Instruction Manual for BI-XDCW
X-ray Disc Centrifuge System

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Please Read

This is your instruction manual for your Brookhaven X-ray Disc Centrifuge system and BI-XDCW control software. Please read it carefully before attempting to plug in and use the equipment to make measurements. The **Installation** section, Section II, describes procedures for checking that the instrument and the software are working properly. Cleaning and changing discs, repositioning the detector and other maintenance procedures are described in the appendices. You may familiarize yourself with some of the features of this software by loading sample data files (Files/Database). You can run Modeling Utility, view results, display distribution tables and graphs to test and understand the operation of the software. In Section V, **Making a Measurement**, all the detailed procedures of preparing a sample, making a measurement and printing reports are given. If you have any questions or suggestions, please contact Brookhaven Instruments.

This program requires Windows 3.X or higher and at least 8 Mb of RAM.

DO NOT ATTEMPT TO DEFEAT THE X-RAY INTERLOCKS ON THE ANALYZER. THESE ARE PROVIDED FOR THE SAFETY OF THE OPERATOR. THE FACTORY SHOULD BE CONTACTED IF THE INTERLOCKS MALFUNCTION.

DO NOT PUT STRONG SOLVENTS IN THE STANDARD DISC. THAT WILL RUIN THE DISC. The standard disc is made of poly (methyl methacrylate), PMMA. Also known as Plexiglas, Lucite, and Perspex, this material will be attacked by strong acids or bases and many common organic solvents such as acetone and toluene. It is resistant to water, simple alcohols, and to aqueous solutions of sucrose, ethylene glycol and glycerol. If in doubt, contact the factory for advice. Brookhaven Instruments cannot be responsible for warranty replacement of PMMA discs ruined by the use of incorrect solvents.

MAKE SURE THAT THE SHIPPING SCREW IS CORRECTLY INSTALLED, BEFORE TRANSPORTING THE ANALYZER. Instructions for installing the shipping screw can be found in Appendix I.

Software is never really finished: there are always additions and changes. As these become available, they will be added to the back of this manual as appendices. Please look at the appendices if you cannot find the answer to your questions in the main part.

Remember the old saying: "When in doubt, read the instruction manual." Sometimes the solution to your problem has already been addressed. You just need to find it. Thanks for purchasing a Brookhaven.

Copyright Notice

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Warranty

Brookhaven Instruments Corporation (hereinafter known as BIC) warrants that the product is free from defective material and workmanship. Under the terms of this warranty BIC agrees to correct by repair, or at BIC's election, by replacement any parts which prove to be defective through no fault of the user.

This warranty is limited to the original purchaser of the product.

The product shall be shipped, freight prepaid and insured in full, or delivered to a facility authorized by BIC to render the service provided hereunder, in either the original package or a similar package affording an equal degree of protection. The purchaser must contact BIC for instructions prior to returning the product.

The product shall not have been previously altered, repaired or serviced by anyone other than a service facility authorized by BIC. The product shall not have been subjected to accident, misuse, or abuse, or operated contrary to the instructions contained in the operating manual or manuals.

BIC shall not be liable for direct, indirect, incidental, consequential, or other types of damages, resulting from the use of this product other than the liability stated above. These warranties are in lieu of all other warranties, expressed or implied, including, but not limited to, the implied warranties of merchantability or fitness for a particular purpose.

The BIC warranty extends for a period of 90 days. This period begins from the date of receipt of the equipment, and it applies only to the original purchaser. The warranty period is automatically extended to 1 year (except as noted below) from the date of receipt of the equipment provided all invoices for said equipment, including transportation charges if applicable, are paid within 30 days after receipt of invoice.

The BIC warranty extends for a period not exceeding the warranty period of the original equipment manufacturer where applicable. The typical warranty period on printers and computer peripherals is 90 days, and on photomultiplier tubes it is 180 days. Please contact BIC for copies of applicable OEM warranties.

The BIC warranty does not cover scratches or haze which may develop in disc cavities as a result of improper sample preparation or use. Nor does the warranty cover damage to a disc from the use of incorrect solvents. SEE THE COVER PAGE OF THIS MANUAL.

Software License Agreement

Carefully read the following terms before using the software provided. Use of the software indicates your acceptance of these terms. If you do not agree with the terms, promptly return the software. BIC refers to Brookhaven Instruments Corporation.

Terms:

1. In purchasing this software you are granted a nonexclusive license to use the software product on one computer.
2. BIC retains title to, and ownership of, the software product. The software product may not be modified without the express written consent of BIC.
3. Duplication of the software product for any purpose other than backup protection, including duplication for any commercial use, is a violation of the copyright laws of the United States of America and of other countries.

Information produced by using this BIC software and its manual, including the resulting displays, reports, and plots, are believed to be accurate and reliable. However, Brookhaven Instruments Corporation assumes no responsibility for any changes, errors, or omissions.

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Appendices

Section I: Introduction

General Description

Absolute particle size distribution for metal oxides, ceramics, clays, catalysts, and many other types of small particles is readily obtainable by sedimentation. As X-rays are absorbed in proportion to particle mass, no optical corrections are necessary. This makes for a simple, accurate mass detector. This method thus produces a cumulative mass/volume distribution by measuring the attenuation of an X-ray beam passing through a liquid medium as the particles pass through the X-ray beam. As the measurement progresses, the source/detector scans toward the surface of the liquid, speeding up the measurement. High throughput is the result. The differential form of the mass distribution can also be calculated. Average sizes and other useful particle size distribution data can be calculated from the distribution data.

The X-ray disc centrifuge (XDC) method allows you to make measurements in either a centrifugal or a gravitational field. Typically a relatively large volume (~ 15 mL) of homogeneous suspension is injected into the disc (disc rotation is commenced for a centrifugal mode experiment), and data collection initiated. This is called the homogeneous start method. Data from two runs on the same sample in different modes (centrifugal and gravitational) can be combined yielding a broad size range for the measurement.

Specifications

The BI-XDC consists of the analyzer and control software. The analyzer system includes the following: motor and controller; keypad entry with LCD readout; disc and disc injection port; low power, air-cooled, scanning X-ray source and photomuliplier + scintillator detector; and built in stroboscope. (The built in strobe has two separate uses. First, it is used to confirm the stable rotation of the shaft. For this reason it is not synchronized to the rotation of the shaft. Rather it is synchronized to the motor drive circuit. Second, it is used for detecting unstable sedimentation during the measurement.)

The analyzer drive system has a microprocessor controlled, digitally driven electronic motor and a digital read-out for setting and monitoring speed. With the PMMA disc the speed can continuously vary from 600 to 6,000 rpm. There is an optional disc (BI-DSCXH) which can spin continuously from 600-10,000 rpm. The speed accuracy and stability is better than ± 0.01 %. The standard disc, the BI-DSCX, is made of poly (methyl methacrylate) with a stainless steel hub. It is dynamically balanced over the range of operating speeds and can hold spin fluid volume from 10 to 25 ml.

A Windows-based software package for data acquisition, analysis, and management is included. A PC is required and may be supplied by the user or optionally by Brookhaven Instruments Corporation.

The BI-XDCW software is designed for use with the BI-XDC particle size analyzer. It is written in C++, suitable for use with Windows 3.11 or higher. We recommend the use of at least 8 MB of RAM and either a 100Mhz 486 or Pentium PC. The program acquires data from the analyzer, calculates the results, manages a library of data files, and allows the user to plot different graphs and reports.

Windows Software Version

Listed below are some of the features of the system:

- Real-time data plotting on the monitor during measurement.
- Provides on-screen and hardcopy reports of selected statistical measures of the mass distribution and tables of cumulative distribution data.
- Generates on-screen and hardcopy plots of the raw data curve with baseline and the differential and integral forms of the volume (mass)/surface-area/number distribution.
- Distributions can be displayed on a logarithmic or linear scale.
- Six distributions can be graphically overlaid and compared.
- A complete database management system by folders and files.
- Ability to design various report printouts, including a preview feature.
- Generates statistical process control chart with printouts.
- Ability to see the detail results on the screen.
- Can import old DOS runs and save then in the Windows' file system.
- Graphs and results can be viewed as volume-, surface-area- or number-weighted distributions.
- Display of cumulative volume/mass, surface area and number distributions on the same graph.
- Detailed size distribution tables can be viewed on the screen or printed as a report.
- Two files can be merged to get a complete size distribution.
- Ability to reanalyze measurements.
- Graphs can be scaled automatically or manually and as log or linear functions.
- Automatic calculation of viscosity and refractive index as a function of temperature for several common liquids.
- Modeling utility to quickly optimize run time and disc speed for a given size range.

DISC SPECIFICATIONS

Number 1: Type Standard

Serial Number:

Disc Cavity Width in cm:

Disc Cavity Radius in cm:

Detector Radius in cm:

Number 2: Type Standard

Serial Number:

Disc Cavity Width in cm:

Disc Cavity Radius in cm:

Detector Radius in cm:

Original Purchaser

Organization

Address

–

Section II: Installation

Installation

Computer Supplied By Brookhaven

Your BI-XDC consists of the analyzer with power cord, software, cable, manual, and a disc. The computer, monitor, and printer are optional. Or, the user may supply a suitable PC. The software is installed on the hard disk. Examine the shipping containers for external damage. Carefully remove all items from the shipping containers. Pay particular attention to manuals and detachable power cords. If any damage is found, please contact the carrier and Brookhaven immediately. Make sure to save all packing materials and containers.

1. Remove the cover of the computer. Check for loose boards or connectors or any other sign of internal damage. Correct any obvious problems, or call your local sales office if the problems appear to me more serious. Replace the cover when finished.

2. Plug the keyboard and monitor into the appropriate connectors on the rear of the computer. These connectors are polarized and labeled, and they can only be inserted in one position. Use the screws or spring clips provided with the connector to secure it.

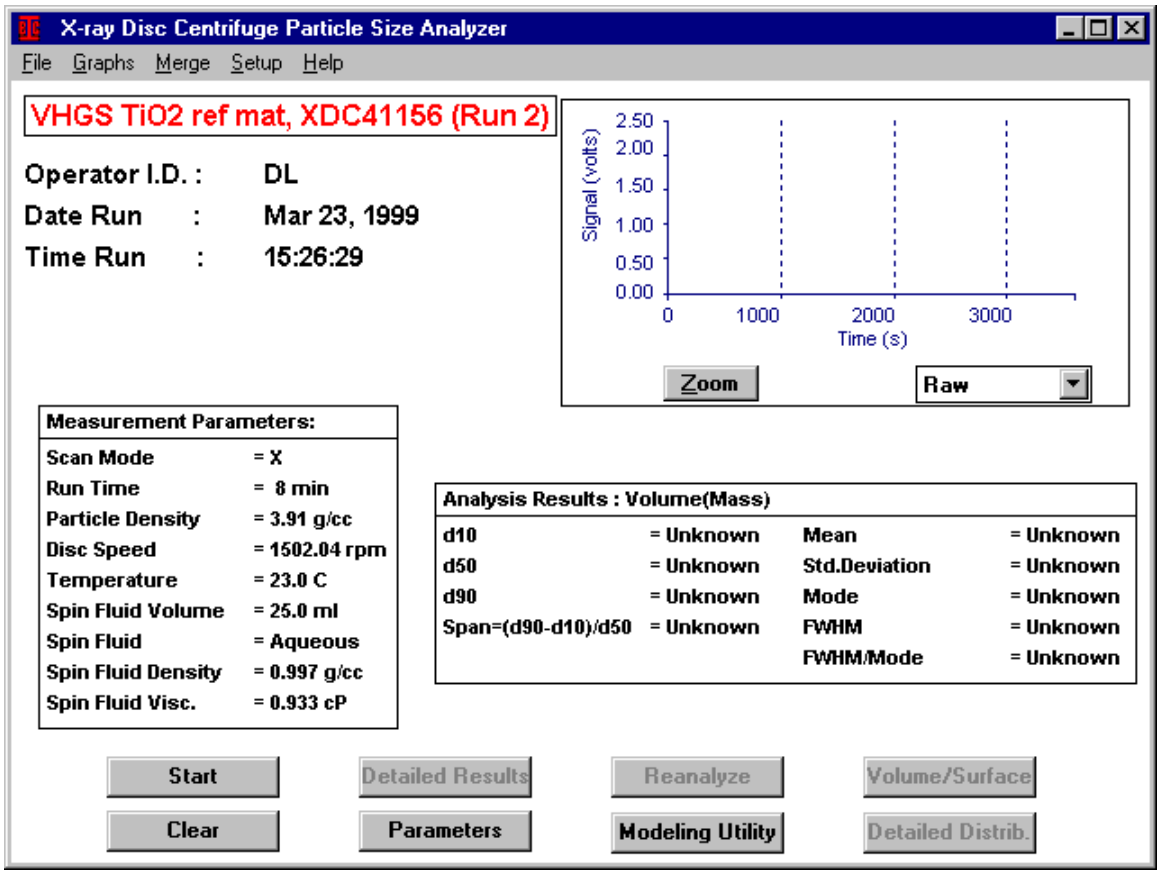
3. Verify the voltage selector on your computer and monitor is set to the proper voltages. Plug the computer into an outlet and plug the monitor into the special outlet located on the back of the computer. [Some of the newer CE marked computers do not have power output capability, and the monitor must be plugged directly into an AC source.] Turn on the computer and monitor.

4. Verify that the Program Manager of Windows, or the equivalent in Windows 95, is displayed and that a BIC Window or icon is displayed labeled “BI-XDCW Particle Size Analyzer”. If the BIC window is not installed, do it now using the diskettes supplied. To install the BI-XDCW software, place the installation disk in the floppy drive. Click on **Run** from the **File** menu. Run A:\SETUP. When finished, a BIC icon with the name BI-XDCW Particle Size Analyzer will be added to your BIC program group, or a group with this icon and name will be added. Double click on the icon to start the program.

5. If you ordered a printer, follow the instructions in the printer manual to test the printer. Connect the printer to LPT1 of the computer using the appropriate cable.

Testing the Software

Double click on the BIC icon, the one labeled BI-XDCW Particle Size Analyzer. An image similar to the following should be visible on your screen. This is the Main Window.



Click on **File** in the Main Window Menubar and then on **Database**. Open a demo file by double clicking it. Now you will see the raw data graph in the right corner of the screen and the results below it. The Sample I.D., date, and other input parameters are displayed in the left corner of the screen. Click on **Zoom** to see a bigger graph. Various graphs, detailed results, and size distribution tables are displayed. Similarly, distributions weighted by volume, surface area or number can be seen. Click on **File** and **Print Report**, you will get a print out of the summary of results for the current file. You can print other reports.

Click on **Setup** in the Main Window Menubar and then on **Instrument Parameters**. The displayed disc parameter values correspond to the disc that is mounted

in the XDC analyzer. Verify that these parameters are the same as the disc parameters given in Section I, Introduction. The default connection between the analyzer and the PC is through the COM1 port. You may change to COM2 using the drop-down list-box.

Analyzer

Note: The analyzer is shipped with the X-ray source and detector assembly mechanically locked to prevent damage to the moving parts during transport. The instructions for removing the shipping screw can be found in Appendix I. Please note that if the analyzer is to be shipped anywhere, the shipping screw and washer must be reinstalled.

Remove the shipping screw.

Install the centrifuge disc as outlined in Appendix II, and then align the detector as outlined in Appendix III.

Plug the power cord into the rear of the analyzer. Plug the other end into a suitable source of power.

Turn on the power of the analyzer. The LCD display should light up.

Press the yellow button on the front panel marked HEAD. The X-ray source and detector assembly should automatically move until it is in front of the disc. Make sure that the head does not touch the disc at any point.

Open the door and inject 15 ml of water into the disc. Insert the key for the X-ray source and turn it on. Close the door. The X-RAY ON indicator lamp on the front panel should light up. The signal on the LCD display should be between 2.0 and 2.4 volts.

Enter 3000 on the keypad. Each digit entered is shown on the last line of the LCD, the COMMAND LINE. Use either the left arrow or the ERASE LINE key to make corrections. Press the RPM button to implement your choice. The exact rotational speed, a value very close to 3000 RPM is displayed on the first line of the LCD.

Press the red button marked MOTOR. Verify that the disc rotates. Press MOTOR again to stop the disc. It should stop very quickly. There is an electronic brake for safety. Start the disc rotating again.

Press the button marked STROBE. Verify stable operation by noting that the disc position is apparently frozen. If it wobbles back and forth, stop the motor and contact the factory.

