**

There are several purposes of this setup. In the case of stable systems, the purpose of this setup is minimizing sample volume. It might be as low as 2 ml. We recommend multiple measurements always with this setup. Zeta potential data is reliable only when CVI signal remains stable over period of time.

In the case of unstable system this setup can be used for studying sedimentation, as described in the Bibliography, Papers [3].

Third purpose of such setup is characterizing porous powders saturated with liquid, as described in the same paper.



### **Zeta Probe up side down with minimum sample cup**

This setup has been designed for measuring proteins. It allows sample volume as little as 100 microliters. It can be combined with heater control for measuring protein samples at fixed temperature.





### **Zeta Probe for characterizing porous body**

This setup has been designed for measuring pore size and zeta potential in porous bodies. There is one critical requirement – surface of the porous body must be flat. There should be no gap between the face of the Zeta probe and the body surface.

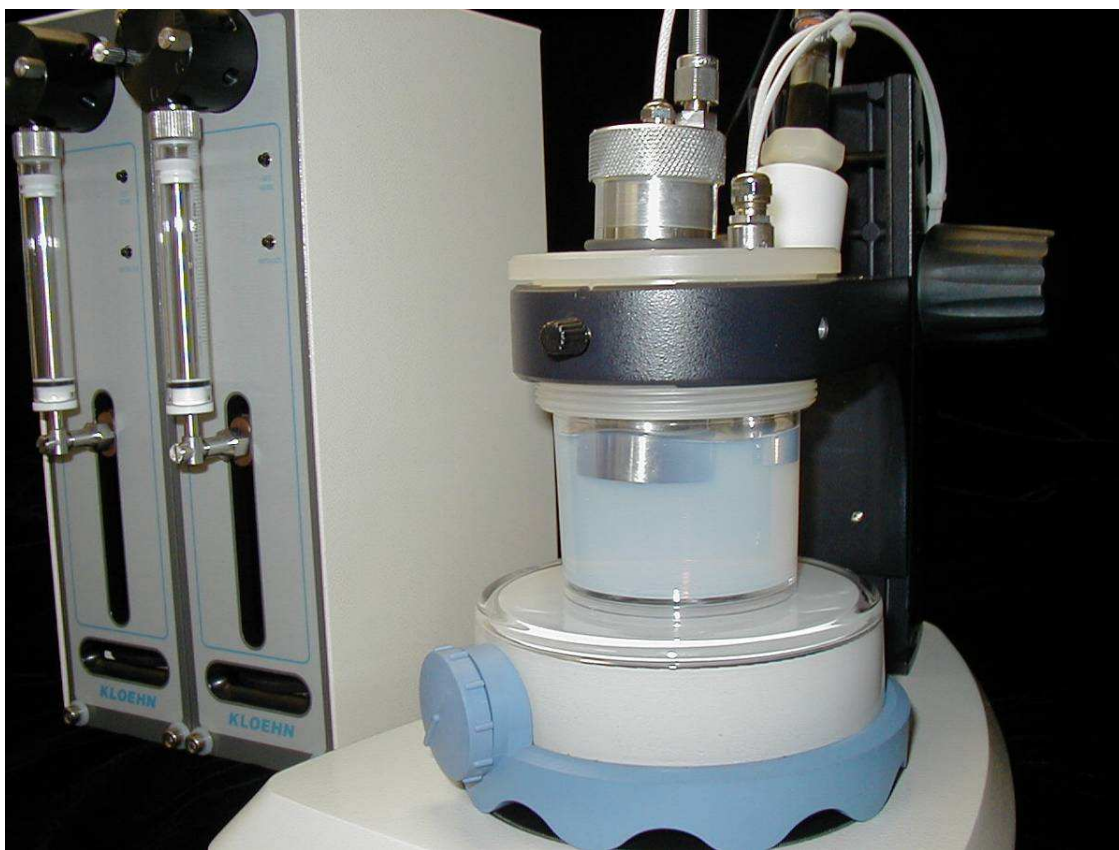


### ***DT-310, Electroacoustic Zeta Potential Probe with titrator.***

Zeta probe can be combined with titrating system for running various titration described in the Sections on titrations. The most important feature of such combined instrument from the viewpoint of sample handling is necessity of proper sample mixing. Injected chemicals must be spread about the complete sample. Otherwise, chemicals additions would be accumulating in one particular spot and slowly diffuse to the places where measuring probes are located. Equilibrium titration is possible only when mixing sufficient. This can be achieved with following setups.

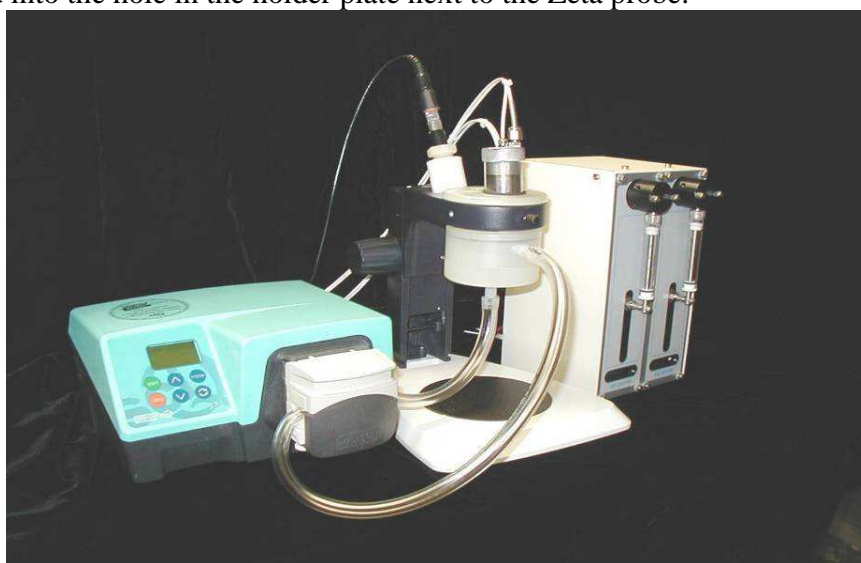
### **Zeta Probe with magnetic stirrer**

This setup is suitable for moderately concentrated systems with rather low viscosity. Stirring is important either for preventing sedimentation, or for proper mixing during titration. Two burettes titration block is shown on background. Injector of the burettes and other sensors could be placed into the hole in the holder plate next to the Zeta probe.



### **Zeta Probe with peristaltic pump.**

This setup is suitable for concentrated systems with rather high viscosity. It is especially important for systems that become strongly flocculated in the vicinity of iso-electric during titration. Bottom block has cone shaped orifice for eliminated stagnation volumes. Sample is being pumped from the top to the bottom. Two burettes titration block is shown on background. Injector of the burettes could be placed into the hole in the holder plate next to the Zeta probe.



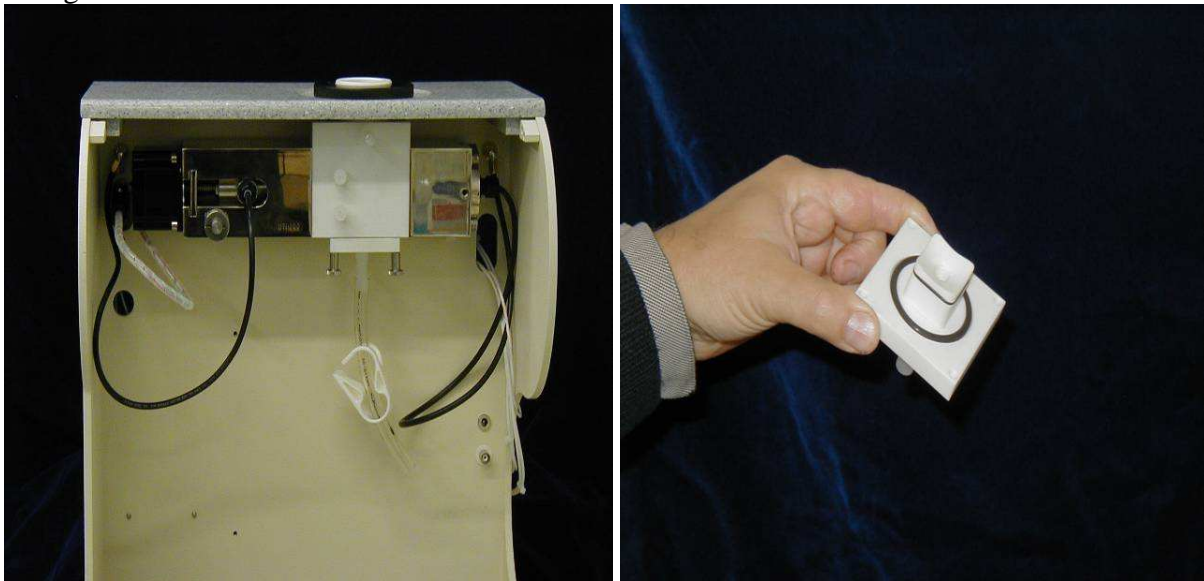
There is detail description of connecting peristaltic pump, loading and unloading samples below.

### ***DT-100, Acoustic particle size sensor.***

The main purpose of this instrument is particle sizing. It can be also treated as high frequency longitudinal rheometer. We show here various setups just for this sensor. This instrument could contain, as options, temperature probe, pH probe, and 2 burettes titration.

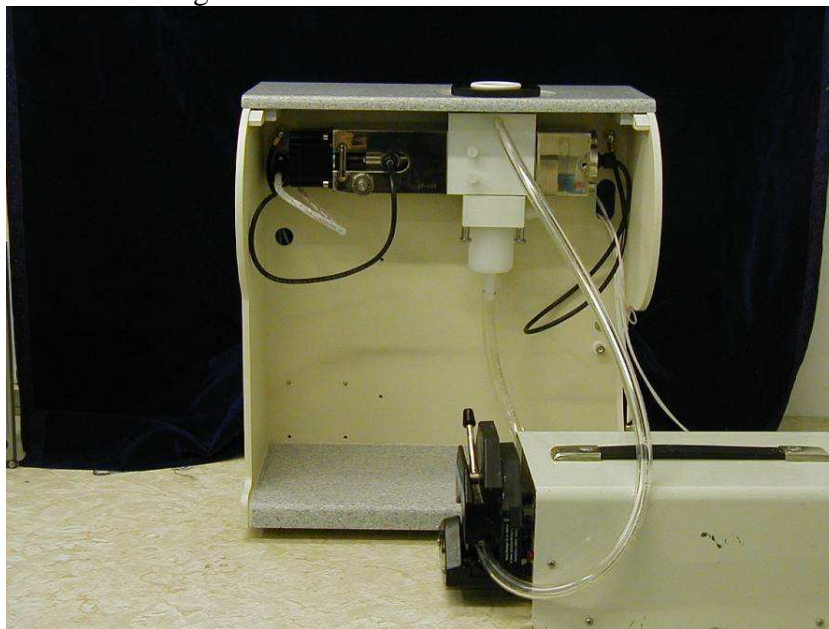
#### **DT-100 with minimum sample volume**

This setup is suitable for stable systems only. It allows us to minimize sample volume for particle sizing down to 15  $\mu$ l.



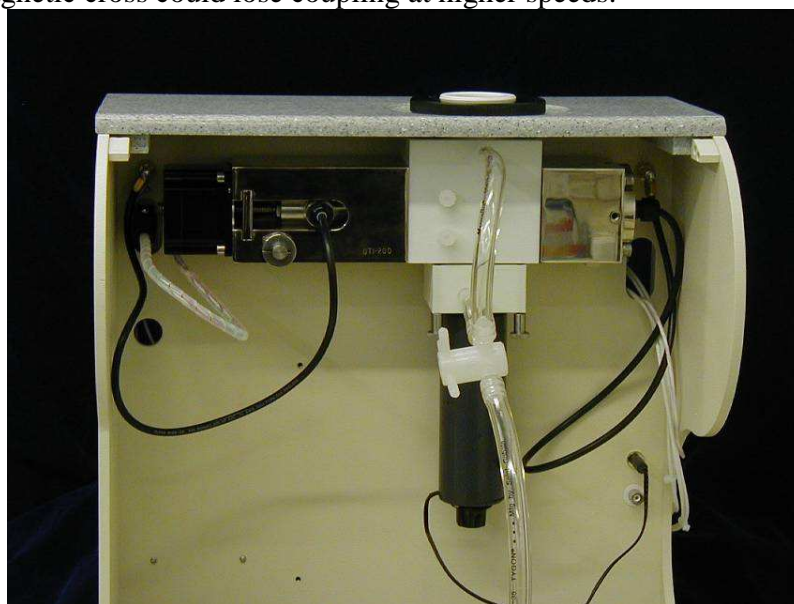
### **DT-100 with peristaltic pump**

This setup is suitable for viscose and unstable systems. Insert on the bottom of the sensor has cone shaped orifice for eliminated stagnation volumes.



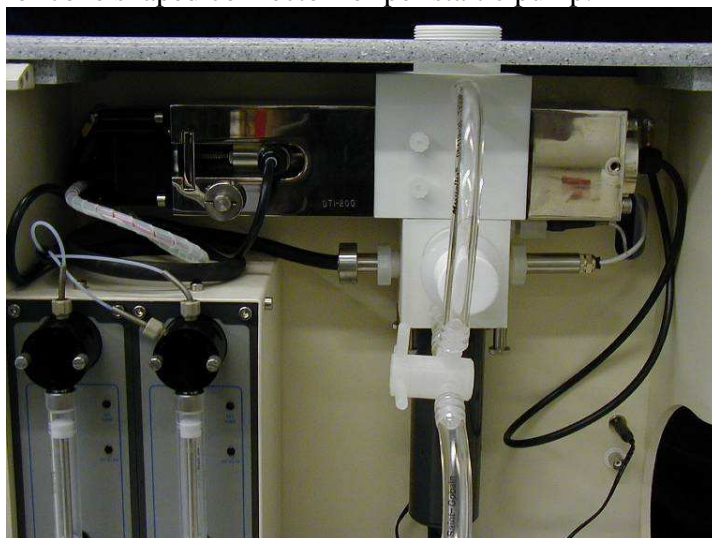
### **DT-100 with magnetic stirrer**

This setup is suitable for moderately concentrated systems. Magnetic stirrer on the bottom of the sensor creates higher pressure there. This pressure pumps sample through the tube from bottom to the top. It is much more efficient than a simple vortex. Magnetic stirrer has a speed regulating knob at the bottom. We recommend using second position of the knob after first position when liquid starts moving. Magnetic cross could lose coupling at higher speeds.



### **DT-100 with titration setup**

This setup allows us connecting burettes to the acoustic sensor for running titration. Other sensors could be inserted into the bottom block. Port on the bottom of the bottom block could accommodate either magnetic stirrer or cone shaped connector for peristaltic pump.

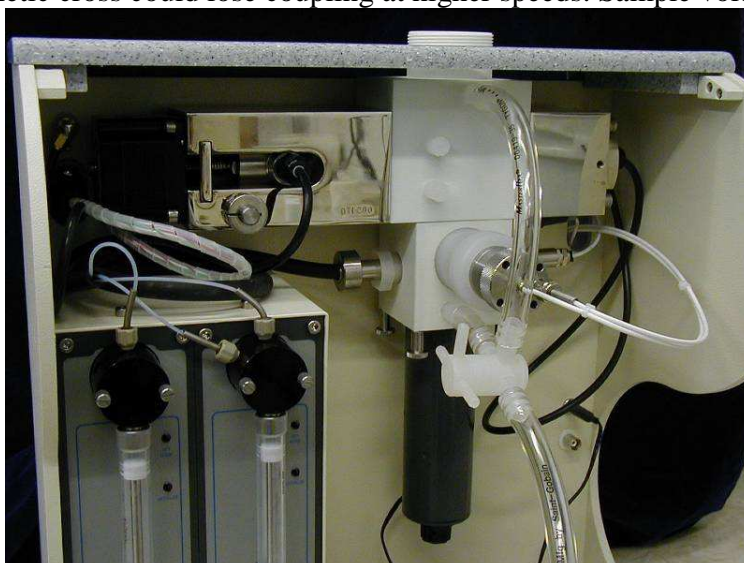


### ***DT-1200, Acoustic and Electroacoustic spectrometer.***

This instrument combines together all probes and can accommodate titration.

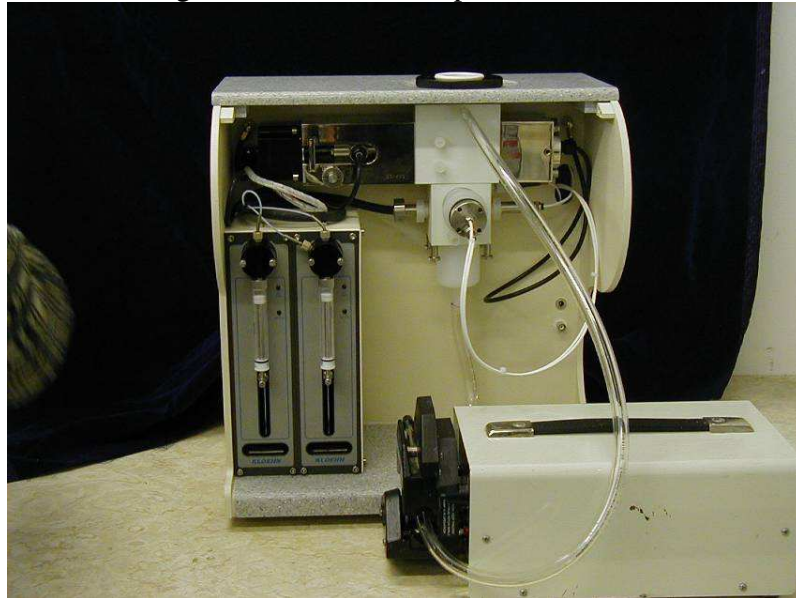
### **DT-1200 with magnetic stirrer**

This setup is suitable for moderately concentrated systems. Magnetic stirrer on the bottom of the sensor creates higher pressure there. This pressure pumps sample through the tube from bottom to the top. It is much more efficient than a simple vortex. Magnetic stirrer has a speed regulating knob at the bottom. We recommend using second position of the knob after first position when liquid starts moving. Magnetic cross could lose coupling at higher speeds. Sample volume is about 120 ml.



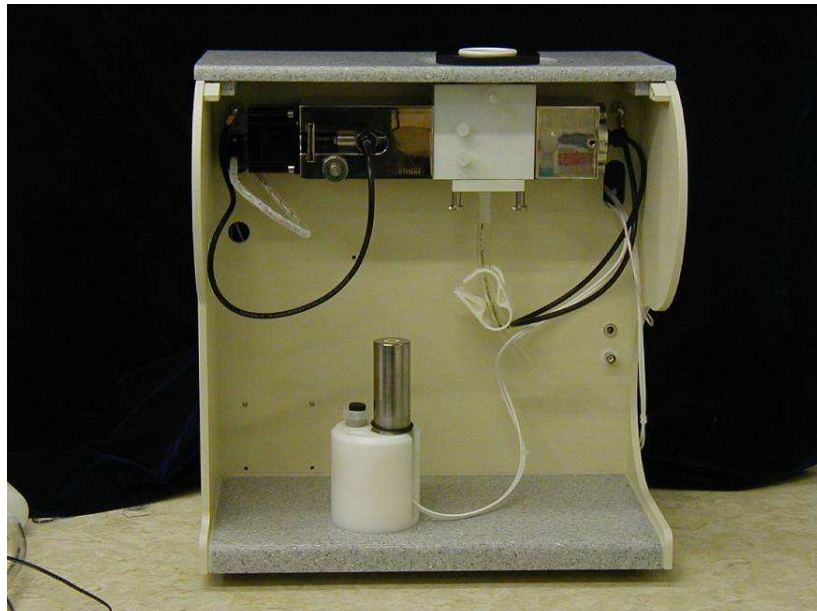
### **DT-1200 with peristaltic pump**

This setup is suitable for viscous and unstable systems. Insert on the bottom of the sensor has cone shaped orifice for eliminated stagnation volumes. Sample volume is 150 ml.



### **DT-1200, separate sensors**

In the basic configuration the zp probe is mounted in the bottom sensor block. However, this electroacoustic sensor can be used externally, completely separate from the acoustic sensor. For example the zp probe can be mounted at some distance from the sensor unit, perhaps connected to some ongoing process.



To use the zp probe externally, rotate the locking ring that holds the zp probe counterclockwise about one turn. Then pull the probe out completely. Insert the Teflon plug in place of the zp probe.

Insert it just far enough that it is flush with the inside of the bottom block. Tighten the locking ring to hold it in place.

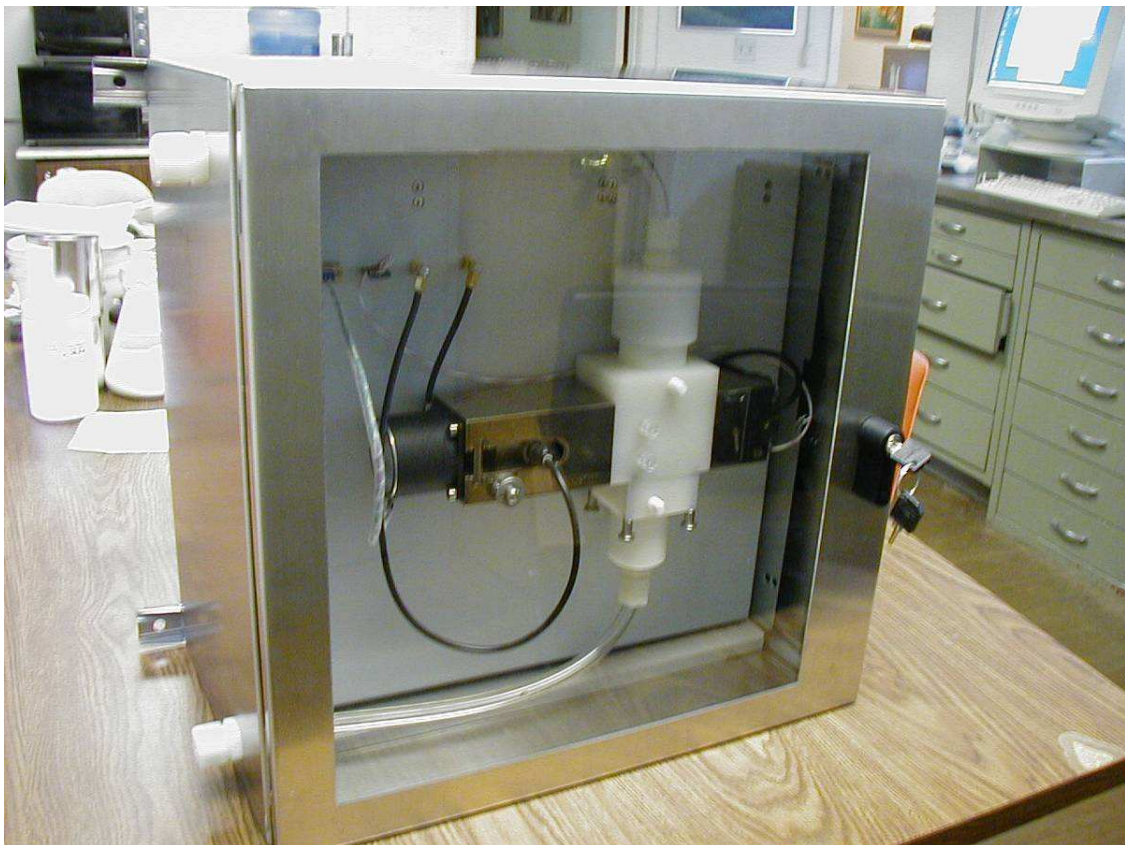
If later it is desired to use the zp probe in the basic configuration, just reverse the above steps.

Sometimes it is convenient to set up the zp probe using the “Ring Stand” setup shown in the figure below. The sample is held in a sample cup and stirred using a magnetic stirrer. A holder is mounted on the ring stand above the sample cup and has provision for mounting the zp probe, the temperature probe, the conductivity probe tip from the burette and the tip of the dispensing probe. These various sensors might be used in any combination depending on the application.