Verify stable operation at 4000, 5000 and 6000 rpm. Speeds may be entered on the keypad and implemented by pressing the RPM key while the disc is rotating. This will not

affect the operation of the instrument as long as a measurement is **not** in progress. Note: The strobe automatically shuts off after 5 minutes to prolong its life. To reactivate the strobe, just press the key again.

Set the speed to 3000 rpm.

Open the door of the analyzer. Verify that the motor stops rapidly and automatically. The X-RAY ON indicator should also go off. Note the small switch in the left hand side of the opening.

Turn the analyzer off.

Connect the analyzer to the computer using the flat cable supplied for this purpose. The connectors are polarized, and care should be taken to install them correctly. You can use either the COM1 or the COM2 port on the computer. The analyzer uses a 25 pin connector, and the PC uses a 9-pin adapter. Brookhaven supplies as standard a 25-pin to 9-pin adapter cable.

Insert the flexible tubing supplied through the guide tube that is installed in the door. Make sure that it goes smoothly into the disc cavity when you push it through. The position of the guide tube can be adjusted by loosening the two small screws that hold the plastic blocks in place with the disc removal tool. Then push it gently in or out as desired and tighten the screws to lock the tube into place. Make sure that it does not touch the disc when the door is closed.

Cooling requirements for the XDC

The BI-XDC is air-cooled. Room temperature air enters through a filter installed in the top panel at the rear and directly flows over the disc and X-ray head. As it does so, it removes the hot air built up by the friction of a high-speed disc rotating in air. The air is then pulled through the motor by a special set of fan blades built into the motor itself. The air exits through rear panel openings assisted by two fans.

NOTE: NEVER BLOCK THE FANS OR THE INTAKE FILTER.

Remove, clean and replace the filter every 3 months.

The installation of the BI-XDCW software and the XDC analyzer are complete:

- The computer works;
- The program can be executed;
- You can print out results;
- The analyzer works and is connected to the desired COM port.

One last piece of advice:

MAKE FREQUENT COPIES OF YOUR DATA FILES

If any of the hardware fails Brookhaven Instruments can repair it. If any of the programs are corrupted or lost, we can send you copies. We cannot, however, recover your lost data files. That is your responsibility. After all, that distorted looking size distribution may turn out to be the founding piece of evidence in a new branch of materials science: if only you had a copy of it.

Section III: Principles of Operation

Basic Operation

X-rays from a low power X-ray tube are passed through the disc. The intensity of the beam is attenuated in proportion to the concentration of the suspension through which it passes. The intensity of the transmitted beam is measured with a detector consisting of a scintillation counter whose output is recorded by the computer as a function of time.

A small volume of the spin fluid is injected into the stationary disc. The signal is measured over an interval of ten seconds and saved. This signal corresponds to 0% concentration, as there are no particles present in the suspension. The spin fluid is then removed.

A large volume (typically 15-25 ml) of the actual suspension is then introduced into the stationary disc, and the signal is measured over 10 seconds and saved. During the measurement, it is important that the suspension is homogeneous and a mixing feature is provided for that purpose. This signal corresponds to 100% concentration.

The motor is started and the signal is measured as a function of time. As the measurement progresses and particles sediment out of the suspension, the concentration decreases until eventually no more particles are present in the path of the detector. In order to speed up the experiment the detector scans upward while the measurement is in progress. The scanning speed is calculated by the XDC program based on the volume of fluid in the disc and the total measurement time.

Measurements can also be made in the gravitational sedimentation mode (i.e. the disc is stationary throughout the experiment). This mode is preferred when the sample contains particles that are too large to be measured in the centrifugal mode, typically when a substantial fraction is above a few microns.

Theory

For a homogeneous suspension, the attenuation of the X-ray beam is proportional to the mass concentration of the suspension at the measurement radius. The size of the largest particle present in the suspension can be calculated using Stokes' equation and the mass concentration undersize can be determined using Kamack's equation[1].

The diameter, D_m, of the largest particle in the x-ray beam at time t_i is given by stokes equation:

$$D_m^2 = \frac{18\eta_f \ln(r_i/S)}{\omega^2(\rho_p - \rho_f)\cdot t_i}$$

[1]Kamack,H.J. (1972), Br. J. Appl.Phys., 5, 1962-8.

where: η_f = viscosity of the liquid medium without particles
r_i = radial position of source and detector
S = radial position of suspension surface (meniscus)
t_i = measurement time
ω = radial velocity of the centrifuge disc
ρ_p = density of particle
ρ_f = density of liquid medium without particles

C_i, the measured suspension concentration at radius r_i and time t_i , is determined from the following equation:

$$I_t = I_0 \exp(-BC_i)$$

Where I_t is the measured intensity of the emergent beam with suspension in the disc
I_0 is the measured intensity of the emergent beam with clear liquid in the disc
B is a constant

The X-ray density, ε, is defined as:

$$\varepsilon = \log_{10} \frac{I_0}{I_t}$$

The mass fraction smaller than D_m is given by:

$$F(D_m) = \int_0^i (r_i/S)^2 \, dC_i$$

This innocent looking equation is Kamack's equation. Kamack solved the above equation for a fixed detector radius r and this solution was later extended to a variable detector radius r_i . It accounts for radial dilution as the particles become less concentrated as they move radially outward during centrifugation.

The solution for the above equation for a variable measurement radius may be expressed by the following iterative equation:

$$F_i = \frac{1}{2}(y_i - y_{i-1,i})C_i + \sum_{j=1}^{i-1}\left[\frac{y_i - y_{i-1,i}}{y_{j+1,i} - y_{j,i}} - \frac{y_i - y_{i-1,i}}{y_{j,i} - y_{j-1,i}}\right]F_j$$

Where:

$$y_i = \left(\frac{r_i}{S}\right)^2$$

$$Y_{j,i} = y_i\left(\frac{D_j^2}{D_i}\right)$$

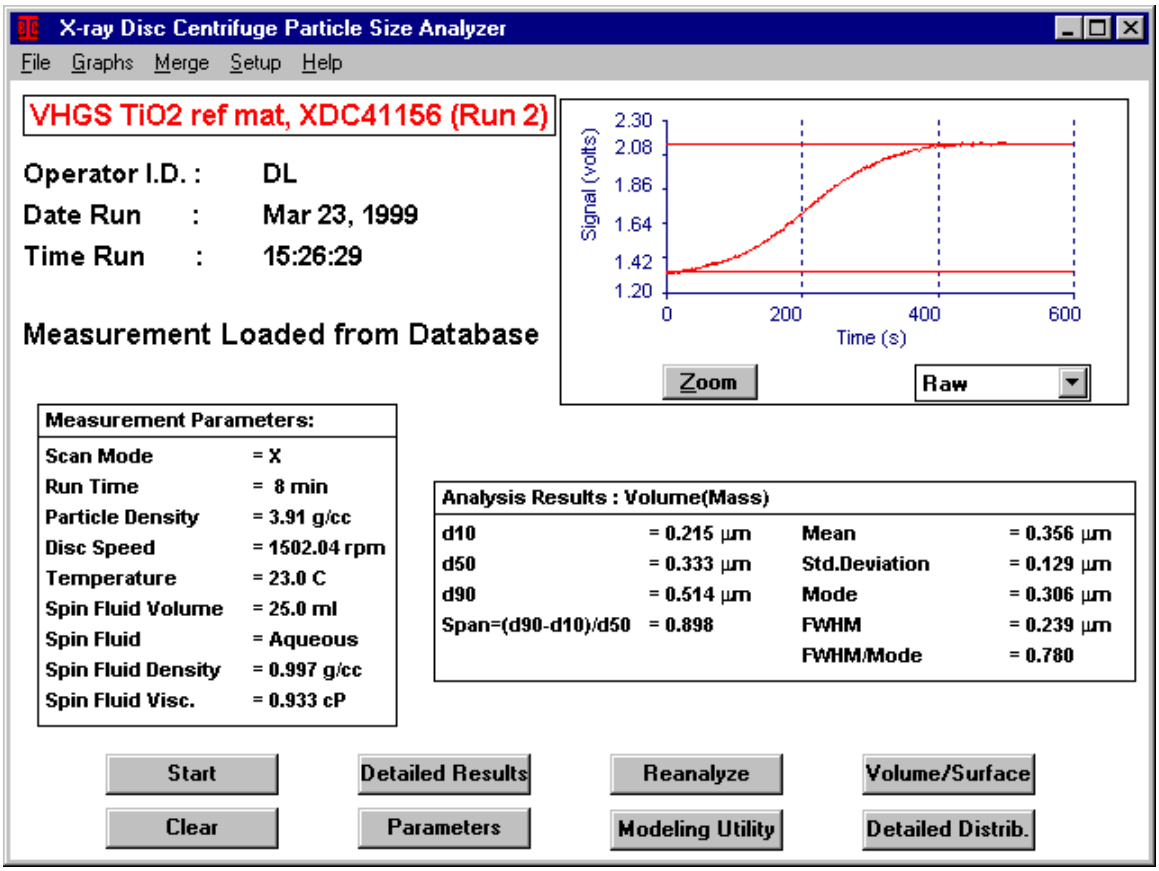
$$y_{0,i} = 1$$

$$y_{j,i} = y_i$$

r_i = the radial distance from the center of rotation to the measurement beam
D_i = is the diameter of the largest particle in the measurement beam at radius r_i and time t_i
n = is the number of data points selected
i = is an integer from 1 to n
F_i = is the mass percentage of the powder smaller than D_i
C_i = is the concentration

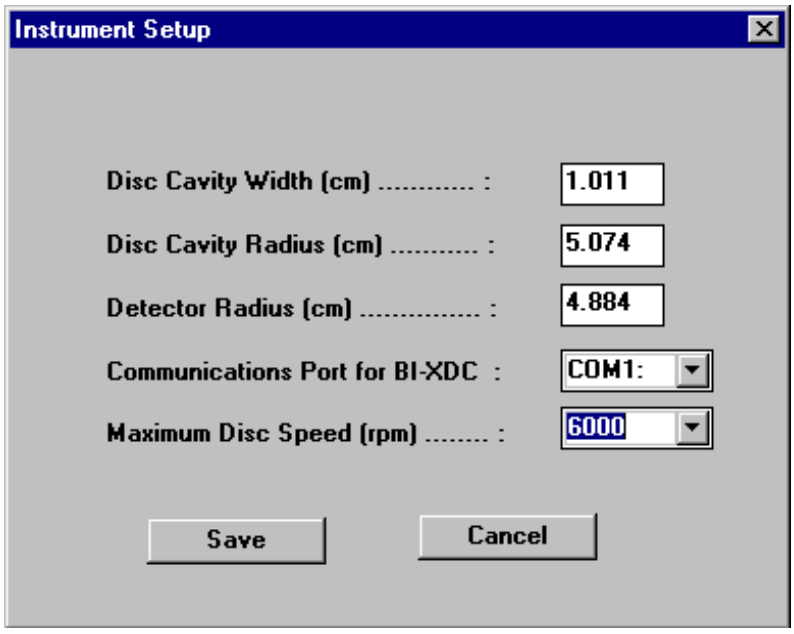
Section IV: Software Overview

A general overview of the program and its important features are presented in this chapter. Double click on the BIC icon labeled BI-XDCW Particle Size Analyzer, and the Main Window with default parameters appears as shown below. The software is self-explanatory. Command buttons at the bottom of the screen enable the user to make measurements, reanalyze data, visualize detailed results, and size distributions. The Menubar of the Main Window has file utilities, data graphs, setup parameters and a merge routine. Try to set the instrument parameters, open sample data files and run the modeling utility, to familiarize yourself with the software before making measurements.



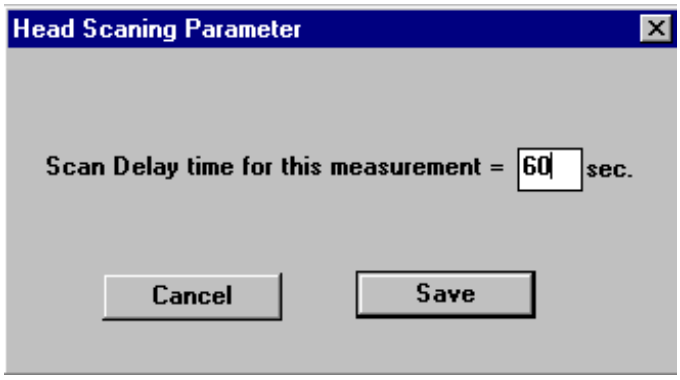
In the Menubar of the Main Window, click on **Setup, Setup Instrument Parameters**. The screen shows the parameter values of the disc that is mounted on the XDC analyzer. Verify that these parameters are correct, and save them for all the measurements. Each time your raw data is saved, the parameters at run time are saved with it. When you reanalyze you can alter the Sample I.D., particle density, etc., but you cannot change disc parameters.

The disc calibration parameters (width, radius, and detector radius) in centimeters are provided with each disc. Please see the end of Introduction section, section I.



When changing a disc, be sure to change these values. Otherwise your answers will be systematically incorrect. The detector radius corresponds to the meniscus position when the disc is spinning at 1500 rpm with exactly 6 ml of water. Therefore when changing discs, the detector position must be reestablished using the procedure described in Appendix III. The RS232 (serial) communications port is used for data transfer between the computer and the analyzer. From the pull down list box, select the communications port, used on the PC. If you don't select the correct communications port, then when you start the measurement, you will get ″Comport initialization failed″error. You only have to input these parameters once, each time a disc is mounted.

With the PMMA disc the speed can continuously vary from 600 to 6,000 rpm. There is an optional disc (BI-DSCXH) which can spin continuously from 600-10,000 rpm. From the pull down list box select the maximum disc speed that corresponds to the mounted disc. By selecting the wrong maximum disc speed there is a danger of damaging the disc. You only have to input these parameters once, each time a disc is mounted.

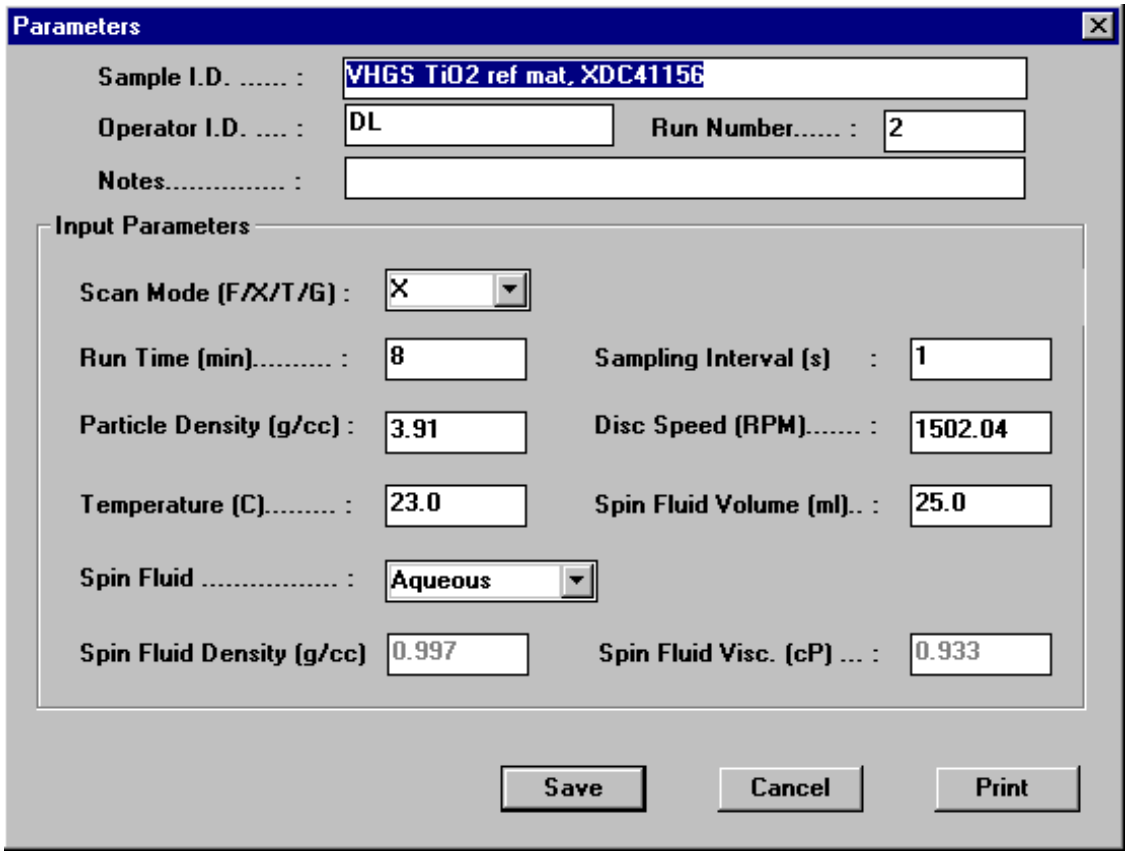


Click on **Setup, Head Scanning Parameter** to enter the delay time in seconds between the start of the run and the start of the scan. For a sample with very large particles, you may want to start the scan quickly after the measurement begins so that the large particles do not fill up the space between the disc cavity radius and the detector. For particles smaller that a micron, we recommend a scan delay time of 60 seconds. This provides a substantially flat initial function against which the lower baseline may be judged. The Setup menu allows the operator to set parameters that seldom vary and need not be reentered each time a sample is run.

Click on the **Parameters** command button in the lower part of the screen. Fill in the **Sample I.D.**, **Operator I.D.**, and **Notes** fields. The Sample I.D. is used in the Database section of the program for naming the files, Therefore, care should be taken in organizing files by the use of appropriate Sample I.D.'s. Use the **Run Number** to distinguish between measurements made on the same sample.

The **Scan Mode** determines the type of measurement being made: F, disc spinning with detector stationary; X and T, disc spinning with detector scanning up to 1 mm below the liquid surface. The only difference between these two modes is the way in which the raw data points are sampled for data reduction and analysis. In the T mode the raw data are sampled at equal intervals in time, while in the X mode the raw data are sampled at equal intervals in diameter space. In the G mode the disc is stationary and the detector is scanning. This mode is used with gravitational sedimentation.

The **Sampling Interval** determines the rate at which data points are collected. Usually the data are collected every 1-second. The program automatically calculates the appropriate interval to use based on the total run time. The **Disc Speed** determines the size range covered in a measurement. Enter the **Temperature** of the liquid. Several pure liquids may be selected in the list box. The refractive index and the viscosity are calculated automatically for the given temperature for these liquids. Select one of these liquids or select **Unspecified**. Then enter the name of the liquid and fill in the **Spin Fluid Density** and **Viscosity** values at the specified temperature.



Click on the **Save** button to save the changes and exit the Parameters Window. NOTE: If you click on **Cancel,** none of the values entered will be saved. Previously entered values will be used.

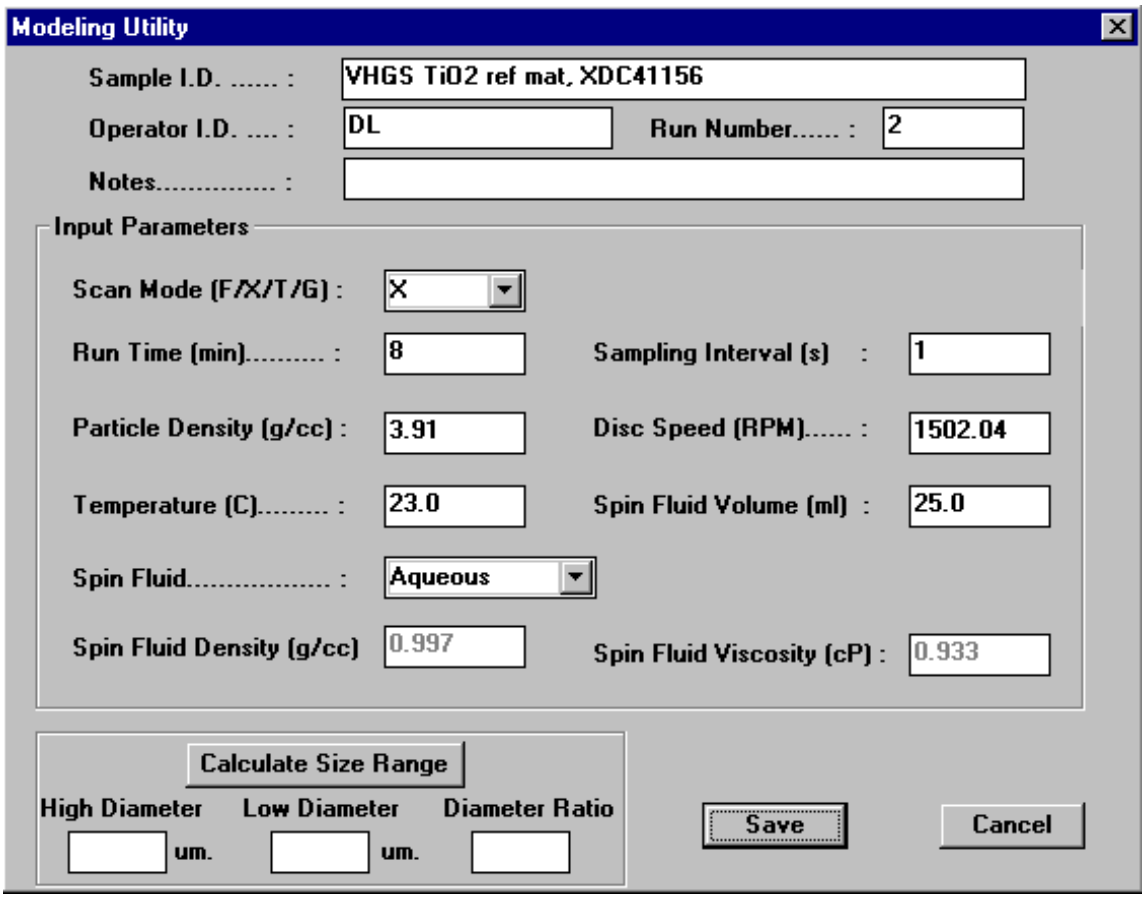
Click on the **Modeling Utility** command button to determine the optimal run conditions for each sample. The information displayed on this page is the same as that entered in the Parameter Window.

Click on the **Calculate Size Range** button to display the size range covered for the specified conditions. The size range is displayed in the lower box. Optimize the run conditions using the following guidelines:

Choosing the Scan Mode

Select the appropriate scan mode. If large particles (greater than about 1 μm) are present in the suspension then use the gravitional (G) scan mode. Otherwise use the centrifugation (X/T) modes. If the size range covered is very broad, then you might consider making two measurements and then merging the results of the two runs. Consider this: If the sample ranges from, say, 20 μm down to 0.2 μm, you can run it in the gravitational mode (say 30 μm down to 2 μm) and in the centrifugation mode (3 μm down to 0.2 μm) and then merge the two data sets. Note that the two ranges should

actually overlap for successful merging to take place. You can also merge results from two centrifugal runs (T+T, X+X, etc.), if necessary. Use the same volume if you have to make two separate runs on the same sample.



Setting the Run Time

Use a minimum run time of 3 minutes. Note that when the detector is scanning, a shorter run time will increase the scanning speed, resulting in some loss of resolution. There is practically no loss of resolution with the scanning speeds involved with a minimum run time of 6-8 minutes. Increasing the run time will extend the low end of the size range measured. However if the run time for a given sample exceeds 30 - 40 minutes, you should consider making two separate runs and combining the results. For example you could make two centrifugal mode runs (X/T + X/T) at different speeds, etc.

Choosing the Sample Volume

In the centrifugation mode of measurement, the typical sample volume used should be 15 to 25 ml. Notice that as you increase the volume, the Low Diameter is scarcely affected, while the High Diameter increases to a much greater extent.

For measurements in the gravitational mode a sample volume of 13 to 15 ml will optimize the size range covered for a given run time. Do not use a sample volume less than 13 ml, as this will affect the accuracy of your results. As you increase the volume in this mode, both the Low Diameter and the High Diameter increase significantly.

Selecting a Spin Fluid

Please see the cover page of this manual for restrictions on fluids that may be used with the standard disc.

If the desired High Diameter cannot be achieved in a given mode, and you wish to analyze the sample in a single run in that mode, consider using a different Spin Fluid for the run. Using a more viscous/dense liquid will slow the particles down as they sediment out of the suspension; thus increasing the High Diameter measured in a run. For example, you can add sucrose or ethylene glycol to water to increase the viscosity of the spin fluid. However, you must then change the Spin Fluid name to anything other than WATER. This will give you access to the Spin Fluid Density and Viscosity entries in this menu. Change these entries to reflect the actual values of density and viscosity at the temperature of the suspension. Tables of density and viscosity are readily available.

Whatever spin fluid is used it must be chemically and physically compatible to the extent that it does not cause aggregation or partially dissolve the particles. It must be reasonably easy to inject, and it must be easily removable from the disc when cleaning.

Avoid running samples below 750 rpm.

Once you have found a satisfactory set of conditions, click on **Save** to save this information for the next run.

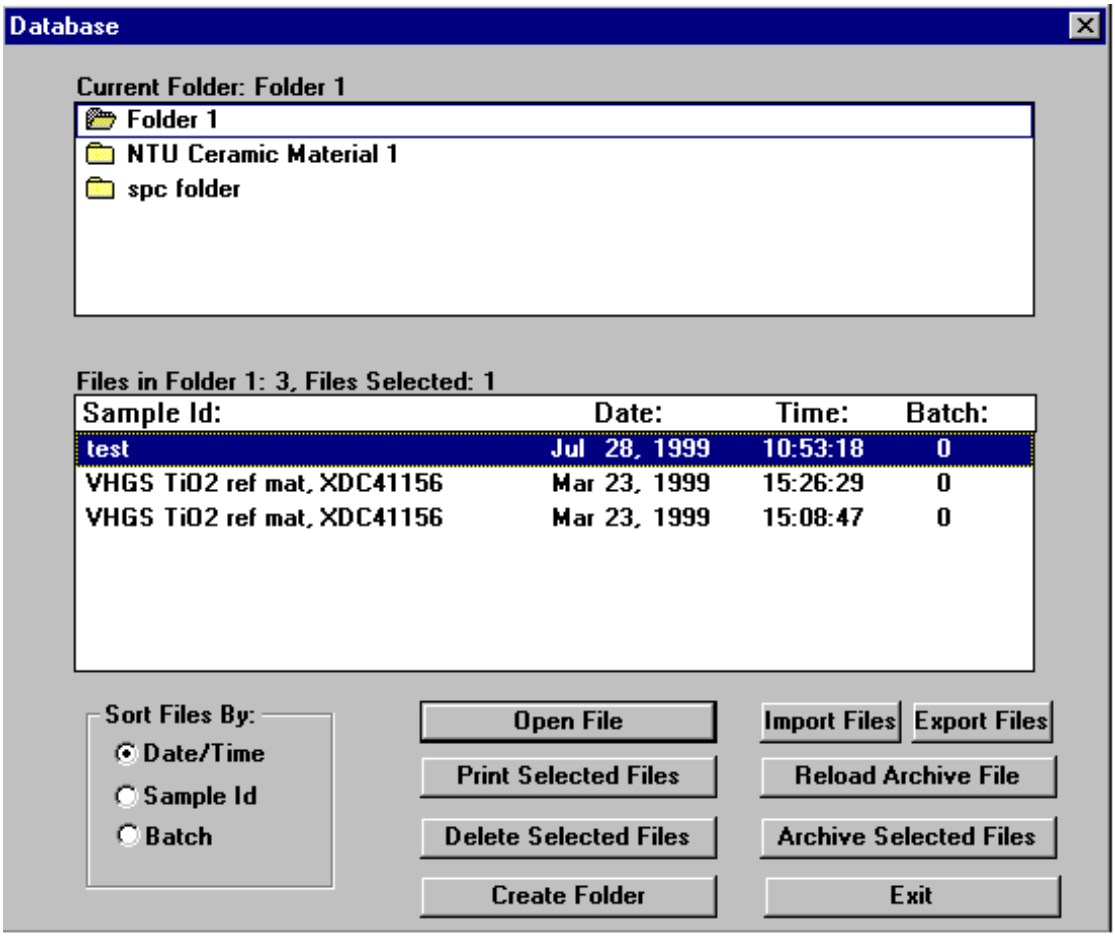
In the Menubar of the Main Window, click on **File** and then **Database** to open a demo file. Results are saved in the currently opened **Folders** as **Files** using the **Sample I. D., Date,** and **Time** as the file identifiers. Files are saved in folders using the **Save** or **Save As** under the **File** menu.

To create folders, click on **File/Database**. Click on **Create Folder**. Enter a folder name; it can be more than 8 characters; it may include spaces and punctuation. Double-click on the Folder to open it. The file folder icon *opens* when the folder is active. Single clicking on a folder selects the entire folder but does not open it. When selected, but not opened, the entire folder and its contents may be deleted, printed, or archived by clicking on **Delete Selected Folder**, **Print Selected Folder**, or **Archive Selected Folder**.

Select a single file by clicking on it. Select a string of consecutive files by clicking on the first, then, while holding down the Shift key, click on the last. Select a string of

nonconsecutive files by clicking on the first, then, while holding down the Control key, click on any number of files, consecutive or not, one at a time. When selected, a single file or multiple files may be deleted, printed, or archived by clicking on **Delete Selected File(s)**, **Print Selected File(s)**, or **Archive Selected File(s)**.

Files are **archived** in a binary format in the drive\directory path c:\bicw\xdcw\data using the extension .bak by default. You may change the drive\directory, but you will then have to remember it. If you do not change the filename, the default filename, archive.bak, is used. You can archive an entire folder by selecting it, but not opening it. You can archive single files or groups of files. When archived to an existing .bak file, the selected folder or file(s) are added to it.



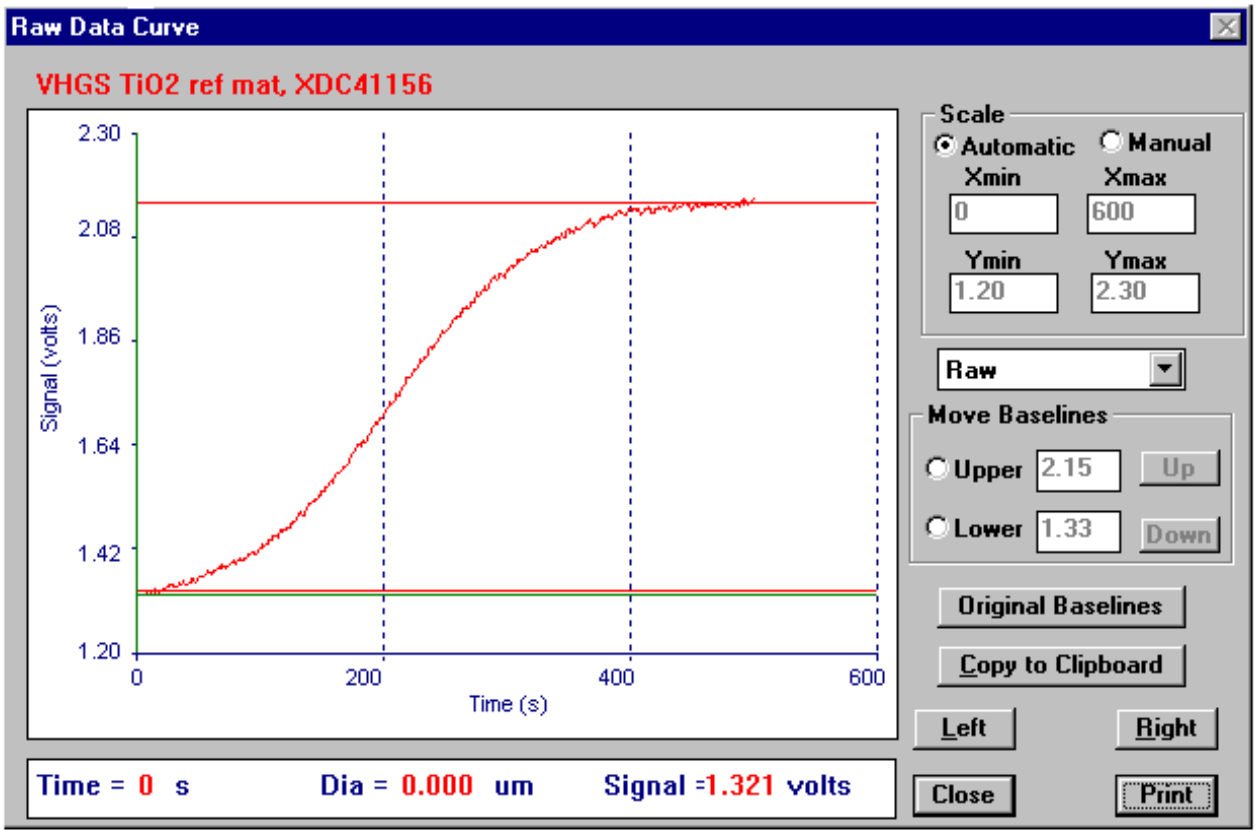
Click **Reload Archive File** when you want to reload an archived file. Click on the drive and directory where it was stored. The default path, mentioned above, is c:\bicw\xdcw\data. However, if you archived the folder\file(s) in another path, you must select that drive\directory and the correct filename. Files are reloaded and merged into the currently opened folder.