This setup is suitable for stable systems. It allows us minimizing sample volume and simplifying cleaning. It is convenient also for making just PSD or just Zeta measurements.

### ***DT-500, On-line sensor block.***

This is sensor block that could accommodate any combination of DTI sensors (Acoustic PSD, Electrosacoustic Zeta, conductivity, pH, temperature). This block should be connected with the independent sample handling system to the process stream.



## *Set up instruments to work with an external pump*

The magnetic stirrer used in the basic configuration may not be adequate in some cases. The sample may be too viscous and any added reagent may not then be mixed sufficiently, or the particles are so large that they settle out in spite of the stirring. In these cases it will be necessary to use the external peristaltic pump. This section describes how to change from the basic to the external pump configuration.

Empty the chamber of any fluid.

Pull out stirrer plug from mating connector on back panel. Remove the stirrer assembly by unthreading it from the bottom sensor block.

Remove the top hose from the barb fitting on the upper sensor block.

Remove the bottom hose from the barb fitting on the bottom sensor block.

Remove the barb fitting from the bottom sensor block using pliers if necessary and install the plug in its place to prevent sample from exiting this port as shown below



### **Removing barb fitting**

Locate the cylindrical funnel assembly. If not already installed, install the O-ring in the groove on the top surface. If not already installed, install the barb fitting in the port on the bottom of the cylindrical funnel assembly.

Attach the cylindrical funnel to the bottom sensor block by screwing it into place. It need only be hand tight.



Place the peristaltic pump to the right of the measuring unit as shown in



. Open the pump by rotating the lever arm to the left.

Connect a hose from the barb fitting on the bottom of the cone, as shown in , through



the pump, as shown in , and finally to the barb fitting at the top of the top sensor block.

Close the pump by swinging the lever arm to the right.

The external pump is now ready for use.



**Peristaltic pump**



**Connecting hose to funnel**

The sample volume when using the external pump (with x cm of y diameter tubing) is about 200 ml.

Close the pump head by rotating the lever arm clockwise.

Set the pump to rotate in clockwise direction such that the fluid is pumped from the bottom of the cylindrical funnel to the top port of the top sensor block.

Adjust the speed control for a mid range speed (about 1/3 full scale)

Fill the chamber with 200 ml of sample. If the sample contains large dense particles that settle rapidly, it may be necessary to mix the sample as you pour it into the chamber.

Turn off the pump by setting the switch to the mid position.

Remove the tubing from the barb fitting on the top sensor block.

Place the container in which you want to retrieve the sample at a position lower than the pump. Some users find it convenient to have a used sample container under the counter, or to have the instrument next to a waste sink.

Place the open end of the tubing in the container or sink to or dispose of the sample.

## Maintenance

We recommend keeping sample chamber of Acoustic sensor always wet, filled with water. This would maintain proper status of o-rings that prevent sample leaking into along the moving rod.

Zeta potential probe requires special care when being cleaned. Gold electrode is actually a very thin layer of gold plated over the plastic. It can be easily damaged by sharp cleaning devices. **WE DEMAND** that **Zeta Probe will be cleaned only with paper towel** or similar non-invasive treatment. Polishing of zeta probe with sand paper would remove gold layer. This damage will not be warranted. When the probe is stored outside of the sample chamber, the provided protective cap must be used to prevent damage to the gold electrode.

Do not leave Zeta potential probe in aggressive solvents longer than measurement requires. Remove it after the measurement finished and clean it with alcohol and then with water.

When the pH probe is not being used, it should be stored safely in the provided storage tube or in the pH stand. ***The tip of the pH probe is delicate and easily broken.*** Take care in moving the pH probe from place to place. ***It is important that the pH probe is not inserted too far, or the probe will be broken during attenuation measurements as the gap is closed!*** A 12 mm long white spacer, also shown in the figure to the right, should be installed on the body of the pH probe that keeps the tip of the probe extended at least 40 mm from the bottom of the cap, ***but no more than 45 mm.*** \*

***Do not use liquids that are not compatible with following materials:*** stainless steel, glass filled PTFE, gold, quartz, polypropylene and Du Pont “Kalrez” o-rings.

***Do not submit sensors to temperatures exceeding 50 C<sup>0</sup> and below 10 C<sup>0</sup>.***

***Burettes must be flushed with water and emptied after titration finished. Do not leave reagents in the burettes, this might cause corrosion of the burettes valves.***

***When using heater option, do not leave heater outside of the liquid and in contact with plastic parts – it would melt them. Do not forget turning heater off after measurements finished.***

Non-aqueous conductivity probe must be cleaned after measurements finished. It should be taken apart by removing only the guard electrode. The inner electrodes must be cleaned, dried and then guard electrode assembled back

Do not use DTI instruments in environment with elevated humidity that might cause corrosion of electronics, similarly to the typical computers.

## Warranty

In accordance with DTI warranty conditions, the warranty period is 12 months, unless it is extended by special agreement in the purchase order. For claims under the warranty please contact your local dealer. Many parts can be replaced at your site. Alternatively, you may send the instrument directly to our factory (after requesting and RMA number), enclosing the delivery invoice and giving reasons for the claim. You will be liable for the freight cost.

The warranty does not cover worn out parts, nor does it apply to faults resulting from improper use, insufficient care or maintenance not carried out in accordance with the instructions in this operating manual.

## Advanced topics

### *Understanding Experiments and Measurements*

In the following lessons we will discuss making measurements and doing experiments. It is helpful to understand how these two terms are used. A measurement is the action of measuring a specific set of parameters under a fixed set of conditions. For example, in the previous lessons you have seen how a given measurement might include zeta potential, particle size, and conductivity, in various combinations depending on our need at the moment. A given measurement is obtained under one set of conditions of temperature, pH, and chemistry. In an experiment, we alter the measurement conditions according to some experiment protocol in order to see how the measurement values change with pH, temperature, added reagent, or just elapsed time.

### *Log in, Log out, Adding new users*

Before making measurements it is useful to log in. If you are logged in any measurements that you make are automatically saved with reference to your initials and project name. This will make it easy for you to later find specific test results.

After the system has completed its startup sequence, you can log in by clicking on “**LogIn/Out**” on the home page menu bar. The LogIn form shown to the right will appear.

The password for a Guest user is simply the word “guest”. Type “guest” in the password box and press the Enter key. Case is not important. The window will then appear as shown at right.

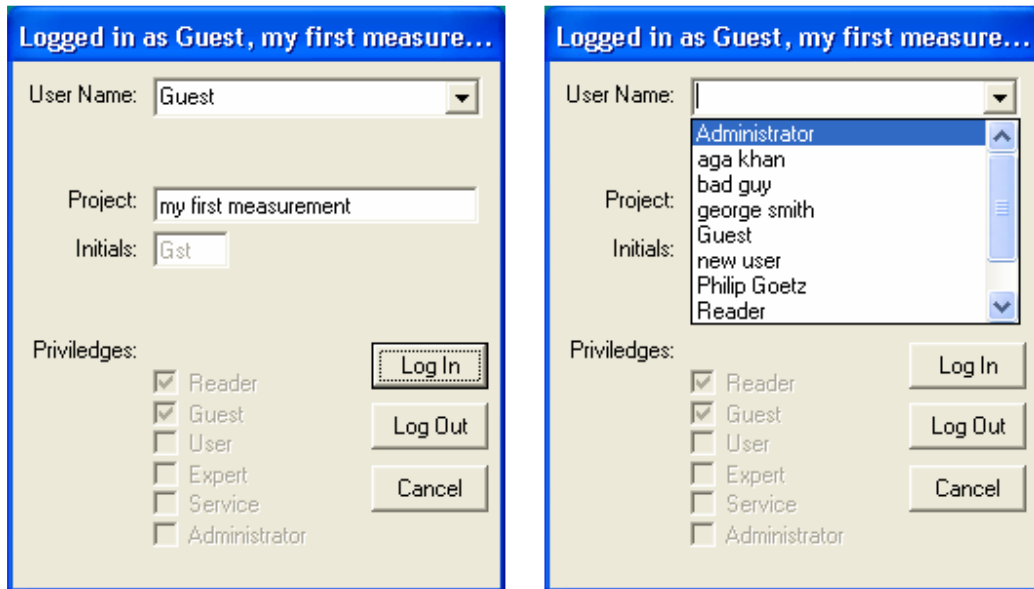
If you wish you can then enter a description of the project you are working on in the Project box. This information can be used later to retrieve specific data related to your project. For now, if you like, just type the phrase “my first measurements”.

The image displays two screenshots of a software login interface. The left screenshot, titled "Please log in...", shows a "User Name" dropdown menu set to "Guest" and a "Password" text box. A "Cancel" button is at the bottom right. The right screenshot, titled "Log in, enter password, press Enter...", shows the "User Name" dropdown still at "Guest". The "Password" field contains "\*\*\*\*\*" and has a "Change password" button next to it. Below are "Project", "Initials" (containing "Gst"), and "Email" text boxes. A "Privileges" section has checkboxes for "Reader", "Guest", "User", "Expert", and "Service", with "Reader" and "Guest" checked. "Log In", "Log Out", and "Cancel" buttons are at the bottom right.

Every person logged-in is defined in the saved data by his/her Initials. As shown in the Initials box, the initials for a Guest is “Gst”. (The initials for an existing User Name can not be changed.)

To log in press the Log In button.

If you are through using the instrument you may want to log out so that another user does not inadvertently make measurements and save data marked with your initials and project.



To log out click the menu item LogIn/Out on the Home page. A window such as the one shown to the right will appear.

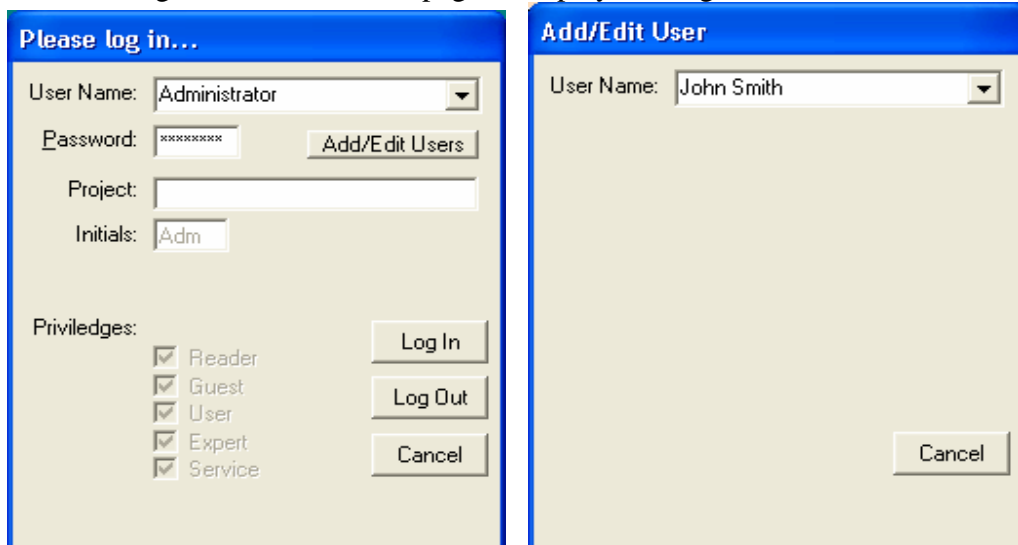
To log out press the **Log Out** button.

Or, if you wish, you can just enter a different Project and then press **Log In**.

If you decide not to logout press **Cancel**.

For adding a new user:

Click on LogIn/Out on the home page to display the Log In Window.



From the list of User Names select **Administrator**.

Enter the Administrator password and press the **Enter** key. (The Administrator password is “dispersion”.)

To add a new user or edit an existing unit press the **Add/Edit Users** button.

The Add/Edit User window will appear. Enter the new User Name, e.g “**John Smith**”, and press the **Enter** key.

The image shows two side-by-side screenshots of a dialog box titled "Defining new user 'John Smith'".