Double clicking on a file opens it. Alternatively, select the file by single clicking on it and then clicking on **Open File**.

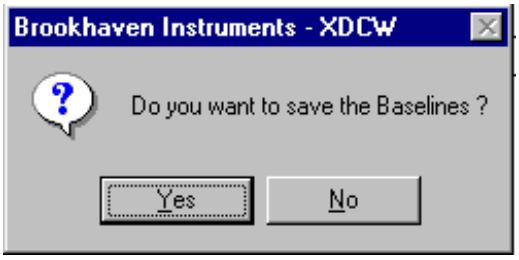
You can create ASCII files suitable for use with spreadsheets and plotting programs. Select a file, then click on **Export Selected File**. The format of exported files is given in Appendix V.

Click on **Reanalyze**, to get the Parameters page. You cannot change the values of Disc Speed, Sampling Interval, Run Time, and the Spin Fluid Volume which are grayed out. The rest of the parameters can be changed to reanalyze the data. When you click on **Save** the change is saved and the raw data curve with the baselines is displayed in a zoom window.

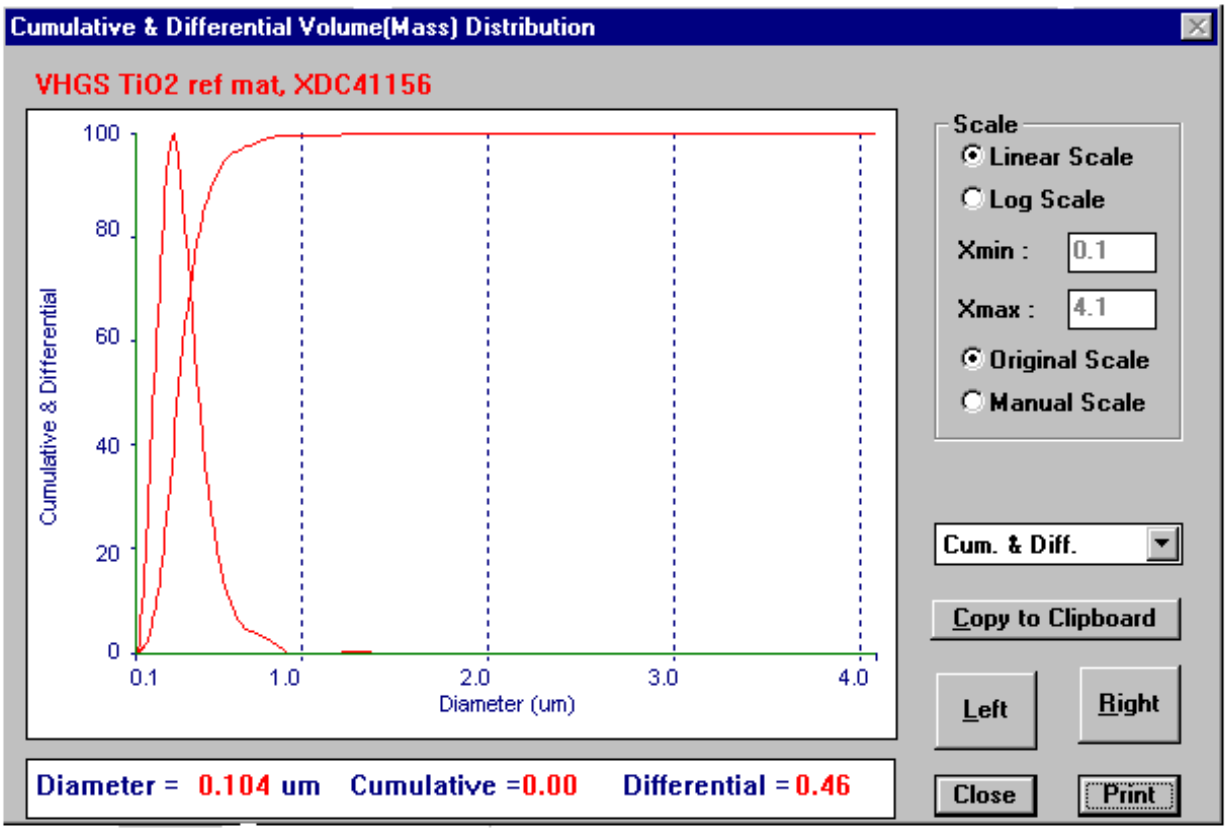
In the bottom of the Zoom Window the time, diameter, and signal value for each raw data point are displayed. The graph scales can be changed by clicking on the **Manual** button and entering the desired X and Y ranges. Then click anywhere on the graph to see the changes. The cursor can be moved left and right either by clicking the **Left** and **Right** buttons or by positioning the cursor at the desired point and clicking the mouse.



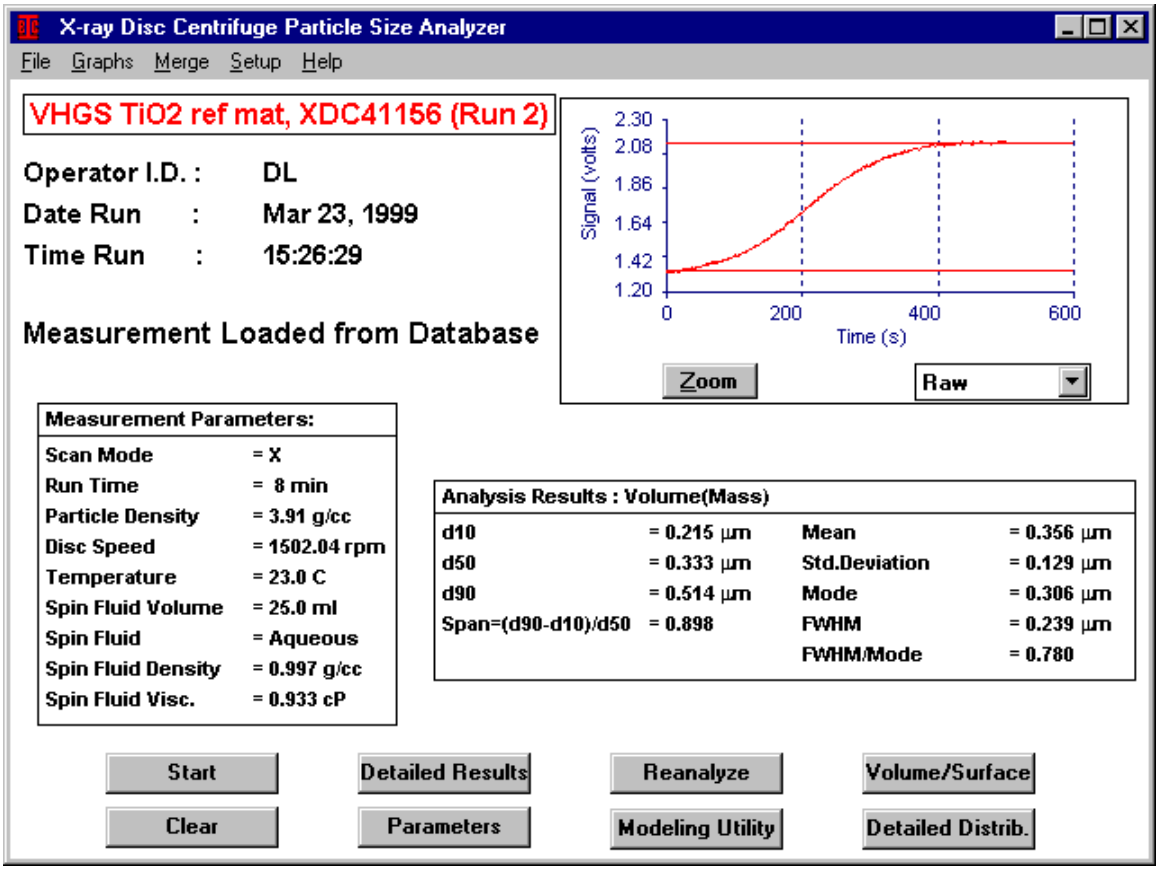
To change the baseline positions, click on **Upper** or **Lower**, and then click on **Up** or **Down**. If the user doesn't like the changed baselines and wants to revert back to the original baselines then he can click the **Original Baselines** button. Once the graph scales and the baselines are set by the user, click on the **Close** button to exit the window. If the settings of the graph are changed in the Zoom Window then the same settings are displayed in the Main Window. If the baselines are changed then the user has to choose to save it explicitly and the following screen appears. When the user clicks **Yes** then the results are recalculated automatically using the changed parameters and new baselines are saved and displayed on the screen.



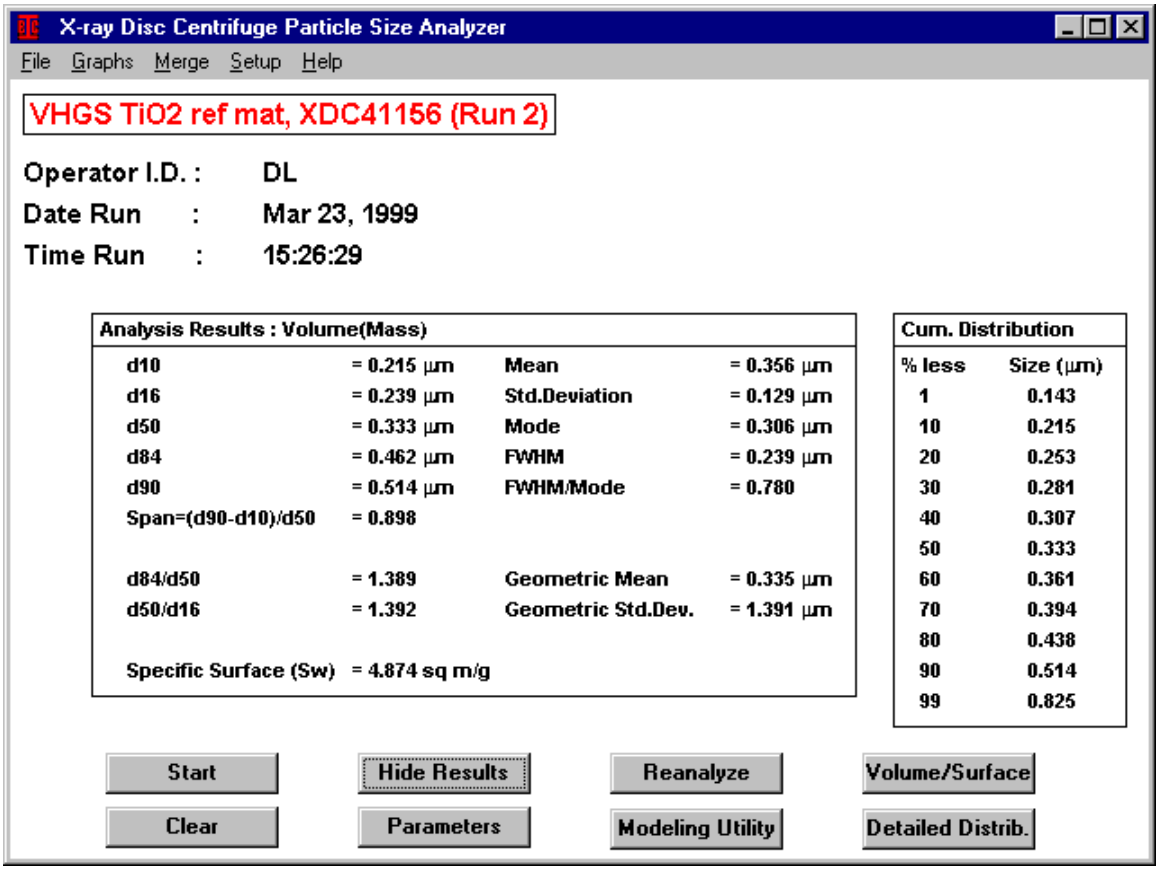
Cumulative and differential curves may be displayed in the Zoom window by selecting them from the pull-down list-box. The diameter, differential, and cumulative values are shown in the bottom of the window. Click on any point in the graph to get the corresponding values, or click the **Left** or the **Right** buttons. The distribution can be viewed in the log or normal scale. The X and Y-axes can be re-scaled. To print the Zoom Window, click on **Print**. Click on **Copy to Clipboard** to copy the image to the clipboard that can later be pasted in any document.



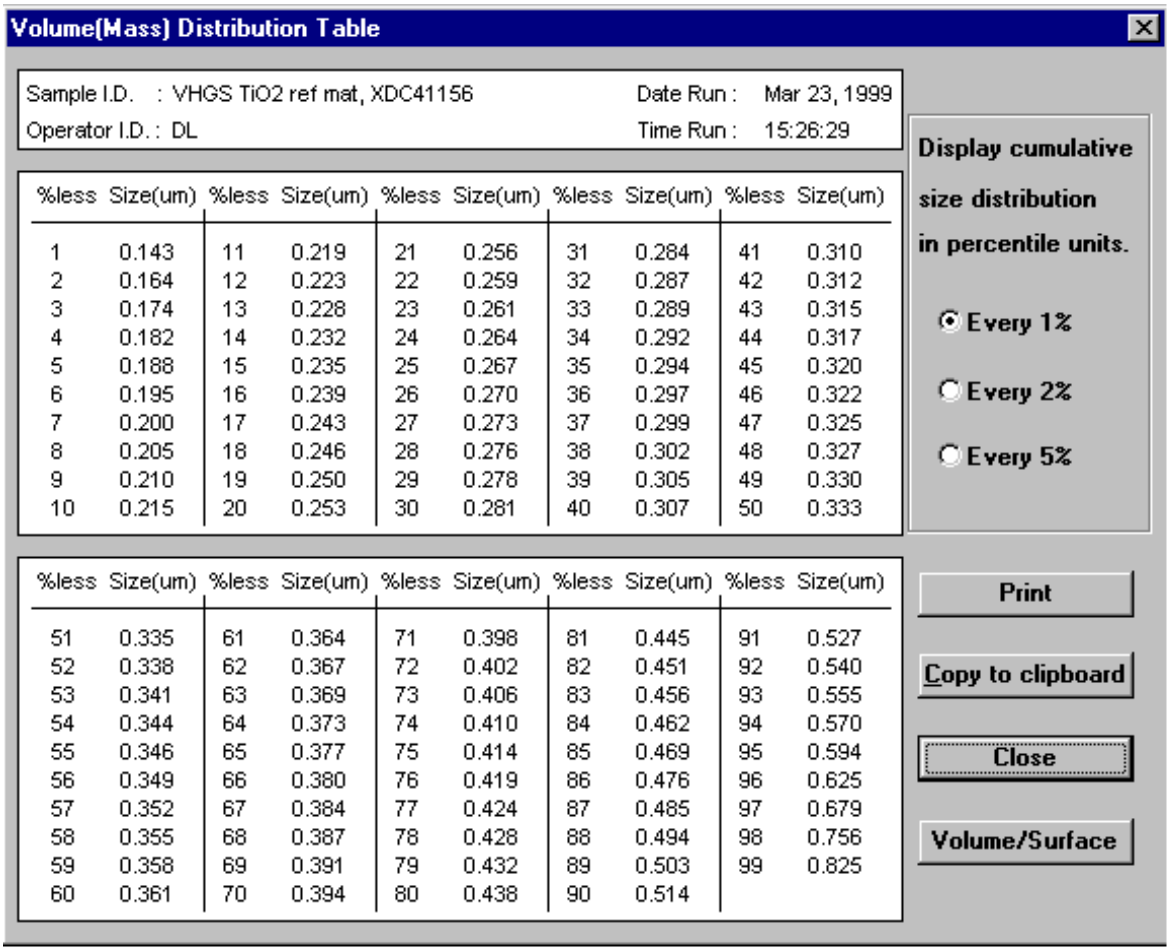
On the Main Window a summary of the results are displayed on the right hand side, just below the graph. The calculated, volume-weighted results are displayed. (If the particle density is independent of size, this is also equal to the mass-weighted results.) Click on the **Volume/Surface** command button to change this to the surface area-weighted distribution, click again on the **Surface/Number** button to get the number-weighted distribution data. Clicking it once more will give you the volume-weighted distribution. Note that the Analysis Results heading changes as you click on this command button. So one can toggle between all the distributions by clicking this button.