**Left Screenshot:**

- User Name: John Smith (dropdown menu)
- Password: (empty text box)
- Initials: Joh (text box)
- Email: (empty text box)
- Privileges:
  - Reader
  - Guest
  - User
  - Expert
  - Service
- Buttons: Cancel, Save

**Right Screenshot:**

- User Name: John Smith (dropdown menu)
- Password: john (text box)
- Initials: JS (text box)
- Privileges:
  - Reader
  - Guest
  - User
  - Expert
  - Service
- Buttons: Cancel, Save

A window “Defining new user ‘John Smith’ will appear.

Enter a password for the new user and then press Enter key. The password will be visible during entry.

If you like, enter a more appropriate three letter initials for this new user. (By default program picks first three letters of first name. Use care picking initials; once initials are saved for this user, the selected initials can not be changed.)

By default, a new user is defined to have User, Guest, and Reader privileges. If appropriate for this individual, you can add Expert and/or service privileges by checking the appropriate boxes.

To save this new user definition, press the **Save** button.

To forget this new user definition, press the **Cancel** button.

### ***For Editing an existing user***

The procedure for editing an existing user is very similar to defining a new user.

Click on LogIn/Out on the home page to display the Log In Window.

From the list of User Names select **Administrator**.

Enter the Administrator password and press the **Enter** key.

Press the **Add/Edit Users** button. The Add/Edit window will appear.

Click on the box to the right of the User Name box to pull down list of existing users. Select an existing user, e.g. John Smith.

The password for this user is visible. You may change the password if you wish. You may also change the Email address for this user.

You can also change the privileges for this user by checking or un-checking the privileges boxes at the bottom of the window.

To save your changes for this user, press the **Save** button. To forget changes for this user, press the **Cancel** button.

### *Using Setup parameters*

Setup parameters define the conditions under which the measurements are made and the manner in which the raw data is processed to yield the final attenuation spectra, cvi signal, or conductivity data. Normally when you make a measurement the software automatically selects

various default values for these Setup Parameters. These default values are generally suited for virtually any sample, whether the sample is very dilute or quite concentrated. The user does not have to think about these values. On the other hand, there may be times when you want to customize these settings.

You can open forms with setup parameters by clicking on this item on the Home page. The form that appears would depend on what sensor check box has been checked on the Home page. Below are these setup parameters forms for attenuation, conductivity and electroacoustic measurements.

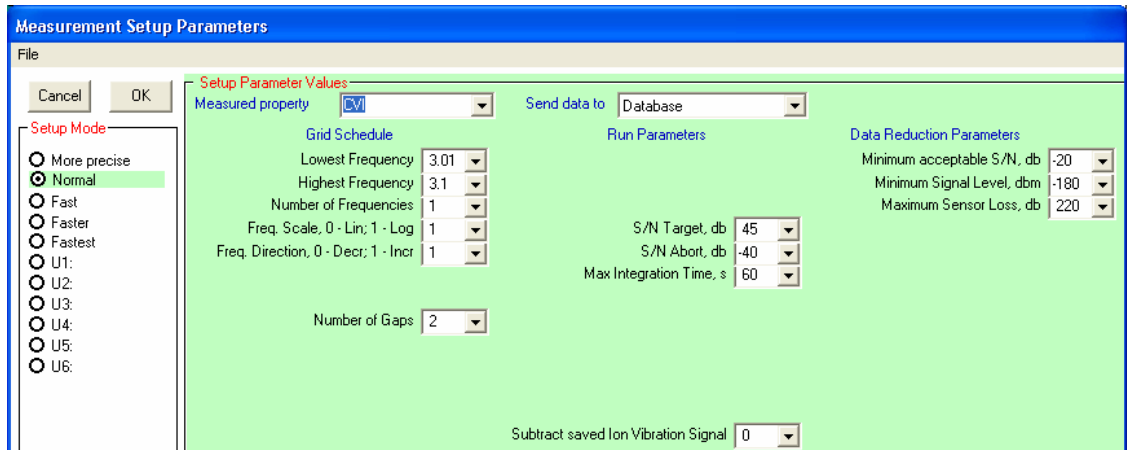
The screenshot shows the 'Measurement Setup Parameters' dialog box with the 'Measured property' set to 'Attenuation'. The 'Setup Mode' is 'Normal'. The 'Grid Schedule' parameters are: Lowest Frequency (3), Highest Frequency (99.5), Number of Frequencies (18), Freq. Scale (0 - Lin; 1 - Log), Freq. Direction (0 - Decr; 1 - Incr), Smallest Gap (0.445), Largest Gap (20), Number of Gaps (21), Gap Scale (0 - Lin; 1 - Log), and Gap Direction (0 - Decr; 1 - Incr). The 'Run Parameters' are: RunLevel (1), Gap Equilibrate Time (0.1), S/N Target (45), S/N Abort (10), Max Integration Time (5), GapMode (0-Norm, 1-Single, 2-Fixed), and Target # gaps each freq (5). The 'Data Reduction Parameters' are: Minimum acceptable S/N (35), Minimum Signal Level (-120), Maximum Sensor Loss (180), Maximum Colloid Loss (60), Maximum Error (100), Maximum Error (1), Maximum Percent Error (20), Minimum # of Gaps (3), and MaxDegreePolyFit (1).

Setup parameters for attenuation measurement.

The screenshot shows the 'Measurement Setup Parameters' dialog box with the 'Measured property' set to 'Conductivity'. The 'Setup Mode' is 'Normal'. The 'Grid Schedule' parameters are: Lowest Frequency (3), Highest Frequency (3), Number of Frequencies (1), Freq. Scale (0 - Lin; 1 - Log), and Freq. Direction (0 - Decr; 1 - Incr). The 'Run Parameters' and 'Data Reduction Parameters' sections are visible but their values are not clearly legible in this view.

Setup parameters for conductivity measurement





Setup parameters for cvi measurement

### ***Measuring Zeta potential at high ionic strength***

The cvi probe measures the total electroacoustic vibration current which is the sum of the colloid vibration current as well as the ion vibration current. Normally the colloid vibration current is much larger than the ion vibration current and we can simply calculate the zeta potential from the total current. However, at very high ionic strength media, it may be somewhat more accurate to subtract any ion vibration current from the measured signal before calculating zeta potential.

Making a zp measurement at very high ionic strength involves two steps. First you will measure the ion vibration in the same media that you will be using to suspend the particles. You will save that ion vibration. This is described as Calibration procedure.

Second you will make the measurement of the CVI for your colloid, specifying that you want to use the ion vibration current that was measured for the media and want to subtract it from the measured signal. Calibration procedure describes how to achieve this using Setup parameters form.

### ***Theories for computing zeta potential in dispersions.***

Characterization of the  $\zeta$ -potential in general requires the following elements:

- 1) **raw experimental data;**
- 2) **input parameters** for the particles and liquid that would be used in the
- 3) **appropriate theory.**

This text presents all these three elements for Electroacoustic method employed by Dispersion Technology Inc instruments. At the end we give the list of database fields for all parameters. DT instrument sensors measure the following electroacoustic and electrodynamic parameters, which can be considered as

**Raw experimental data includes:**

**Colloid Vibration Current magnitude**

**Colloid Vibration Current phase**

**Conductivity,** optional.

The measurement by itself is self-consistent and requires no input data. It is done at one frequency. In default it is 3 MHz. Setup parameters of the Main DT form makes it possible to change this frequency, or ask for multiple frequency measurements. However, interpretation software

requires and assumes just one frequency. It is sufficient for  $\zeta$ -potential calculation. These measured parameters are shown in black on the Analysis form in two frames “CVI experiment and input” and “Conductivity”.

Sample Definition	
0.1	wt. fraction of silica, Ludox in water measured on 10/24/2003 11:43:28 AM
test fresh	File Name vfr 0.048 pH 4.1 Temperature 25.76

CVI experiment and input	Classic theory	Conductivity	Advanced CVI aqueous	Non-aqueous
magnitude 3321141	$\zeta$ -33.03	S/m 0.2364	$\zeta$ -35.08	$\zeta$ 0
size 0.1	phase 193.2	ka 20.825	Du 0.002	surface charge 0
d. mobility 3.412	phase th 1	1/k 0.0024	MW freq 54.04349	Henry-Ohshima 1.000
st.dev 0.2			Diff. freq 1.592357E-02	
size-phase 0.56				

#### Input parameters includes:

- Liquid density, dynamic viscosity and dielectric permittivity;
- Particles density
- Particle size distribution
- Particles dielectric permittivity in some cases
- Weight fraction of particles

Term “Input parameters” does not mean that user must know all of them from independent sources. DT sensors and software allow user to determine some of them. These are the options that DT provides, for instance measuring particle size with acoustic sensor.

User has always an option to specify any input parameter from his independent sources.

Values of the Liquid density, viscosity, dielectric permittivity and Particles density, dielectric permittivity must be specified in the Database Materials table for the materials of the Liquid and Particles.

Weight fraction is specified on the Main Form.

Median particle size and standard deviation must be specified on the Main Form if Acoustic attenuation is not measured and size exceeds 300 nm. It must be done using Apriori Size text box that becomes visible from the menu View on the Main form.

Alternatively software provides possibility to select one of the there options for defining particle size when CVI measurement starts. The third option – size from the CVI phase, is the least reliable.

Median particle size and standard deviation come automatically from the particle size distribution calculated from the Attenuation spectra if Acoustic sensor is employed together with CVI probe.

There is opportunity to change any of the Input parameters and recalculate  $\zeta$ -potential using Run on the Analysis form. Button Run becomes available using Analysis View, Re-calculate.

#### Electroacoustic theories.

It is traditional in electroacoustics to use one intermediate step that converts various electroacoustic signals to the same parameter of the “electrophoretic dynamic mobility”  $\mu_d$ . In the case of the measured CVI magnitude this conversion is done according to the following expression:

$$CVI = AF(Z_T, Z_s) \varphi \frac{\rho_p - \rho_m}{\rho_m} \mu_d$$

where  $Z_T$  and  $Z_s$  are acoustic impedances of the transducer and dispersion,  $\varphi$  is volume fraction,  $\rho_p$  and  $\rho_m$  are densities of the particle and liquid.

Introduction of the “dynamic electrophoretic mobility” makes it possible to compare results for electroacoustic theories of various electroacoustic effects between themselves and with theory of electrophoresis.

We use 3 different theories for all variety of aqueous systems.

#### **Classic theory of Smoluchowski type for aqueous systems.**

The most widely and successfully used electrophoretic theory is so-called Smoluchowski theory. Its success is related to the peculiar fact that within range of this theory validity electrophoretic mobility is independent on the particle size, shape, volume fraction, conductivity. It makes it very simple and attractive to use.

#### Conductivity measurement is not required.

It turns out that there is a version of the electroacoustic theory that is similar to Smoluchowski theory, has its attractive features and valid within the same range of parameters. That makes it certainly desirable for use, following success Smoluchowski theory for electrophoresis. We call this theory “Classic theory” and display results in the frame on the Analysis with the same name.

This simple theory is described in the book, Section 5.2, page 193.

When one compares results of the electroacoustics with traditional microelectrophoretic instruments from Malvern, Brookhaven, Coulter, etc, which are based on the Smoluchowski theory, he must use results from the “Classic theory” frame.

Unfortunately there are 3 restriction on the validity of the Smoluchowski type theories.

**Restriction 1.** It is valid when Double Layer is thin comparing to the particle radius  $a$ . This means that DL thickness  $l/\kappa$  must be much smaller than  $a$ :

$$\kappa a \gg 1$$

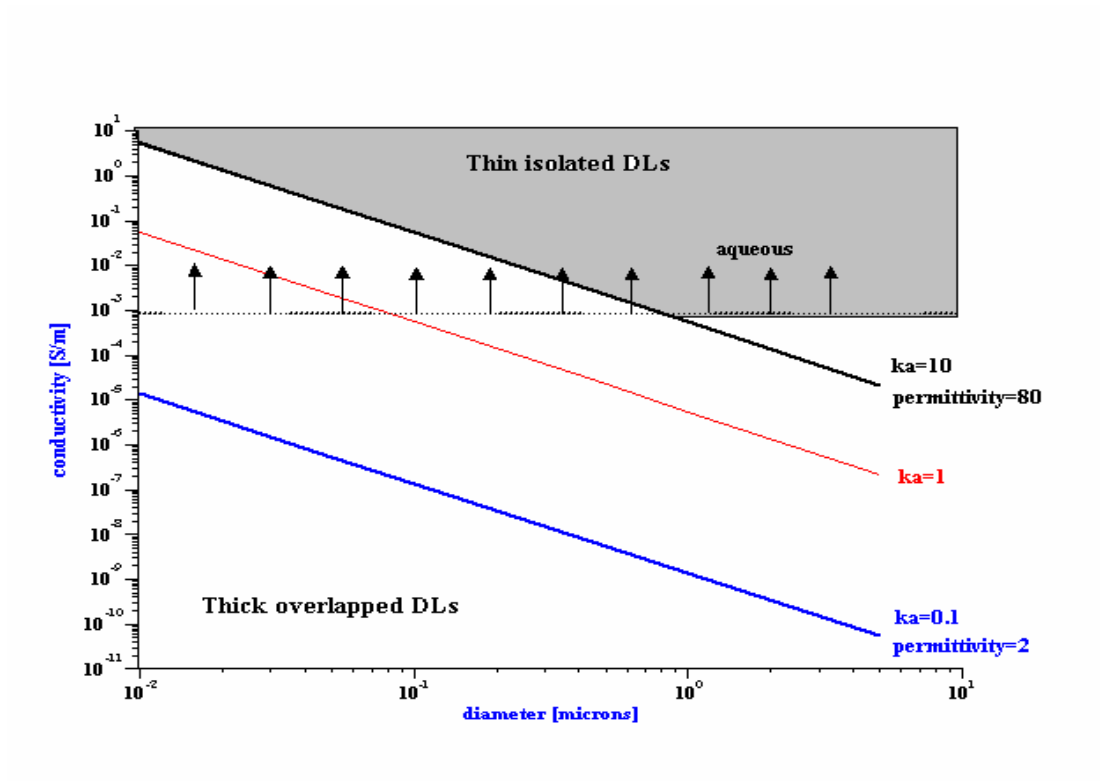
DL thickness depends on the conductivity of liquid. That is why this restriction limits conductivity and particle size values. The following picture illustrates roughly range of conductivity and size values when Smoluchowski type theories are valid. We assume here  $\kappa a = 10$  as a critical smallest value for illustrative purposes. In reality the smallest acceptable value of  $\kappa a$  depends on volume fraction, surface conductivity, etc. We give below a simple method to determine validity of the Classic theory for particular system.

**Restriction 2.** Smoluchowski type of theories neglect surface conductivity effects. Surface conductivity reflects the higher conductivity within DL due to the higher ion concentration there. This condition is expressed in the terms of small “Dukhin number”:

$$Du \ll 1.$$

This parameter was introduced by H.Lyklema and currently is accepted as official term by JUPAC draft on electrokinetic phenomena. User can find definition of the “Dukhin number” in the book, page 41.

**Restriction 3.** This type of theories consider double layers isolated, not overlap. This condition restricts volume fraction for the given conductivity and particle size.



The more sophisticated theories, which are presented in the frame “Advanced CVI aqueous” remove all these restriction. That is why we have a simple criterion for estimating validity of Smoluchowski theory in particular system:

**Validity Criterion:** *If difference of  $\zeta$ -potential values displayed in the frames values “Classic theory” and “Advanced CVI aqueous” is less then 10%, Smolukowski version of electroacoustic theory is valid with this error margin.*

#### **Advanced CVI theory for aqueous dispersions.**

Implementation of this theory requires Conductivity measurement for calculating  $\kappa a$  value.

We use two different theories for covering the complete conductivities range typical for aqueous systems as shown on the above Figure,  $K > 0.001$  S/m.

For the range of “thin DL” we use theory that is presented in the book, Sections 5.4.2-5.4.4. This theory removes restrictions 2 and 3. It is valid for any Dukhin number and frequency. However, restriction on  $\kappa a$  remains.

For the intermediate values of conductivities and particle sizes, between “thin DL” range and “thick overlapped DLs” range, we use for priory theory.

For the range of  $\kappa a$  where double layers overlap we use a new theory developed by Prof. Shilov and submitted for publishing to Journal of Colloid Interface Science. It is described in the book, chapter 5.6. It is described below because it is used for non-aqueous systems as well.

#### **Non-aqueous dispersions theory.**

Implementation of this theory requires Conductivity measurement for calculating  $\kappa a$  value.

Case of non-aqueous systems requires its own theory because DL is thick comparing with the particle size. These thick DLs overlap. For instance, DLs start to overlap at the volume fraction 0.04% if  $\kappa a = 0.1$ . **This means that DLs must be considered as overlapped. Any other electroacoustic theory for non-aqueous systems is not adequate.**

Shilov’s theory yields unusually looking expression for the dynamic electrophoretic mobility:

$$\mu_d = \frac{2\varepsilon_m RT}{9\eta\Omega F} (\kappa a)^2 \frac{1-\varphi}{\varphi} \sinh \frac{F\zeta}{RT} \frac{\rho_m}{\rho_s + i\omega(1-\varphi) \frac{2a^2}{9\eta\Omega} \rho_p \rho_m}$$

where  $\Omega$  is a drag coefficient introduced as following in the Stokes law  $F = 6\pi\eta a\Omega v$ .

Dependences on several parameters are very peculiar..

First of all it is dependence on volume fraction and  $\zeta$ -potential. It reflects very important distinction between “thin DL” and “thick, overlapped DL”. In the case of the traditional “thin DL” theory, both surface charge and  $\zeta$ -potential are surface properties, independent on the volume fraction.

In the case of the “thick overlapped DLs” surface charge and  $\zeta$ -potential is as following:

$$\sigma = \frac{1}{3} \frac{RT}{F} \frac{1-\varphi}{\varphi} \varepsilon_m a \kappa^2 \sinh \zeta$$

In the case of “thick DL”, surface charge is a true parameter of the surface properties. Electrokinetic  $\zeta$  potential might lead to erroneous conclusions because it depends on the volume fraction, not only on surface charge. For instance, if we have surface charge caused by adsorption of ions, the value of the surface charge characterizes this adsorption. It keeps constant, volume fraction independent for the same adsorption. At the same time  $\zeta$  might be different due to the volume fraction dependence, as shown in Eq. 25 or 26. This means that  $\zeta$ -potential value might lead to the wrong conclusions about adsorption. This all indicates that both,  $\zeta$  and surface charge, must be reported when dealing with concentrated dispersions with thick DL, as it is done in the frame “Non-aqueous” on the Analysis form.

Dynamic electrophoretic mobility expressed in the terms of the surface charge is given with the following expression:

$$\mu_d = \frac{2\sigma a}{3\eta\Omega} \frac{\rho_m}{\rho_s + i\omega(1-\varphi) \frac{2a^2}{9\eta\Omega} \rho_p \rho_m}$$

This expression is practically as general as Smolukowski type of theories. All hydrodynamic interactions are included in the drag coefficient.

It is important to mention that conductivity becomes necessary only if we want to calculate  $\zeta$ -potential for non-aqueous system. If surface charge is sufficient, conductivity measurement could be eliminated. It is important feature because conductivity measurement in non-aqueous systems might be complicated.

Shilov’s theory has restriction on the  $\kappa a$  value from above. For instance,  $\kappa a$  must be below 2 for 10% error margin at volume fraction 20%. The lower volume fraction the smaller is this highest acceptable  $\kappa a$  value. For rather dilute systems with several volume percents solids  $\kappa a = 0.1$  is a very conservative estimate of the Shilov’s theory limit. We use this number on the Figure above.

### Database fields used for $\zeta$ -potential characterization.

wtf	weight fraction, as %. Do not use for correcting existing record
Chemical name	optional sample ID info entered on Meas Form
C1	measured CVI magnitude at first freq, for zp using probe design
Measurement Date	date and time of the measurement
PSM	median unimodal size.(but not for modified unimodal. See Book)

PSW	standard deviation of unimodal PSD
zp	zeta potential, mV
err_th_con	CVI phase, with theoretical correction, determines sign of zeta
pH	pH
C2	measured CVI magnitude at second frequency, for zp using split sensor
K1	conductivity, S/m
Temp	temperature, degrees Celcius
Medium	name of the liquid
Dispersed Phase	name of the particle material
Density	density of particles material, kg/m <sup>3</sup> . Use when correcting existing record
Weight_Fraction	weight fraction (not in %). Use when correcting existing record
K2	zeta potential with Du and MW corrections calculated with conductivity
K3	std deviation of unimodal PSD, for calculating zeta potential
K4	th.phase for Du=0 and no MW correction
K5	ka, ratio of the particle radius to the Debye length
K6	Debye length, microns
K7	dynamic mobility, micron/sec/V/cm
K8	phase angle of complex conductivity, degrees
K9	Imaginary part of complex conductivity, S/m
K10	Magnitude of complex conductivity, S/m
K11	Maxwell-Wagner frequency, MHz
K12	Du calculated using measured conductivity
K13	diffusion frequency, MHz
K14	zeta potential, for non-aqueous media
K15	zeta potential, for Du=0 but MW freq from K
K16	zeta potential phase for Du=0 but MW from K
K17	CVI gap, m
K18	CVI Phase size
K19	surface charge for non-aqueous
K21	th.phase DU and MW taken into account
F_c_low	lowest frequency for CVI measurement
F_c_high	highest frequency for CVI measurement
numfreq_c	number of frequencies for CVI measurement

### ***Measuring weight fraction from cvi***

In some applications it is desirable to continuously estimate the weight fraction of the sample. We can estimate the weight fraction from the measured CVI if we know the median size of the particulates, the standard deviation,

To make this series of measurements of weight fraction we first need to enter some calibration data. On the Home page, click on menu item Calibration – Weight fraction from CVI. The following form appears:

Calibrate weight fraction from Cvi

Enter the zeta potential, median size, standard deviation, and conductivity of reference material.

Save

Cancel

Zeta potential, mV -38

Median size, um 0.100

Standard deviation 0.3

Conductivity, S/m 0.14

Follow the instructions and enter the zeta potential, median size, standard deviation and conductivity of the sample for which you want to monitor the weight fraction.

On the Home page click on File – Analysis to open the Analysis window. On the Analysis menu click on View – Recalculate. On the far right hand portion of the Analysis window, check the box “vfr search”.

Define your sample as usual and start an experiment. The weight fraction for each measurement will be written to the WFR field in the database.

## Characterization of porous bodies.

There are several DTI models that can handle porous materials. This function can be as an Option for DT-1202. There is Universal Zeta Potential probe DT-330 for measuring zeta potential of both, particulate systems and porous materials. In addition, there are special dedicated instruments DT- 800, DT-350 and DT-900, which is being marketed by Quantachrome Corp., Florida under the names WAVE-3805, WAVE-2305 and WAVE -1905.

This Manual does not cover WAVE-1905 (DT-900) and WAVE-3805 (DT-800). These instruments are described in a separate Manuals.

Quantachrome sold models make possible characterization of **porosity**, **pore size** and **zeta potential** of 5 different types of porous bodies: deposits of solid particles, deposits of porous particles, dispersion of porous particles and macroscopic porous bodies like cores – monoliths and membranes.

Models sold by DTI use the same methods for **porosity** and **zeta potential** measurement. These models do not measure **pore size**. This measurement is described in the Manual for DT-800.