Click on the **Detailed Results** command button to see more calculated results. You can click on the **Volume/Surface** command button to view results weighted by surface area. Click again to see results weighted by number. The detailed result view shows the size distribution statistics on the left side of the screen. On the right side is a short table of the cumulative distribution. Click on **Hide Results** to get the Main Window back again. See Appendix VI for the definition of the various distribution statistics.



Click on the **Detailed Distribution** command button to view the cumulative size distribution table. To display values every 1%, 2%, or 5%, click on the corresponding radio button. By clicking the **Volume/Surface** button one can toggle between data weighted by volume, surface area and number. Click **Close** to exit the window and to return to the Main Window.



%less	Size(um)	%less	Size(um)	%less	Size(um)	%less	Size(um)	%less	Size(um)
1	0.143	11	0.219	21	0.256	31	0.284	41	0.310
2	0.164	12	0.223	22	0.259	32	0.287	42	0.312
3	0.174	13	0.228	23	0.261	33	0.289	43	0.315
4	0.182	14	0.232	24	0.264	34	0.292	44	0.317
5	0.188	15	0.235	25	0.267	35	0.294	45	0.320
6	0.195	16	0.239	26	0.270	36	0.297	46	0.322
7	0.200	17	0.243	27	0.273	37	0.299	47	0.325
8	0.205	18	0.246	28	0.276	38	0.302	48	0.327
9	0.210	19	0.250	29	0.278	39	0.305	49	0.330
10	0.215	20	0.253	30	0.281	40	0.307	50	0.333

%less	Size(um)	%less	Size(um)	%less	Size(um)	%less	Size(um)	%less	Size(um)
51	0.335	61	0.364	71	0.398	81	0.445	91	0.527
52	0.338	62	0.367	72	0.402	82	0.451	92	0.540
53	0.341	63	0.369	73	0.406	83	0.456	93	0.555
54	0.344	64	0.373	74	0.410	84	0.462	94	0.570
55	0.346	65	0.377	75	0.414	85	0.469	95	0.594
56	0.349	66	0.380	76	0.419	86	0.476	96	0.625
57	0.352	67	0.384	77	0.424	87	0.485	97	0.679
58	0.355	68	0.387	78	0.428	88	0.494	98	0.756
59	0.358	69	0.391	79	0.432	89	0.503	99	0.825
60	0.361	70	0.394	80	0.438	90	0.514		

Once back in the Main Window, click on the **Clear** command button to clear the data in memory and to input new parameters for a new experiment.

The next section describes the details of sample preparation, making a measurement, saving the data, printing reports, comparing data, merging data and viewing the statistical process control chart.

Section V: How to Make a Measurement

The procedures described below contain some general information useful for every type of analysis. These procedures are not all embracing. They should be considered as guidelines. The actual procedures used will vary depending upon a number of factors. Among these are composition and type of suspending medium, amount of spin fluid, etc. Make your initial measurements, if possible, on relatively narrow size distributions that are well characterized. Do not make the common error of practicing with broad unknown size distributions.

Repeatability should be your first goal. And repeatability should be on the order of 1% or 2% with the BI-XDC. Use the modeling program to guide you in deciding run conditions for your samples. You will need the following items: syringes of suitable size (two 20 - 30 ml) with approximately 15 cm of flexible rubber tubing attached; small dilution bottles or vials with caps (25 to 50 ml); a plastic plug for the disc opening to prevent sample spillover; and a siphon to clean the disc cavity after analysis. For siphoning, we recommend a 50 ml, syringe with attached rubber tubing or a plastic wash bottle with attached rubber tubing.

RUNNING A SAMPLE -- A Quick Overview

NOTE: It is recommended that the instrument be switched on 20 minutes prior to use in order to obtain stable operating conditions. Inject 10 ml of water into the disc, before turning on the X-ray source.

For the novice there are many things to do the first time you run the BI-XDC. Once you grasp the procedures, they are quite simple and will become, therefore, obvious. To assist in learning these procedures, they are presented here as an overview and in more detail below.

1. Use the Modeling Utility to estimate the optimum experimental conditions and save these conditions for the next run.
2. Prepare the sample and power on the analyzer.
3. Turn the X-ray key to the on position.
4. Click the START command button in the Main Window and follow the subsequent instructions on the screen to measure or load the baselines, inject a sample, and start the run.
5. Watch the sample for mixing/turbulence and monitor for a real time plot of the raw data.
6. The run will automatically stop on completion. If the upper baseline has not been reached, increase the run time before the run is completed.
7. Remove the sample and clean the disc.
8. Move the baselines if necessary and reanalyze the data.

9. Combine two separate analyses if necessary (Merging).
10. View and print appropriate results.
11. View various graphs, compare results and build the SPC chart if desired.

Modeling Utility

From the Main Window, click on the **Modeling Utility** button. Enter the sample information, the details of which have been described in the previous chapter. Click on the **Calculate Size Range** button. The size range is displayed in the lower part of the screen. Select the run conditions (refer to the previous chapter) and save the data for the next run. [Note: Run conditions are a compromise between minimizing total run time and maximizing resolution of the measured size distribution.]

Sample Preparation

For inorganic samples with a high atomic mass (e.g. Titanium Dioxide, Iron Oxide, Barium Hexaferrite, etc.), a volume concentration of 0.2 % gives adequate attenuation but this needs to be increased with powders having a lower atomic mass and a volume concentration as high as 1 % - 2 % is necessary with quartz. In general however, use a concentration that yields attenuation in the detector signal of 25 % - 40 % with respect to the signal seen with the pure Spin Fluid in the disc. If the concentration is too low, the resulting signal-to-noise ratio is low, and the repeatability will be poor (random error). If the concentration is too high, hindered settling may occur, and the results will be wrong (systematic error). The difference between the upper and lower baselines should be at least 0.3 volts.

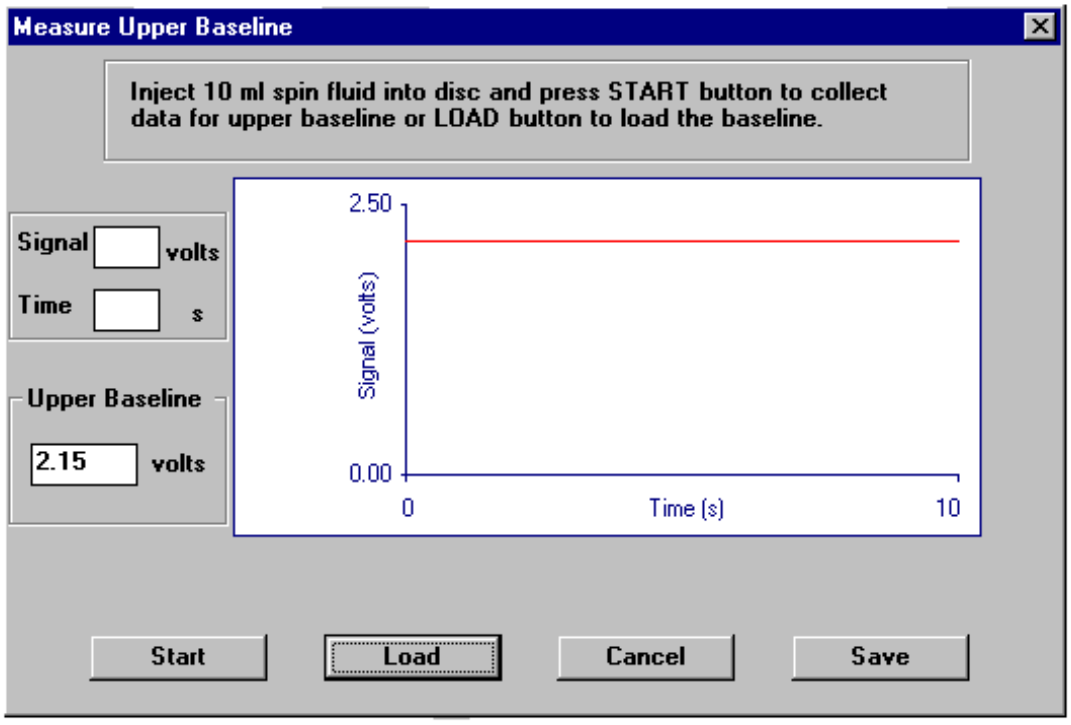
Proper sample preparation involves wetting the entire surface of the particle, breaking apart unwanted aggregates, and forming a dispersion that is stable for a time long enough to make a good measurement.

Wetting and spreading are usually accomplished by a wetting agent, sometimes in combination with a small amount of a liquid with a smaller surface tension than the final suspension liquid. Lists of wetting agents are available. Wetting agents are surfactants, and, in too high a concentration they form micellar particles. Although these particles are small and, as a percent of the total sample weight, usually insignificant, their presence can cause particles to aggregate. Therefore use no more than 0.02 % to 0.04 % by volume of the wetting agent.

Normally energy is applied to the sample to break up aggregates. Sometimes this energy is very little: Gentle swirling and shaking. And sometimes the energy is a lot: Ultrasonic probe. The energy should be enough for the aggregates to break apart, but not so much as to increase particle-to-particle collisions that promote aggregation. Short bursts of energy from an ultrasonic bath or probe are best. A good dispersion does not settle, aggregate, or otherwise change noticeably during the measurement time.

Running the Sample

Click on the **Start** command button and follow the instructions on the subsequent windows to make a measurement. There are, however, some shortcuts that you can take as you gain experience with this type of measurement. Note that the XDC analyzer and the X-ray source must be powered on and the communications cable must be connected to the correct comport before you start the measurement. The software gives an error message if this is not the case. Once you click the **Start** button, it switches to **Stop** and will switch back to **Start** when the measurement is completed. The first window lets the user either measure or load the upper baseline. To measure the upper-baseline, inject 10 ml of spin fluid into the disc, and then click on the **Start** button. The signal input is displayed on the graph as the baseline is measured. After 10 seconds, the measurement is



completed and the average measured baseline value is displayed in the left side of the screen. This value can be edited if needed. Click on **Save** to save the baseline and close the window.

If the spin fluid used is the same throughout the day, then you do not have to measure the (upper) baseline with spin fluid each time you run a sample. Measure this baseline when you run the first sample and save it. Then for subsequent runs, do not inject the spin fluid at all. Instead simply click the **Load** button to load the baseline value that you have saved earlier. When the load button is clicked the baseline saved in the previous run is loaded and displayed on the screen. Again you can edit the value of the baseline if needed and save it to close the window. (If you reload the upper baseline, it is assumed that the disc walls have not been coated with particles from run-to-run. Clean the disc after each run to ensure the assumption is valid.)

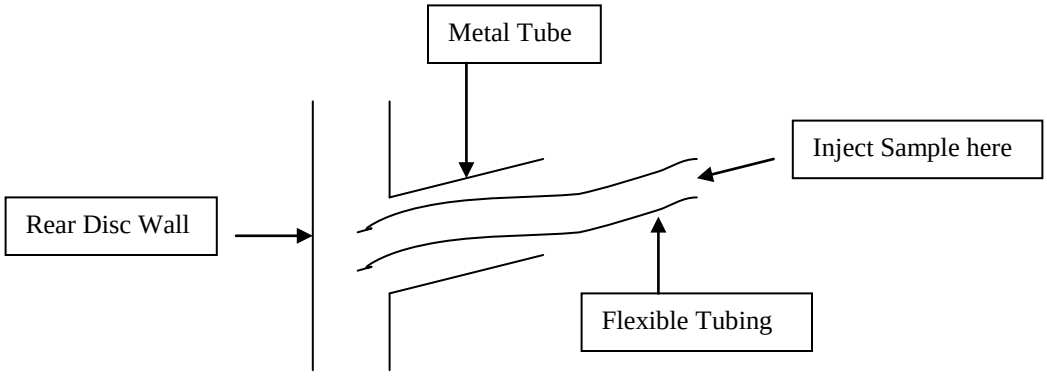
The next screen instructs the user to remove the spin fluid, dry the disc and then click the **OK** button to continue the measurement. Again if the baseline is loaded, simply click on the **OK** button.

The subsequent window allows the user to measure the lower baseline. You must always measure the lower baseline, since it depends on the sample and the concentration of the suspension, and will be different for different runs. Inject the volume of the sample that will be used to make the run into the disc. This is the volume entered in the sample information menu.

Draw 10-15 ml of suspension into a syringe with a short piece of flexible tubing. Inject it back into the sample bottle, keeping the end of the tubing submerged. Repeat this procedure – draw and re-inject – two or three times to ensure a representative sample in the syringe. Finally, draw a few milliliters more than will be required for the measurement. For example, if the desired sample volume is 20 ml, draw up roughly 25 ml of suspension into the syringe.

Invert the syringe and draw in some air so that the flexible tubing is devoid of all liquids. Tap the syringe to expel air bubbles. Now bend the flexible tubing around until it is over the sample bottle and press the plunger while keeping the syringe vertical until all the air is expelled from the syringe and the tubing. Now depress the plunger further to adjust the volume to the desired mark, in this case 20 ml. Inject the sample into the analyzer following the recommendations below.

The door of the analyzer need not be opened to inject the sample into the disc. Insert the flexible tubing through the metal tube in the door. This acts as a guide and bends the flexible tubing down at the end so that the suspension enters the disc without splashing. Cut the flexible tubing so that its tip is close to the rear wall of the disc when the tubing is fully inserted. See the figure below.

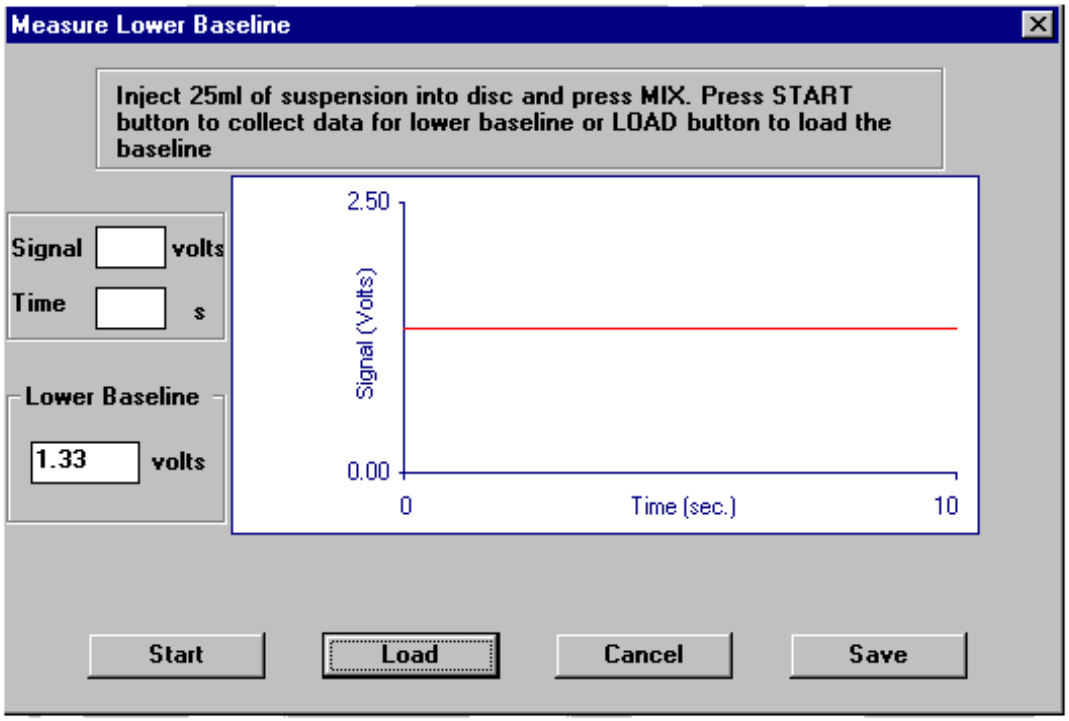


The liquid in the flexible tubing does not change the volume injected as it remains in the tubing after the sample is injected into the disc. Difficult as it may sound, this procedure is actually quite simple and easy to master with very little practice.