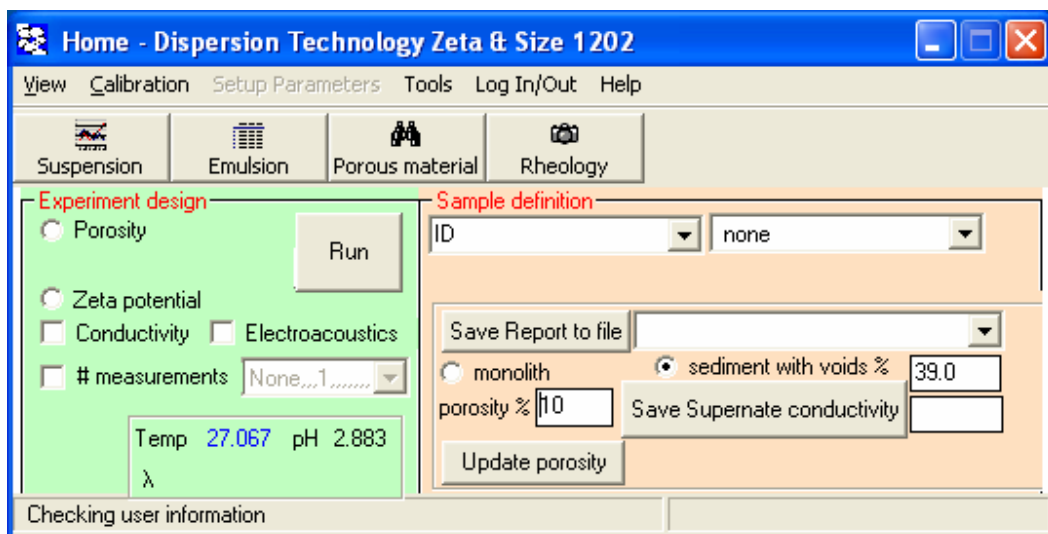
Measurement of **porosity** is done with aqueous conductivity probe.

Measurement of **pore size** and **zeta potential** is done with electroacoustic probe.

This Manual describes software and sample handling systems required for different types of porous materials.

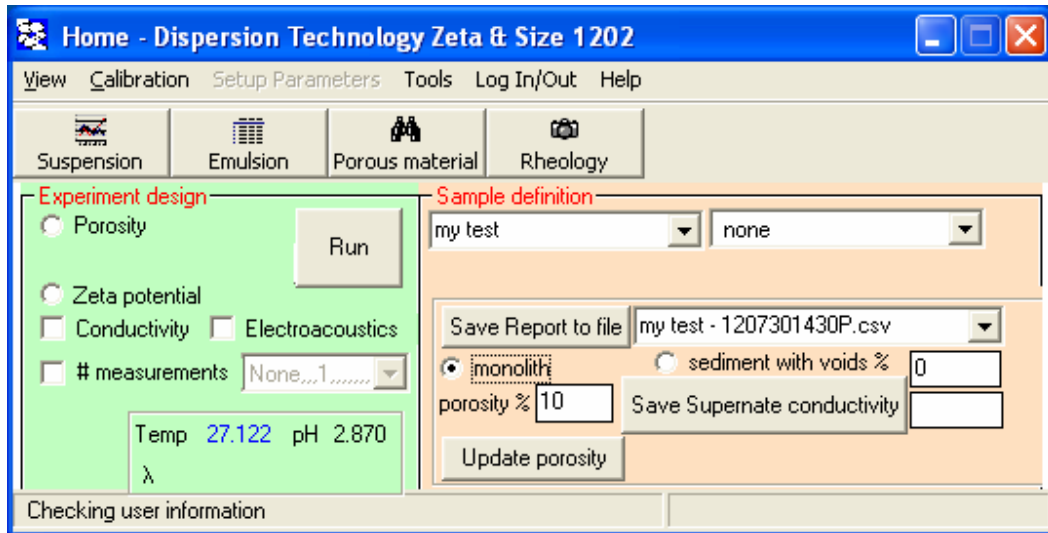
### *Home page*

Experiment setup and results presentation are done directly on the Home page, which looks like this:

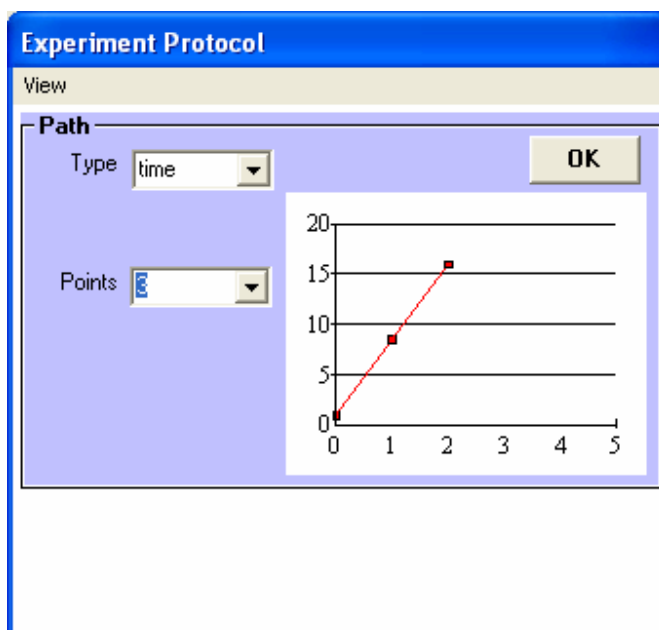




Sample definition box (ID) serves double purpose. It is sample ID and this sample ID is used as a basis for creating a unique file name for saving multiple measurements of all 3 parameters for given sample. We suggest to use it right after you loaded a new sample. Type this sample ID and then click check “POROSITY”. Box next to the button “Save Report to File” contains unique file created in the directory C/data\_aco/Excel where data for this sample would be saved. This file is discussed below in section “Data Storage”.



You can make multiple measurements for any of the 3 measured parameters. Click on the check “# measurements”. The following form appears. You can select desired number of measurements by either selecting number from the list or typing your own. Maximum number is 5000.



Temperature and pH (if installed) are measured continuously every 2 seconds and data is displayed on the bottom frame in the Home page.

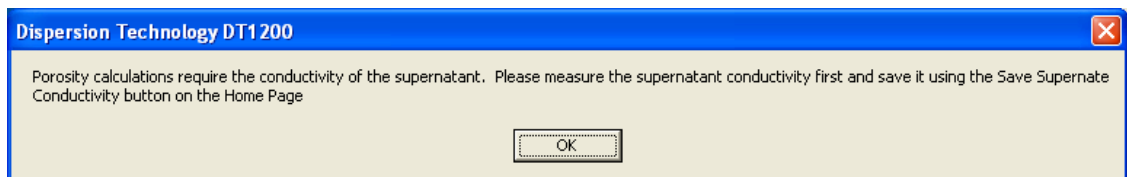
### ***Measurement of POROSITY.***

Measurement of porosity is based on comparison conductivity of the water electrolyte solution that fills pores with conductivity of the porous material that is wetted with this water electrolyte solution. We suggest using 0.1 KCl solution for suppressing surface conductivity.

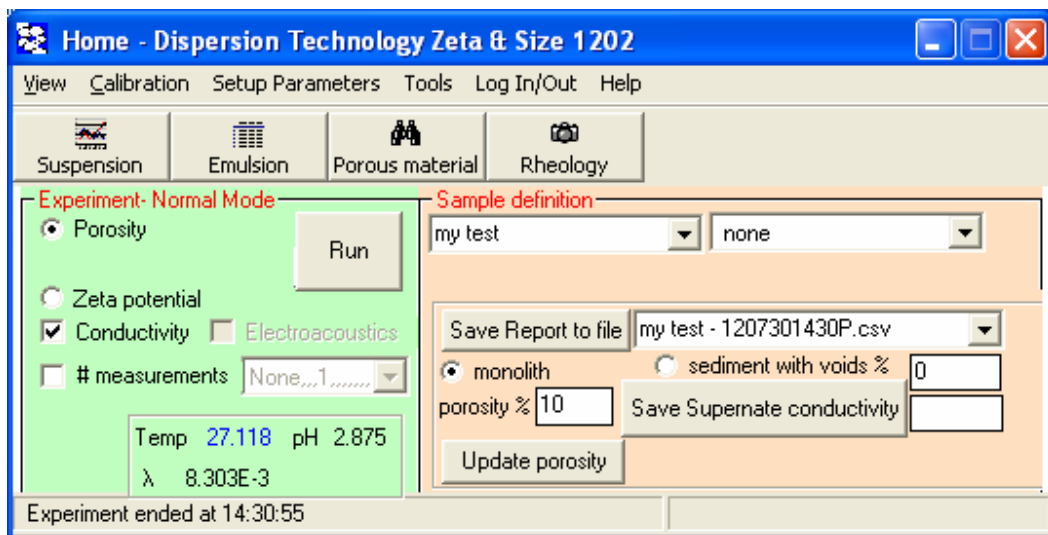
When electric field is applied all ions in pores start oscillating with very small amplitude (<1 A) because of MHZ frequency of applied AC field. It is not important if pores are dead-end or open. All porous space will generate AC electric current.

Measurement requires 2 conductivities (supernatant and porous material). That is why it should be done in two steps. First step is supernatant measurement, the second – porous material.

Home page has check button on the left hand side for Porosity measurement. Check this box as shown on Figure below and insert the conductivity probe in supernatant. Click RUN.

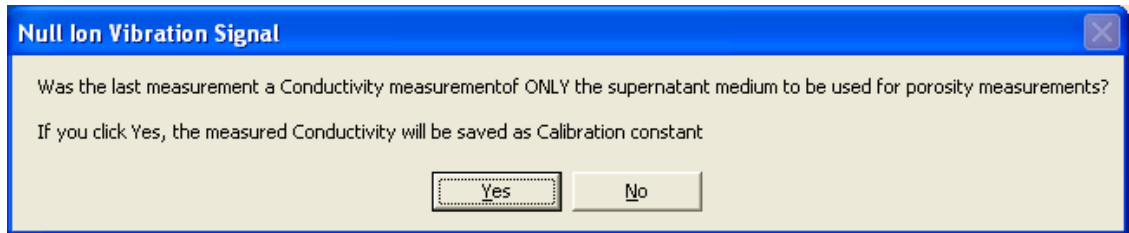


OK.



Conductivity would be measured. Click button “SAVE SUPERNATANT CONDUCTIVITY”

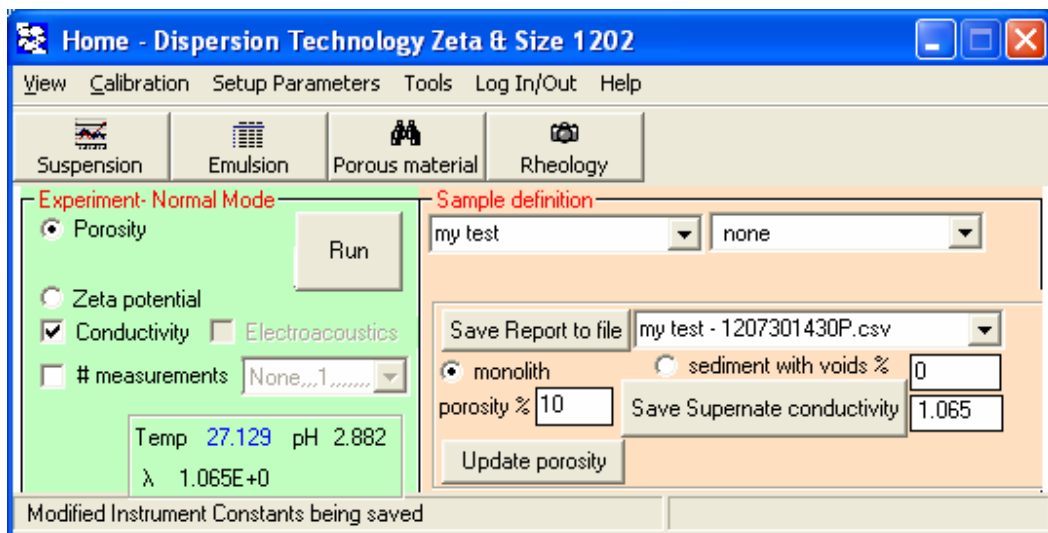
Following message would appear. Click Yes.



Confirming message would appear. Click OK.



Conductivity of supernate would appear in the box, as shown below – 1.065 in S/m.

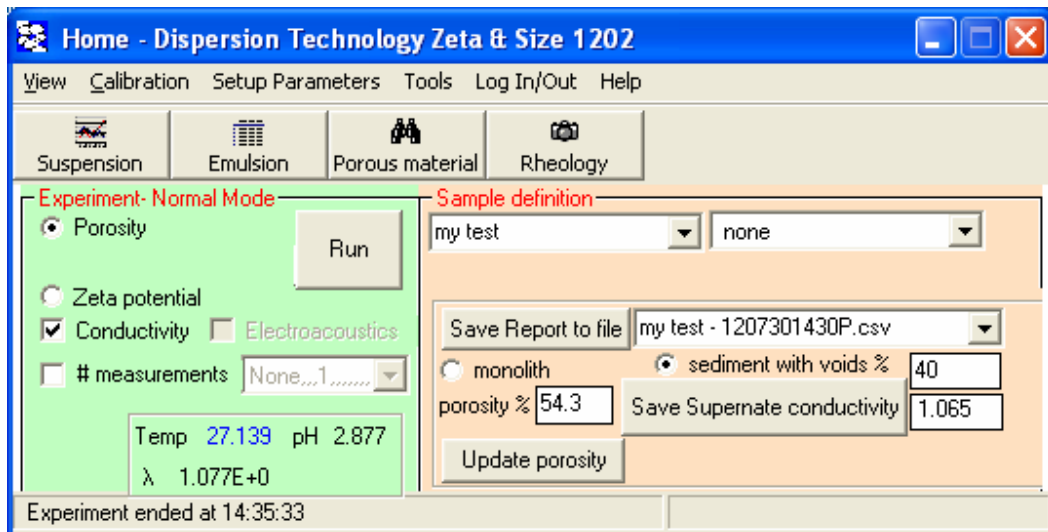
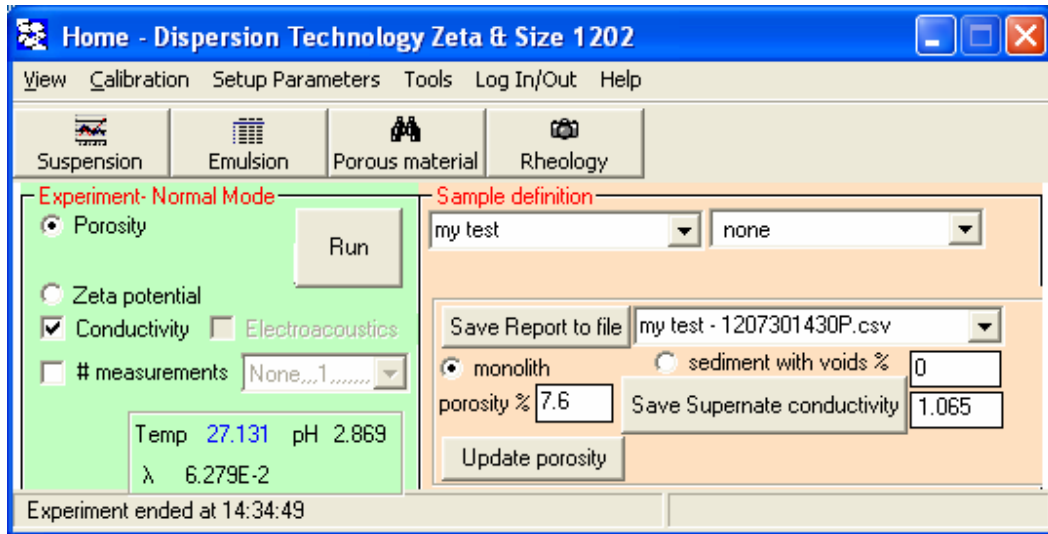


Now you can measure porosity of the porous material that is saturated with this supernate.

Place probe in contact with this material. Then select which type of the material it is: monolith or sediment of porous particles. If it is aa sediment – select volume fraction of VOIDS in %., as it is done on Figure below -40%.

Click RUN.

Porosity would appears in the accordingly labeled box.



You can save this porosity as a property of material. It is important for zeta potential measurement. You should use Define Material form that can be opened from Home Page menu Tools.

Example of this form is shown below. It demonstrates properties of a porous material “alumina test porous”. Currently it is 12.3%.

**New Material** [X]

View

**Specify property value**  
 Click Caption for HELP

material type

density in g/cm.cu

porosity

*SEI=A0+A1\*d+A2\*d\*d; d:pore diameter in nm*

A0

A1

A2

**Modify existind material**  
 Select material name, modify property and click Save

If you click button UPDATE POROSITY on the Home page, then porosity on the New Material form changres to the number that is displayed on the Home page.

**New Material** [X]

View

**Specify property value**  
 Click Caption for HELP

material type

density in g/cm.cu

porosity

*SEI=A0+A1\*d+A2\*d\*d; d:pore diameter in nm*

A0

A1

A2

**Modify existind material**  
 Select material name, modify property and click Save

**CAUTION:** *If you have porous bodies saturated with different liquid, each liquid must be measured and saved before measurement of the corresponding body.*

## ***Measurement of ZETA POTENTIAL***

Measurement of zeta potential of porous material is done with standard DTI zeta potential probe. Software is also very similar. If you click on check box "Zeta potential" on the Home page, it will bring you to the following form:

Liquid media	Density	Content %wt solid
water	.997	
alumina_test_porous	3.9	59.64
Dispersed system Total		

Name of the material that is shown in the box "Solid matrix" is the same as was selected initially on the Home page.

Box "%wt solids" presents weight fraction of solids. It is calculated using 2 numbers: posoty of the selected material as it was saved to the Material database and % of Voids shown on the Home page.

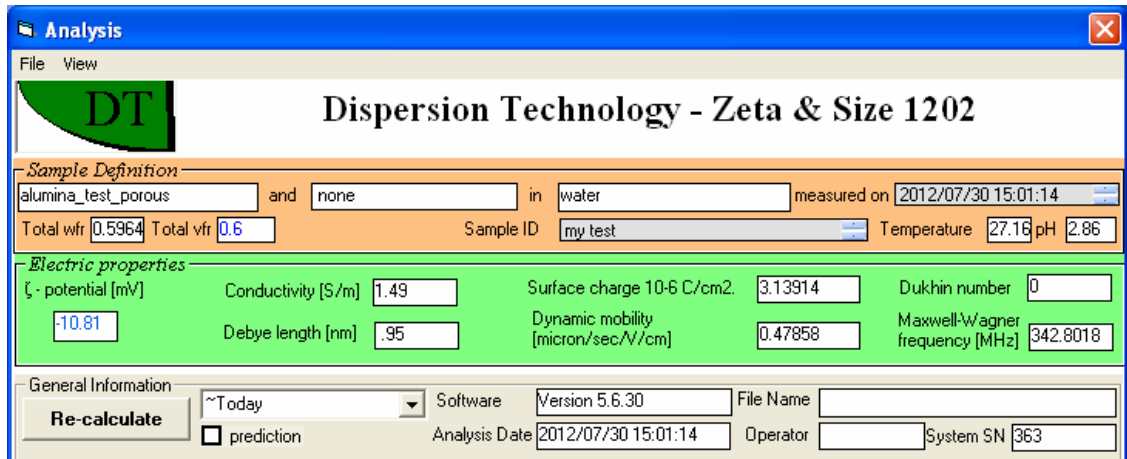
In principle, you can change both, the material and weight fraction of solids. This should be done in particular if you are going to measure dispersion of porous particles. Then you should place %wt of solids of these particles.

When you place focus in %wt solids box, the following message would appear informing you that old data would be erased.

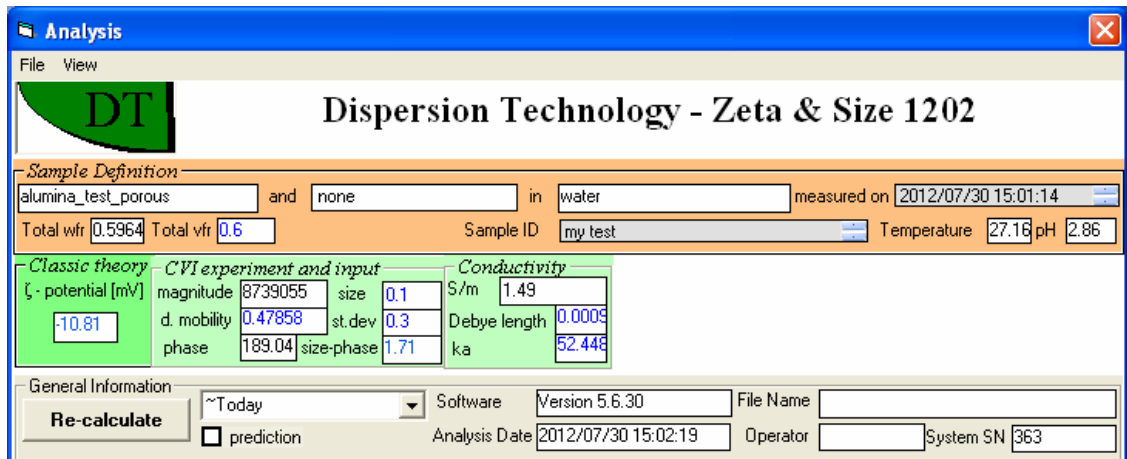
Sample will be over-defined if you make changes. Click OK to erase present values. Click Cancel to keep values

After click RUN, measurement would be conducted and the following form appears..

It shows Zeta potential and other important parameters. On the form below they are given because conductivity was measured together with Zeta potential.



If you want to see raw data, then go to View and uncheck menu Report. The following form appears.



Calculation of zeta potential was performed with simplified version of the Frenkel's theory.:

$$I_{see} = \frac{\epsilon_0 \epsilon_m \zeta}{\eta} \phi_p \left(1 - \frac{\rho_m}{\rho_s}\right) \frac{K_s^{cond}}{K_m} \nabla P$$

where  $\epsilon_0$  and  $\epsilon_m$  are dielectric permittivities of vacuum and liquid,  $\eta$  is dynamic viscosity,  $\phi_p$  is volume fraction of particles, which differs from the volume fraction of solids  $\phi$  in case of porous materials,  $K$  is conductivity. Conductivity of the system  $K_s^{cond}$  that contains conducting porous particles differs from the conductivity of the system with the same volume fraction of solid particles. Ratio of conductivities is

$$\frac{K_s}{K_m} = \frac{1 - \phi}{1 + 0.5\phi}$$

Conductivity of the system with porous particles was estimated with the same expression, but conductivity of particles was different due to internal conductivity.

For comparison we present here expression for CVI of the dispersion solid small particles in Smoluchwsi limit

$$CVI = \frac{\epsilon_m \epsilon_0 \zeta}{\eta} \phi \left( \frac{\rho_p}{\rho_s} - 1 \right) \frac{K_s}{K_m} \nabla P$$

The difference is related to densities and conductivities ratios, which would depend on porosity of particles.

Porosity is assumed to be a property of the material and comes from the Material database.

## Calibrations

Calibration of porous materials requires calibration of two sensors – electroacoustic and high frequency conductivity. There is a special liquid, silica Ludox and 10%wt, for calibrating electroacoustic sensor. Conductivity sensor is calibrated with KCl 0.1 and 0.01 M solutions.

### Aqueous Conductivity calibration

To calibrate the aqueous conductivity probe, first click on the menu item Calibrate – Conductivity. The Conductivity Probe Calibration form will appear as shown in Figure below. Follow the step by step instructions on the form.

**Conductivity Probe Calibration**

Step 1) Empty chamber. Fill chamber with 0.01 M KCl standard and stir 10 sec. Empty chamber, again fill with 0.01 MKCl, and stir. Check that stirrer is working properly. Click on OK when ready to measure

---

**Measured Data**

Standard	Temp.	Actual K	Actual $\epsilon$	Measured K
0.01 M KCl	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
0.1 M KCl	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>

---

**Calibration results**

	New	Old
Response		7.136E+04
Linearity		0.4698

### Zeta potential calibration

The zeta potential is calibrated using a standard colloid having known zeta potential. The DT-1200 employs a standard 10 wt% colloidal silica that is prepared from a concentrated 50 wt% slurry by diluting it with 0.01 M KCl media.

The calibration procedure assumes that the last measurement was of such a zp standard, and that only zp was measured ( i.e. not in combination with attenuation or conductivity).

Following this measurement, on the home page click on Calibrate- Zeta potential. The Zeta Calibration form will appear as shown in Figure below. The measured value will normally be fairly



close to the calibration value if the probe has not been replaced. The zeta potential is calibrated using a standard colloid having known zeta potential.