With this method of sample injection repeatability is better than 1 %. If you desire greater accuracy in the sample volume use an automatic pipette or similar equipment to dispense the sample. In this case, the door of the analyzer will have to be opened to inject the sample. Keep the door open for only as long as it takes to inject the sample, as the X-ray source is turned off as soon as the door is opened. The shorter the duration for which the X-ray source is turned off the better its stability.

After injecting the sample insert the plastic plug into the center of the disc to prevent loss of liquid due to splashing. The greater the injected volume and the stronger the mixing (described below), the more likely liquid will be lost.

Press the MIX button on the analyzer (in some units this button is labeled RPM/M). This will make the disc rock back and forth, ensuring that the suspension is well mixed prior to measurement. The mixing mode can be varied from weak to strong mixing using numbers 1,2,..,9 (strong). Five is the default. Use a higher value for larger and denser particles to ensure a homogeneous suspension.



Click on the **Start** button to measure the lower baseline and then the **Save** button on the screen to save the value and close the window. DO NOT REMOVE THE SUSPENSION FROM THE DISC. The next window instructs the user to press the MIX button on the analyzer to stop the disc. As soon as the disc stops turning, press the START button on the XDC front panel or click the **OK** button on this window to start the run. Now the Main Window is displayed showing the baselines and a real-time graph in the top right corner. The signal input, Stokes diameter, and the total run time are displayed below the graph.

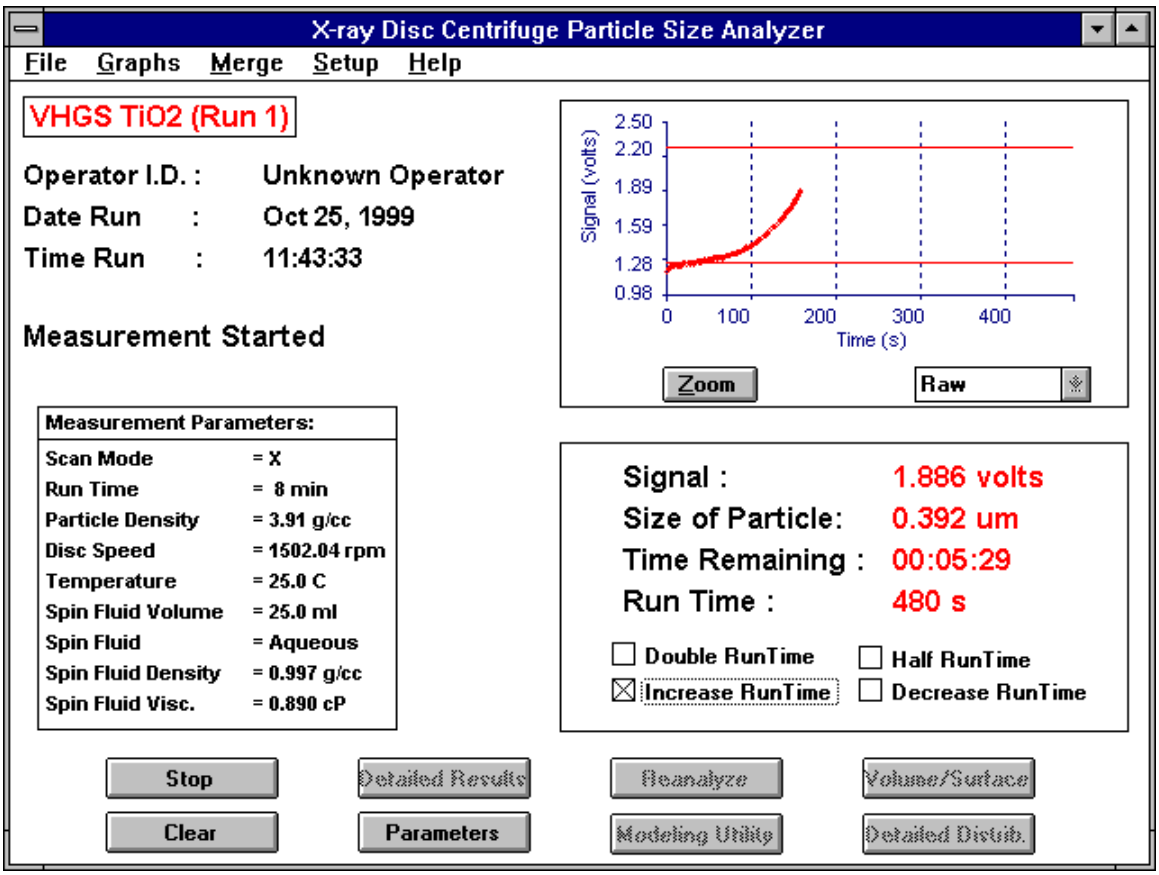
Monitoring the Run

Observe the sample in the disc. Use the built-in strobe to freeze the motion of the disc. You should be able to observe a clearing of the liquid near the surface. While the sample is running it should be observed, periodically, for any unusual behavior, such as turbulence or mixing. This can be seen as a swirling motion of the liquid when the strobe is on.

Observe the monitor to ensure that the data collected is a smooth curve (taking the inherent noise in the detector signal into account).

Stopping the Run

The duration of the run is the same as the Run Time entered in the sample information menu. The run will automatically stop when this time has elapsed and the data are saved. This is the normal completion mode.



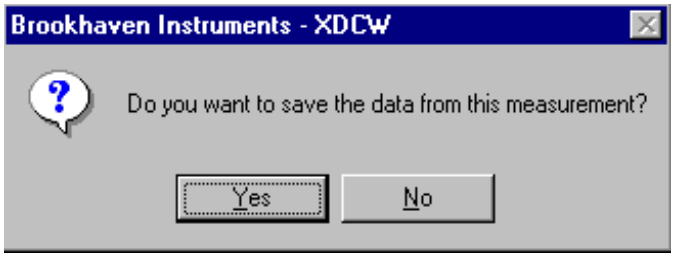
The run can be stopped earlier by clicking the **STOP** button, and the user is offered the opportunity to save the data. This is usually indicated when the upper baseline

is reached much before the normal completion of the run. However, wait for ~30 seconds after the raw data first reaches the upper baseline before terminating the run.

The run can be aborted at any time by pressing ESC or CANCEL. All the data collected will be discarded in this case.

If the upper baseline has not been reached and you wish to extend the experiment duration, click the **Increase Run Time** button to increase the time in steps of 60 seconds. This can be done in any mode of measurement.

Once the run is completed the window below is shown and the user is explicitly asked to save the run. If for any reason the user doesn't want to save the measurement then the data is lost.



The disc will continue to spin until the MOTOR button on the analyzer is pressed. That is, data collection, and disc rotation are separate functions.

Cleaning the Disc

If the suspension tends to spill out when the disc is stopped, use the HEAD button on the analyzer to retract the head safely out of the way, before pressing the MOTOR button to stop the disc. If any spillage has occurred, open the door once the disc has stopped moving, and then wipe up any excess liquid, or consider using the plastic plug before the measurement has begun. Then close the door and press the HEAD button to restore the head in front of the disc.

Use the large syringe or other siphoning arrangement, with flexible tubing attached, to withdraw the suspension from the disc. The tubing should be long enough to reach down to the bottom of the disc cavity.

Now inject 10 – 15 ml of water or a dilute soap solution into the disc cavity. Press the MIX button on the analyzer (in some systems this button is labeled RPM/M). The disc will start rocking gently, introducing a swirling motion into the liquid. This should remove residual particles. Remove the liquid and repeat this rinsing procedure until the disc cavity is clean. The final rinse should be with deionized water. Dry the disc cavity

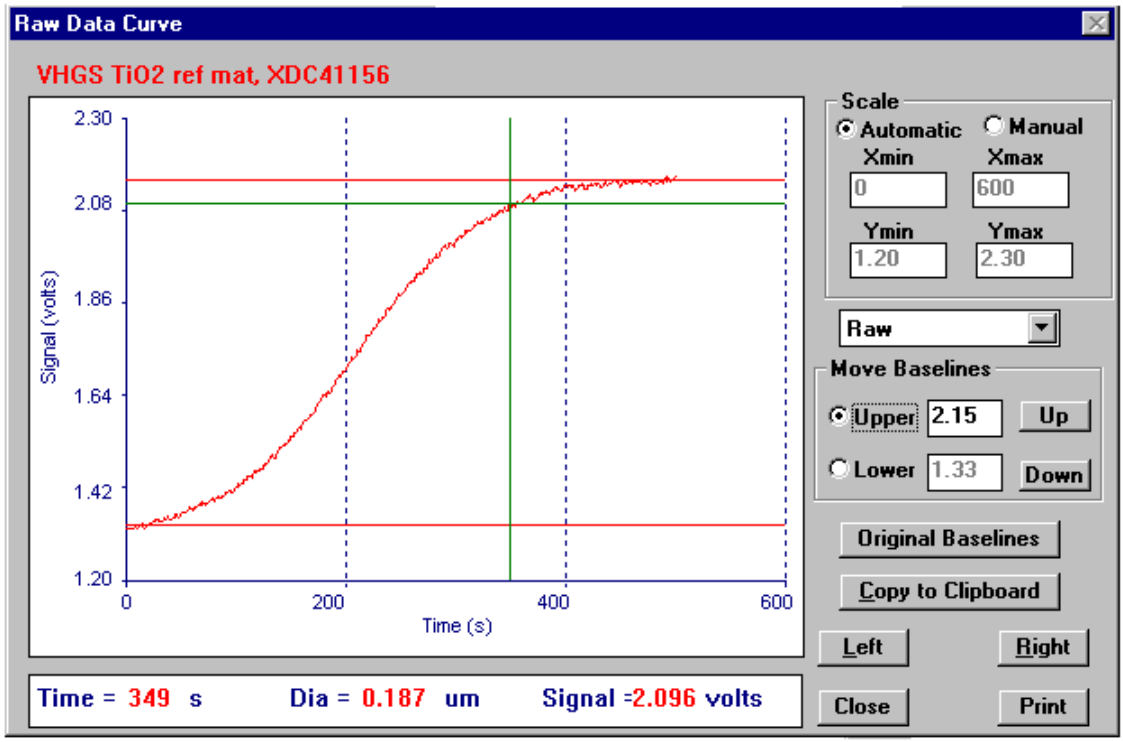
with a paper towel folded such that it can be pushed into the cavity and ride along its inner perimeter. The disc is made of a relatively hard plastic, but it can be scratched. So clean it thoroughly and carefully. NEVER LET A SAMPLE DRY IN THE CAVITY.

Different samples tend to coat the disc walls differently. Some are easier to remove than others are. You may need to modify the above procedure to clean the disc.

If you do not properly maintain the cleanliness of the disc, your results will suffer over time.

Setting the Baselines and Reanalyzing the Data

After the measurement is completed and the data is saved, the raw data curve is displayed along with the baselines in the zoom mode. You can reset the baselines graphically or numerically if necessary. The baselines can be moved up or down by clicking the **UP** and **DOWN** buttons. Details have been explained in the previous section. The following points are very important in placing the baselines correctly.



Lower Baseline

If the first few points of the raw data are well above the lower baseline, this indicates the presence of particles larger than the upper limit of measurement for this run.

These particles have settled out of the suspension too rapidly to be observed by the detector. You may have to run the sample again at a reduced speed or in the gravitational mode to obtain the complete size distribution. However, if all you need to know is the exact percentage of particles above a certain diameter, you may not have to repeat the run. Just complete the analysis of this run to obtain the distribution of the sample up to the upper size limit for this run. In other words you will know exactly what fraction of the particles lies above the High Diameter. NOTE: If you feel that aggregation is taking place, try using a lower concentration (reduce it in steps of 20 %), and review the procedure used for dispersing the sample.

Upper Baseline

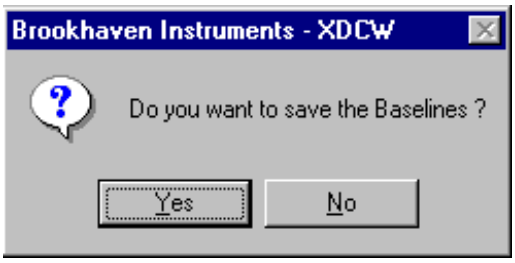
As the run progresses the signal increases and steadily approaches the upper baseline. If the run is terminated before the upper baseline is reached, you can complete the analysis with the knowledge that you will know exactly what mass fraction of the sample lies below the lower size limit for this run. However, please keep in mind that there are a number of reasons why the upper baseline may not be reached. These are as follows:

1. There are actually particles smaller than the lower size limit for the run.

2. The sample is partially soluble in the spin fluid.

3. The baseline was measured with pure water, whereas the suspension is actually made up in a solution of the dispersant. For example the upper baseline measured with a 1 % solution of sodium hexametaphosphate will differ significantly from that measured with pure water.

4. Turbulence in the suspension may cause mixing and never allow the fluid near the meniscus to clear completely.



Click on the **YES** button to save the changed baselines. The Main Window is displayed with the results calculated with the new parameters and changed baselines.

You can view or print any of the tables, screens or reports at this time. You can compare the results of this run with samples run earlier, or compare V/S/N distributions,

or build SPC chart. The Main Window Menubar under **Graphs** gives the user these options. The details of these features are described later in this section.

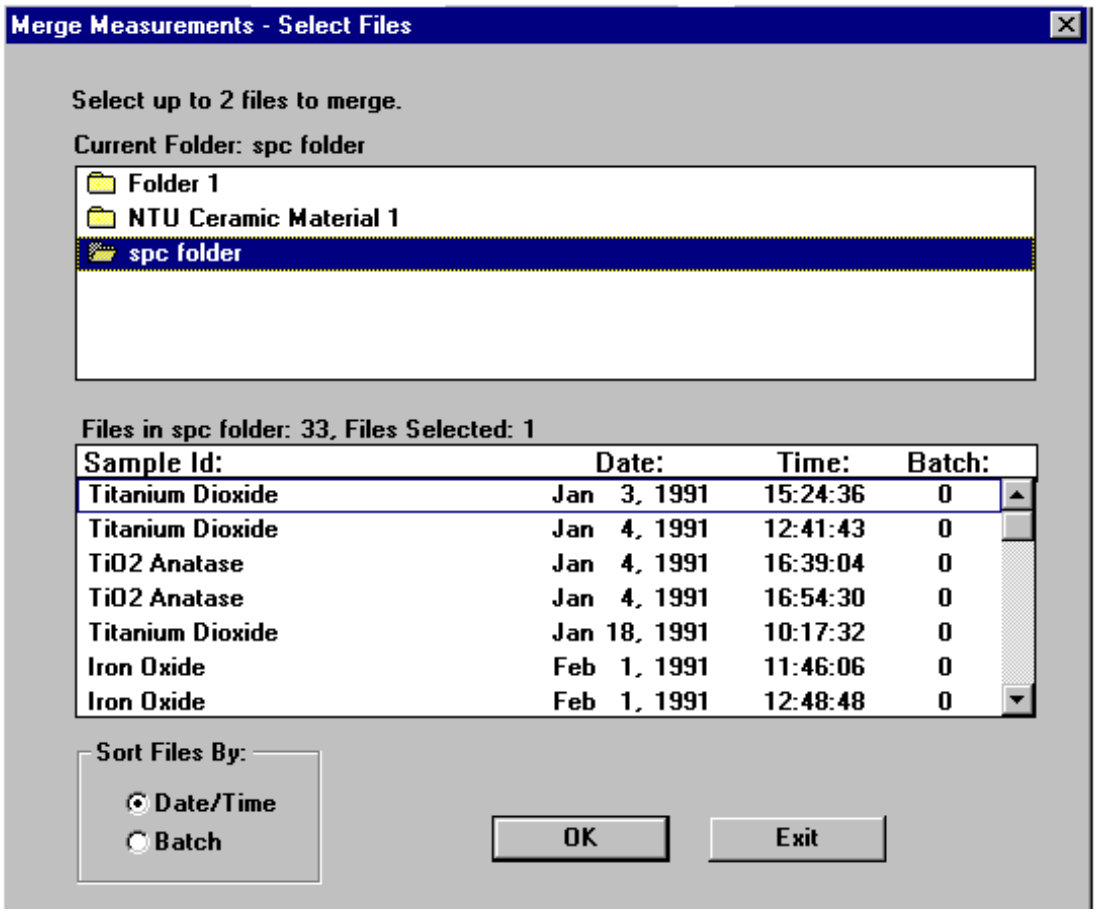
Merging of Data from Two Separate Runs

In many cases the size range of the sample may be too broad to permit an analysis with a single run in a reasonable amount of time. For example if the sample extends from 20 μm down to 0.1 μm, it will take a very long to complete the analysis. Besides, it is not recommended to use the gravitational mode for measurements roughly below 1 μm (this limit depends on sample density). The idea for merging is to obtain a larger dynamic range in the size domain than is possible with a single run, gravitational or centrifugal.