The calibration procedure assumes that the last measurement was of such a zp standard, and that only zp was measured ( i.e. not in combination with attenuation or conductivity).

Following this measurement, on the home page click on Calibrate- Zeta potential. The Zeta Calibration form will appear as shown in Figure below. The measured value will normally be fairly close to the calibration value if the probe has not been replaced.

Measured Data		
	Standard	Measured
Zeta potential, mV	-38	
Cvi Phase, deg	180.00	

Calibration results		
	Old	New
Cvi Response	7.945E+08	
Cvi Phase offset, deg	327.84	
Reference Phase, deg	101.27	
Temperature, C	25.57	

When you Click OK, Home Page would change Setup as shown below:

Then, after measurement finished, Calibration form would show results of measurement.

**Zeta Potential Calibration - 2011/08/23 14:14:25**

10 wt% Ludox silica in 0.01M KCl at pH 9.1-9.3

Step 3) Click Save to save new calibration permanently. Click on Cancel to forget new calibration.

Cancel

Save

Multi-Freq

Measured Data	Standard	Measured
Zeta potential, mV	-38	1.18
Cvi Phase, deg	180.00	356.49

Calibration results	Old	New
Cvi Response	7.945E+08	2.5587E+10
Cvi Phase offset, deg	327.84	142.32
Reference Phase, deg	101.27	97.26
Temperature, C	25.57	25.85

Click on the Calculate calibration button to compute the new calibration constants. If the calibration is successful, the Save button will appear, as shown in Figure below. Click on the Save button to permanently save the new constants and exit this calibration form

### ***Define new material.***

Software operates using database located in the directory C/DATA\_ACO, file DT\_ACO.MDB. It is MS Access database. It has table Materials. Introduction of the Material saved to the database is important because calculation of zeta potential and pore size requires some input parameters that can be assigned to the material.

For zeta potential these are:

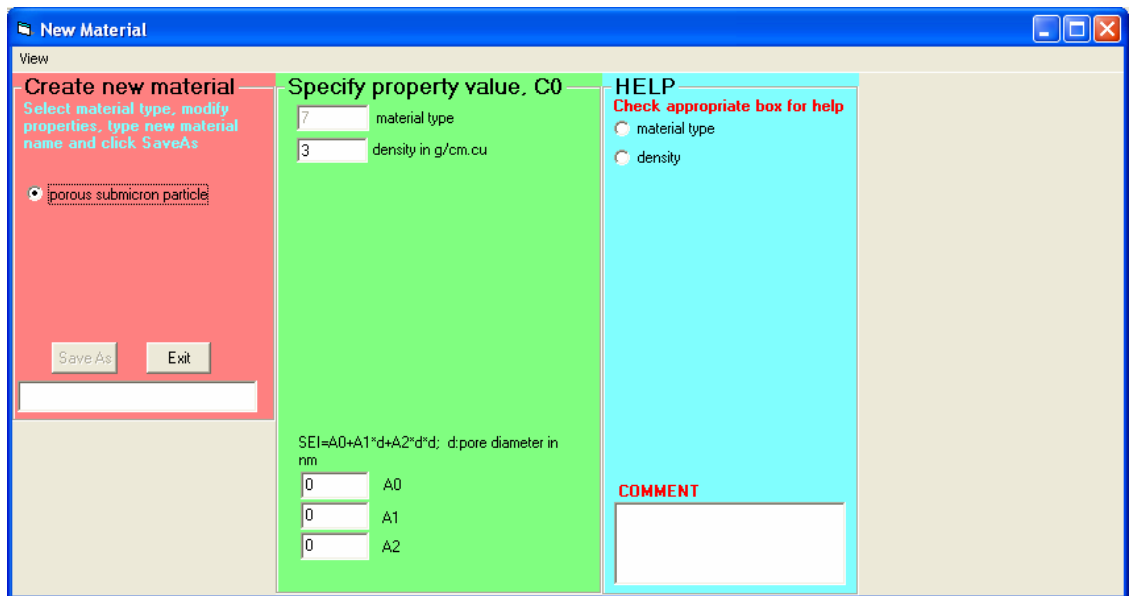
- viscosity, density, dielectric permittivity of the liquid
- density of the porous material solid

For pore size: polynomial coefficients of the electroacoustic dependence on pore size for given material.

Software contains a special form that allows introduction of the new material with particular properties. This form appears after Home page, Tools, Define material:

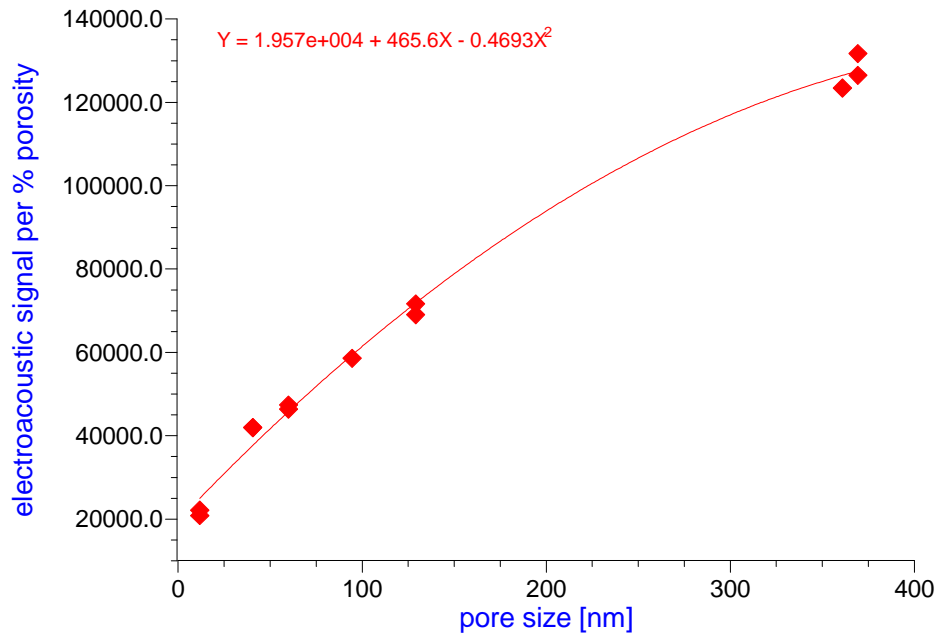


Click on the check button “porous submicron particles”. Boxes for material properties appears:

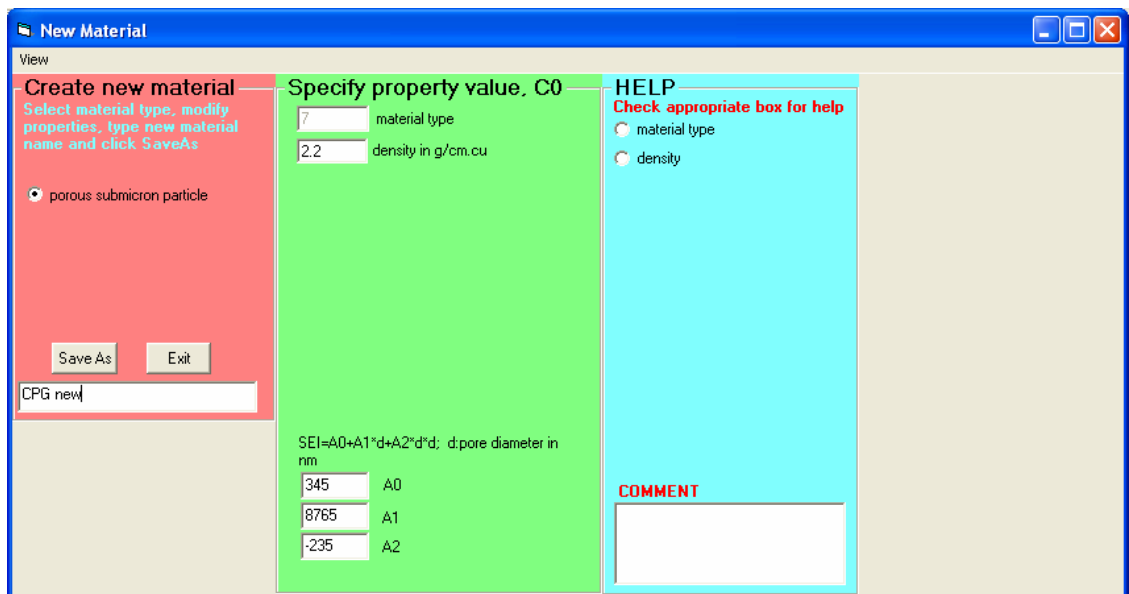


Density corresponds to the density of the porous material solid matrix. For instance, for silica it is 2.2.

Bottom 3 boxes are reserved for polynomial fit of pore size dependence. One must have electroacoustic signal measured for several powders of the same chemical nature but different pore size. Then, these data should be fit with second degree polynomial as shown below.



Coefficient in the expression are placed in boxes, as shown below with their signs.



Then type name of this new material.

After saving, you would be able to select it from the combo box on the top right hand side corner of the Home page.

### ***Data storage.***

All results are automatically saved to the MS Access database C/data\_aco/dt\_aco.mdb. This file **MUST** be backup often.

There is form allowing seeing, exporting and importing data, as well as making queries. It is Home page, View, Data.

In addition, results of the particular sample measurement can be saved to the unique Excel file in the directory C/data\_aco/Excel. Here is an example of such file.

In addition to the results of individual measurements it contains statistical averages and deviations.

"Sample ID"	"CPG DTI"	"CPG water"		
"Measurement Date"	"Porosity %"	"Pore size nm"	"Zeta potential mV"	"Temperature"
#2011-08-23 10:42:59#	" 72.6 "	" 0 "	" 0 "	"25.46 "
#2011-08-23 10:43:22#	" 72.4 "	" 0 "	" 0 "	"25.46 "
#2011-08-23 10:43:44#	" 72.4 "	" 0 "	" 0 "	"25.46 "
#2011-08-23 10:44:46#	" 72.4 "	" 56 "	" 0 "	"25.45 "
#2011-08-23 10:45:08#	" 72.4 "	" 54 "	" 0 "	"25.45 "
#2011-08-23 10:45:31#	" 72.4 "	" 54 "	" 0 "	"25.45 "
#2011-08-23 10:46:11#	" 72.4 "	" 0 "	"-5.36 "	"25.45 "
#2011-08-23 10:46:33#	" 72.4 "	" 0 "	"-5.34 "	"25.45 "
#2011-08-23 10:46:55#	" 72.4 "	" 0 "	"-5.2 "	"25.45 "
#2011-08-23 10:50:09#	" 72.4 "	" 56 "	"-5.2 "	"25.45 "
"Average"	" 72.46667"	" 55 "	"-5.3 "	
"Ave. Abs. Dev. "	" 0 "	" 1 "	" 6.666676E-02"	
"St.Deviation"	" 0 "	" .5 "	" 4.109618E-02"	

### ***Sample Handling***

Sample handling is different for porous body build by particulates as deposits and macroscopic porous body.

In case of particulates, it is convenient to built up deposits directly on the surfaces conductivity probe and electroacoustic probe. There are special fixtures that are placed on the top of the probes for forming a cups. Samples can be filled directly in the cup.

***Figure 1.*** Electroacoustic probe with a cup top for porous deposits.



***Figure 2.*** Conductivity probe with a cup top for porous deposits.



Measurements of macroscopic porous bodies can be done by direct contact of the probe with the body saturated with appropriate liquid. This should be done directly in the cup filled with liquid saturating porous body. Equilibration of the macroscopic porous bodies takes a long time, hours.

Contact of the probe with the body must EXCLUDE liquid. This can be done only for very flat parts of the porous body surfaces. We suggest to perform sanding of appropriate parts.

It is important to ensure that all prepared flat parts of the porous body are exposed to the liquid.

Do not measure bottom part of the porous body. Equilibration there is slowed down due to narrow gap between the vessel and the body.

*Figure 3. Electroacoustic probe in contact with sandstone core.*



*Figure 4. Conductivity probe in contact with sandstone core.*





There is also fixture that allows accommodation electroacoustic, conductivity and pH probes together, Figure 5.

***Figure 5. Electroacoustic, conductivity and pH probes together.***







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