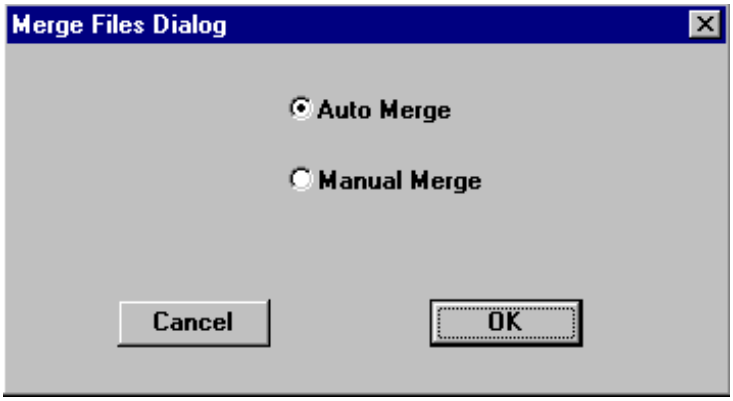
In such cases it is advisable to run the sample in two separate runs, with the size ranges covered by the two runs overlapping to enable merging. Use the Modeling Utility to determine run conditions that give a significant degree of overlap. For example you can run the above sample in the gravitational mode down to 1 μm and then in the centrifugation mode from 3 μm down to 0.1 μm. Similarly if you have a sample that covers a size range of 5 μm down to 0.05 μm, you can run it in two centrifugation mode runs at different speeds.

Maintain the same sample volume for both runs. The size distribution is, in theory invariant to the volume, but in practice does effect the results due to experimental variations. All parameters in the runs to be merged should be kept constant. Of course, the most important condition for a successful merge is to keep the upper and lower baseline constant (no upper baseline adjustment for the larger sizes, no adjustment of the lower baseline for smaller particles). You can set the baselines graphically or numerically if necessary.

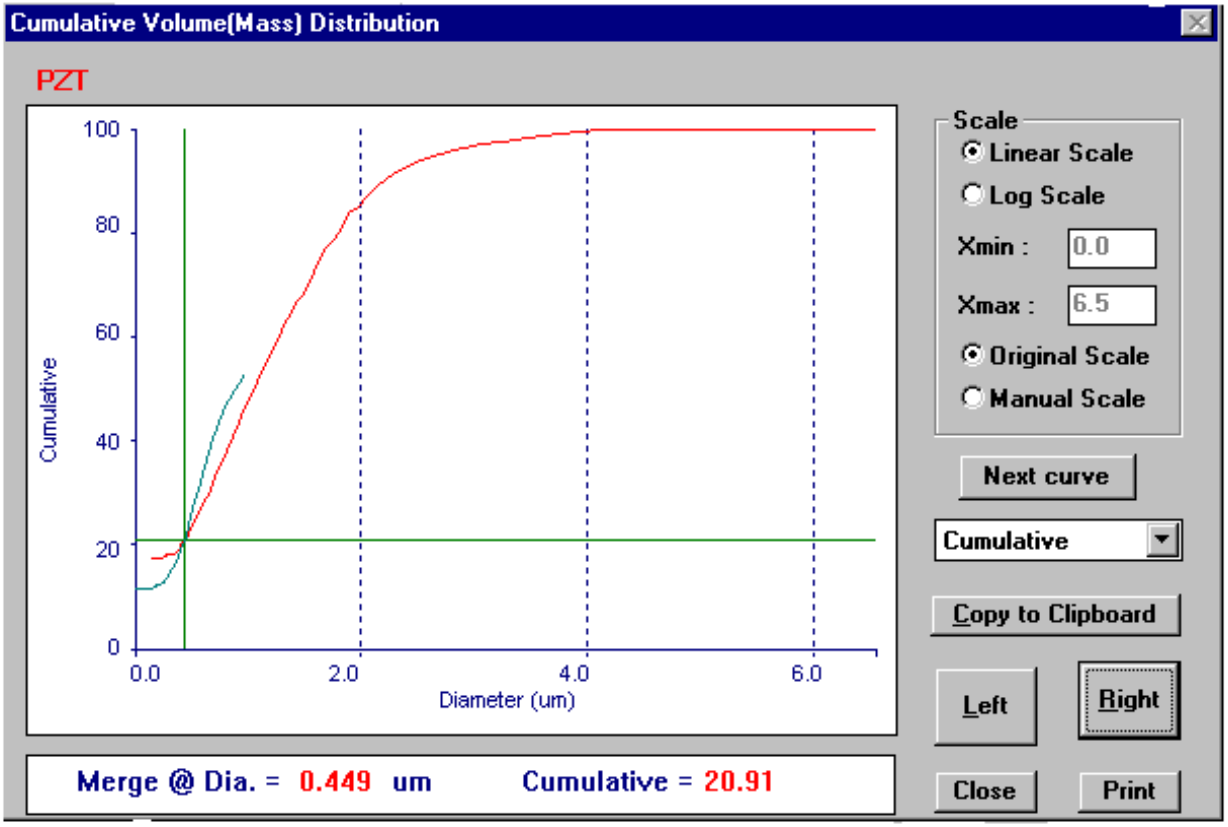
On completion of each of the two separate runs, analyze them as you would a normal run. After the second run has been saved click on the **Merge** button, in the Main Window Menubar.



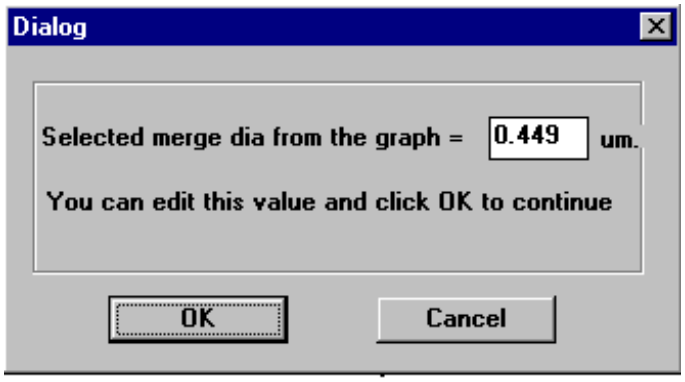
The file database is displayed and the user is asked to select the two files to merge. Hold the Ctrl key down, select two files and click the **OK** button. The following screen appears. Select Auto Merge if you want the program to calculate the merge diameter. Select manual merge to graphically see the two overlapping cumulative curves and to select merge point. Click the **OK** button to continue, or click **Cancel** to exit the merge routine.



Two intersecting cumulative curves are displayed in the manual merge mode. Scale the curve along the X-axis to show the region of overlap in greater detail. Move the vertical cursor over to the region of overlap and pick a diameter where the two curves intersect smoothly and click on the graph (the diameter at which the cursor is located is displayed in the lower left hand side box).



Now exit this window by clicking the **Close** button**.** Another window displaying the merge diameter is displayed and the user is asked to confirm merge or cancel the merging process. The merge diameter can be edited. Clicking on **OK** merges the two files and creates a merged file in the File/Folder list.



Now one can open this new file from the database. You can view it, print and plot the results, etc. as you can with a normal file, but you cannot reanalyze it with different conditions. To do this you must reanalyze the two parent files with the new conditions and merge them as explained before. Another file will be created; the earlier merged file will not be lost. You can distinguish between the two with the help of the Run Numbers on the right side of the index file.

A merged file always has a negative Run Number. The note line of the merged file contains information about the parent files and the diameter at which the two files were combined.

Sometimes the two cumulative distributions of the parent files may not intersect smoothly or may not overlap at all. For such cases consider this: The resolution of the parent distributions is greater at the low end of the size range covered. Thus the run that covers the larger sizes has better resolution in the region of overlap than the run that covers the smaller sizes. Keeping this in mind, make small adjustments to the lower baseline of the run that measures smaller sizes so that the intersection of the Cumulative Distributions is smooth and well defined.

Tips for the Experienced User

There are some shortcuts that can be taken with this type of measurement, but use these shortcuts only after you have gained some experience with this technique.

If gravitational and centrifugation runs must be combined, carry out the gravitation mode run first. Measure both the upper and lower baselines carefully (of course the upper baseline can also be loaded from memory).

At the end of the gravitation mode run press the MIX (RPM/M) button on the analyzer to re-disperse the sample.

Complete the analysis of the first run. When the raw data is displayed make a note of the upper and lower baseline values by expanding the plot along the Y-axis and moving the cursor on top of the appropriate baseline. Exit to the Main Window.

Next, visually ensure that the sample has been properly mixed and is not coating the walls of the disc. DO NOT REMOVE THE SUSPENSION FROM THE DISC.

Now click the **Start** command button to start the second run. CHANGE THE SAMPLE INFORMATION TO REFLECT THE CONDITIONS FOR THIS RUN: Scan Mode, Run Time, Sample Volume, etc. It would be good idea to append the letter for the scan mode to the Sample I.D. field for quick reference later on.

When you reach the Operating Procedures to load or measure the upper baseline, click on **Load** then on **Save** to continue.

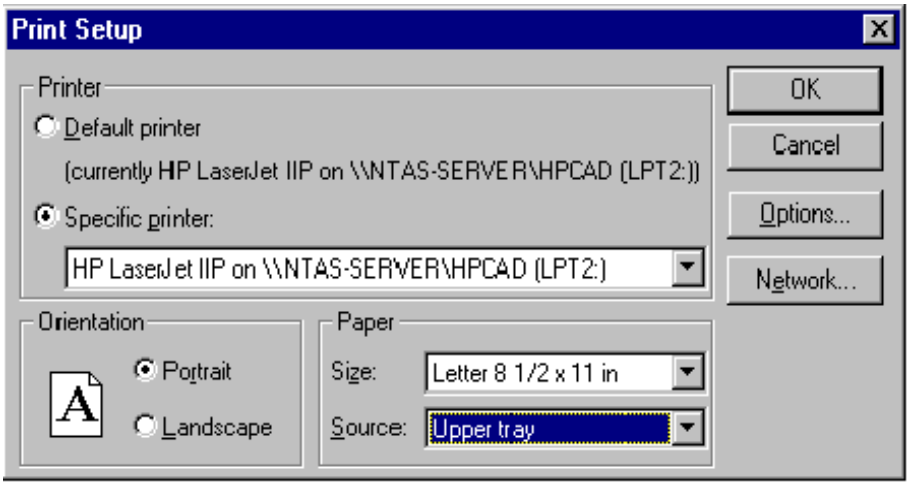
If the Sample Volume for the second run is greater than that for the first add a suitable volume of suspension so that the volume in the disc is that entered in the sample information page. If the volumes for the two runs are the same, you are saved this additional step.

Measure the lower baseline, and click **Save** to save and continue. Now start the second run in the normal manner, first stopping the rocking motion of the disc by pressing any button on the analyzer and then clicking the **START** button after the disc has stopped moving. Upon completion of the second run, save the sample information and bring up the raw data on screen. Enter the value for the upper baseline numerically, if necessary, and click **Save** to complete the analysis.

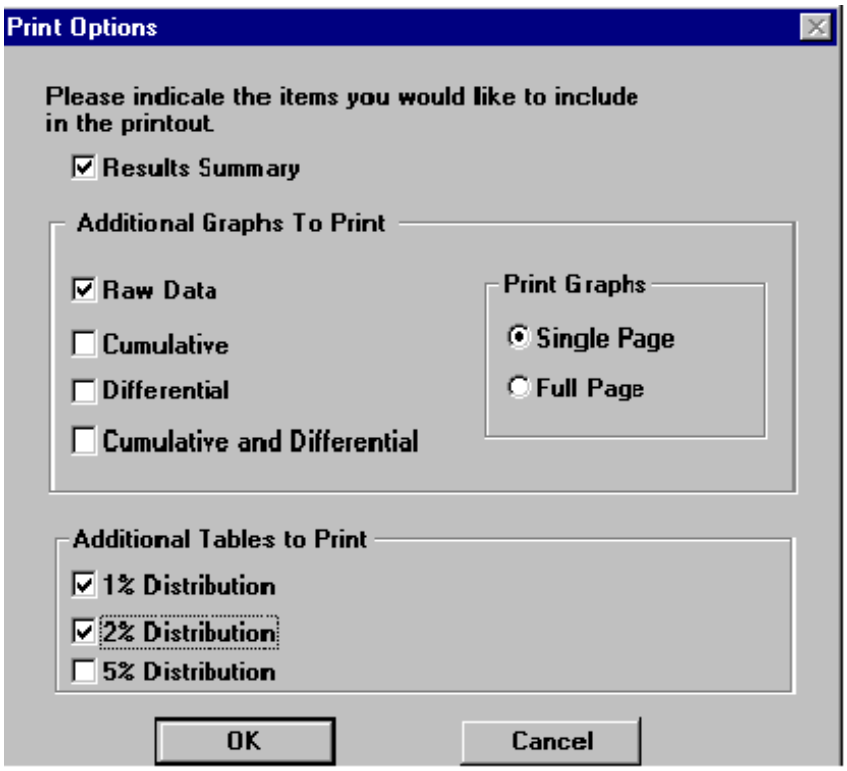
Now follow the procedure outlined before to merge the two data files.

View and Print Results

After the measurement is completed and saved, size distributions are calculated and results are displayed. Now from the main menu options the user can preview reports before printing, print reports, compare measurements, compare distributions, print SPC (statistical process control) charts. Click on the main menu under **File/Print Setup**, to get the following window. Set the appropriate printer, orientation and paper selection from this menu.

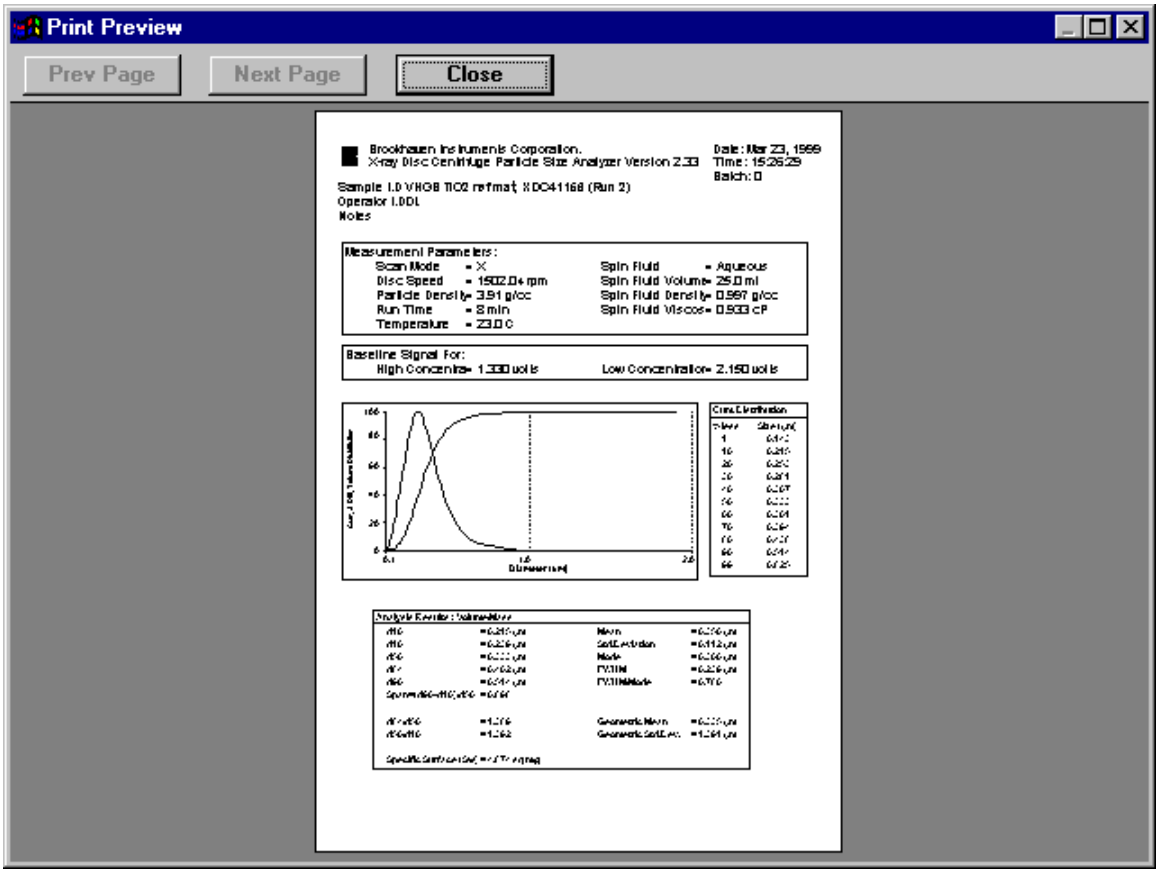


On the main menu under **File**, click on **Print Report Option** and select the different reports to be printed. The Result Summary is a single page summary of the sample parameters, the cumulative and differential graph and the analysis results. The user can check on any additional graphs and tables to print. The user can also print the size distribution graphs and the raw data graph on the same page.

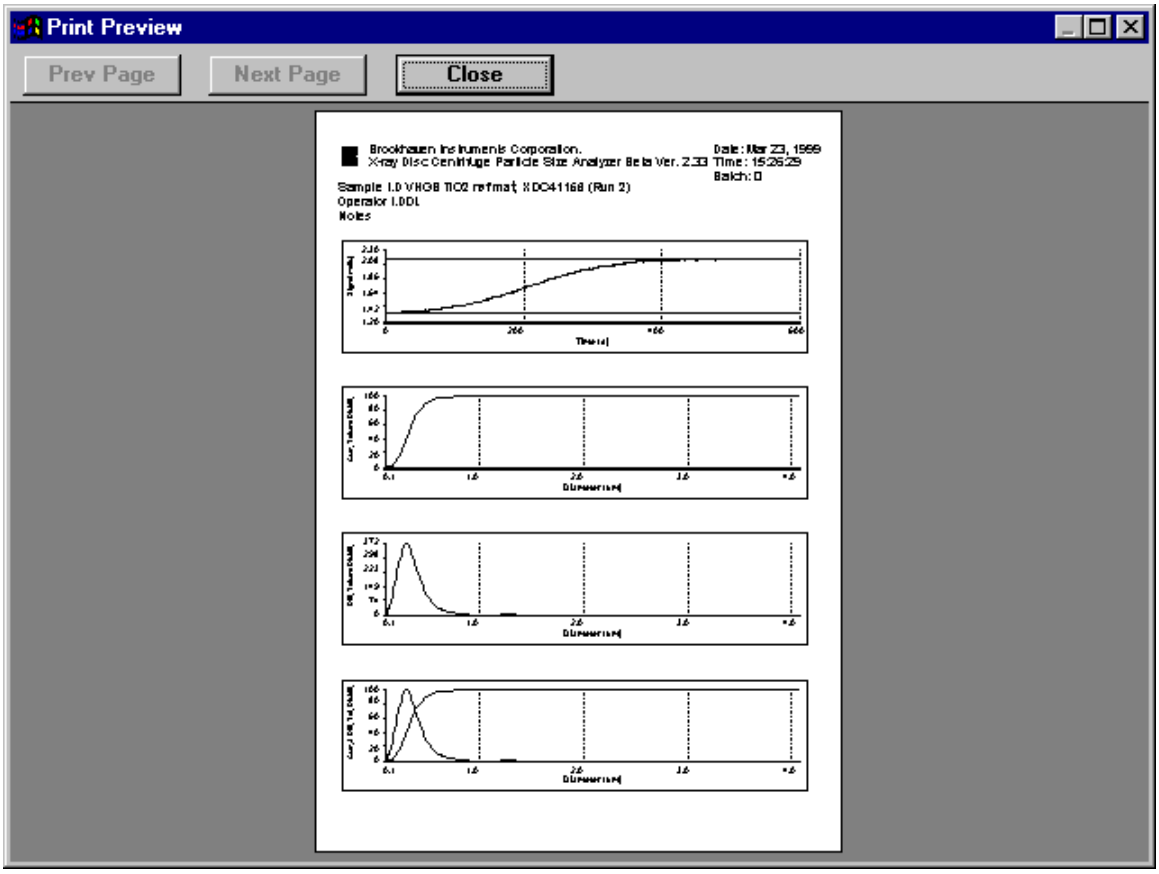


Click on **File/Print Preview** to view the reports and graphs before printing. After the Report Print Options are set, click on **File/Print** to print the reports in either proof or draft quality. Use draft quality to speed the printing process and increase the life of the printer cartridge.

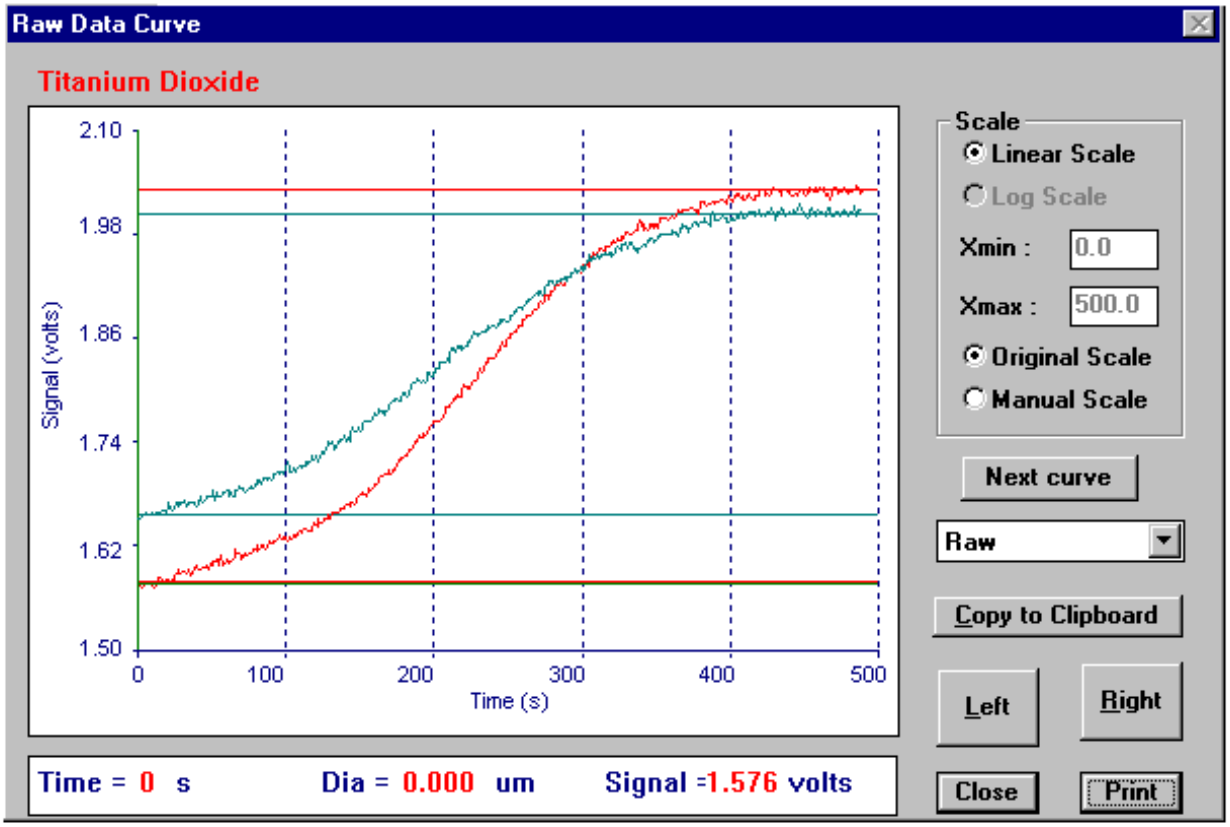
Here is an example of the Results Summary printout.



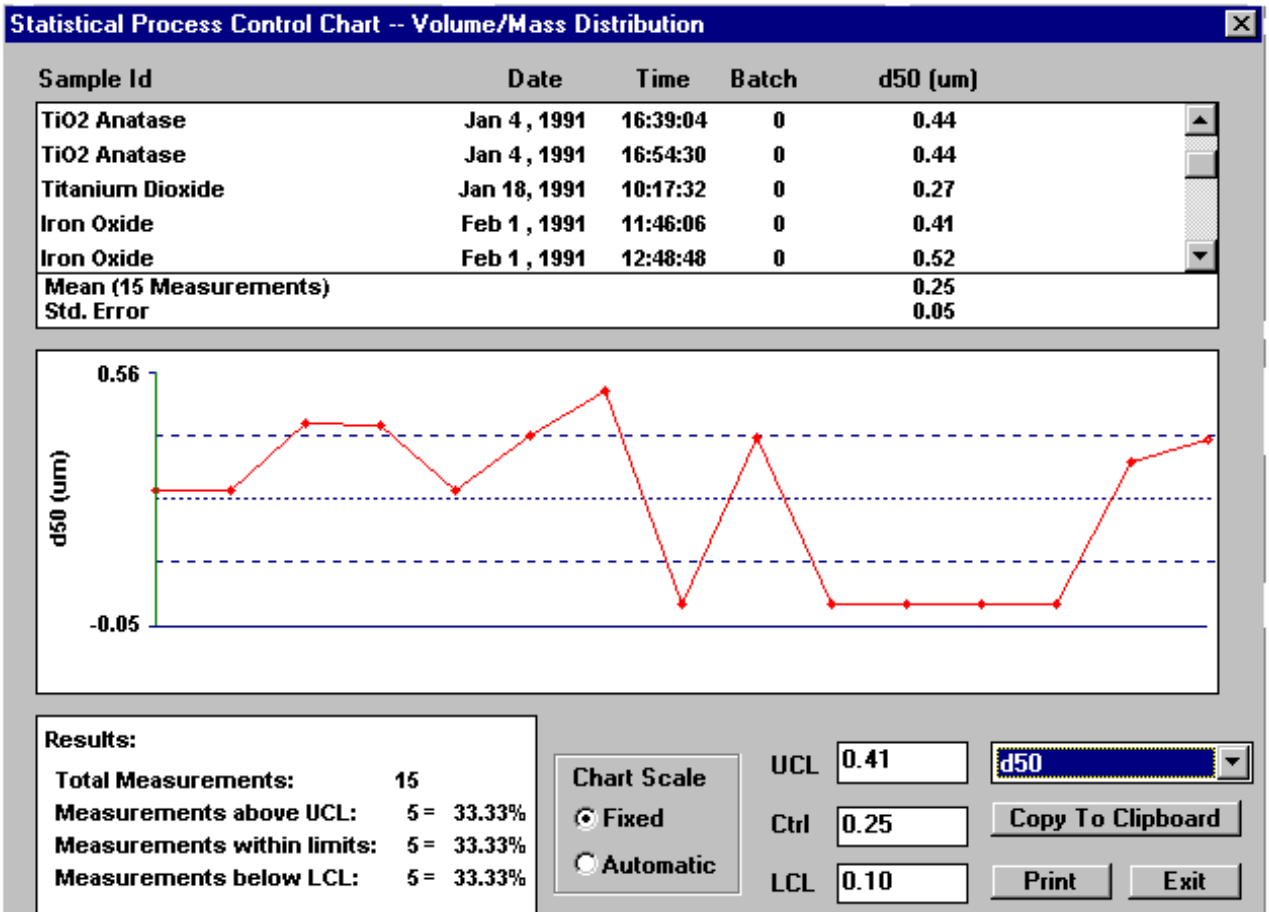
Here is an example of all graphs printed on a single page.



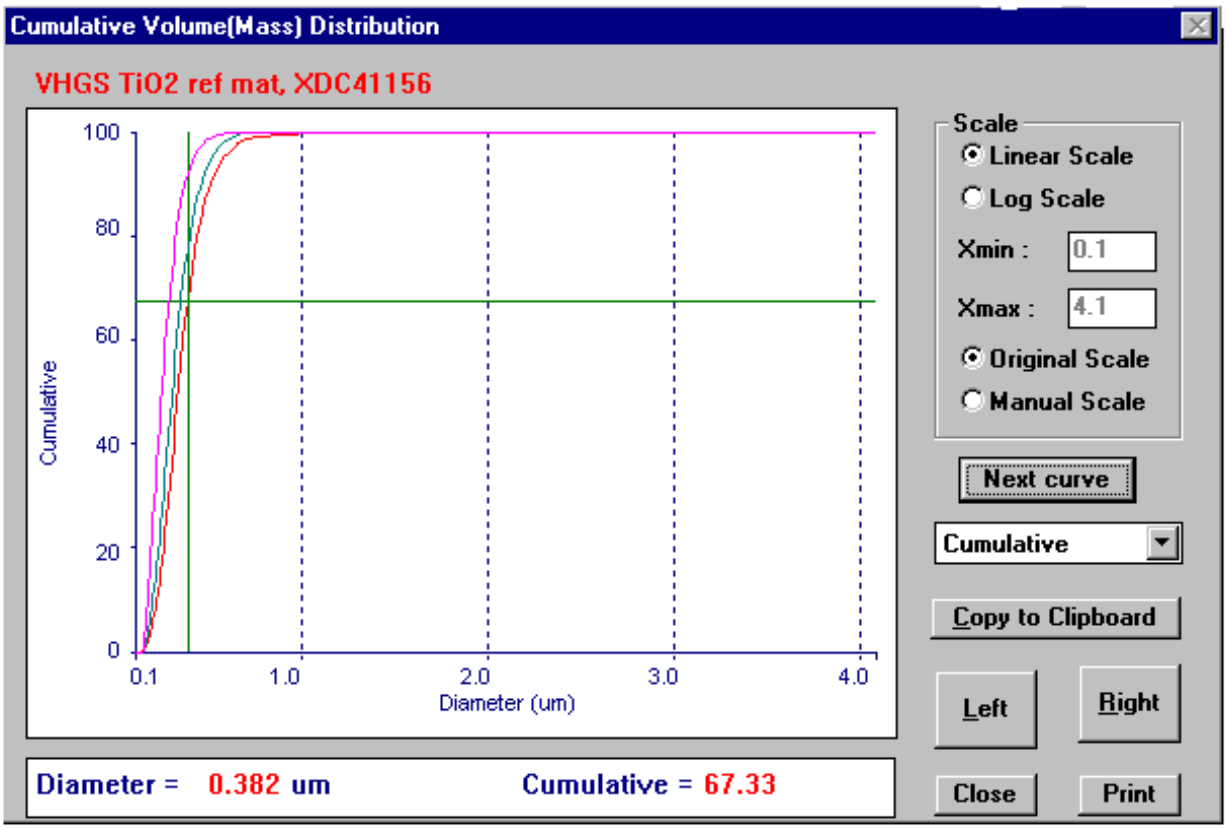
Click on **Graphs/Compare Measurements** to over lay up to six files graphically from the database. Choose the files to compare by holding the Ctrl key down and clicking on the files. Click **OK**. The File/Database window is closed, and the familiar Zoom window is displayed with the graphs for all the selected runs. Note that the user can now select the type of graph to view by the pull down menu option. Click **Next curve** to view each of the selected files. The user can also re-scale the X-axis and print the graph.



From the Main Window Menubar, click on **Graphs**/**Build SPC Chart** to get the Statistical Process Control chart. Just like in the file compare window the user will choose the files from the file database list and then click the **Ok** button. An example of the SPC chart is shown below. The SPC chart shows the Upper and Lower Control Limit values of the selected statistic or run condition. Select the statistic or run condition from the pull-down list-box. All values are plotted. If a value is within the limits or not. The statistics or run conditions included are D_{50} , the Mean, the Mode, the FWHM, and the Upper and Lower Baselines. By default the control, upper control limit, and lower control limit are defined as follows: Ctrl ≡ Mean Value; UCL ≡ Mean value + 3 ∗ Std. Error; and LCL ≡ Mean Value – 3 ∗ Std. Error. You may edit either limit as well as the control value.



Click on **Graph /Compare V/S/N Distributions** to again get the database file list. Choose one file to compare, and click the **OK** button to get the three cumulative distributions displayed on the same graph. The graph displays the volume/mass, the surface area and the number distributions in different colors. Click the **Next curve** button to cycle through the graphs. The active graph is the one designated in the title bar of the window. Click on the active graph to read out the x and y values.



Appendix I: Removing and Replacing the Shipping Screw

The screw must be fastened securely to prevent damage to the scanning head assembly during transport.

TO REMOVE THE SHIPPING SCREW DO THE FOLLOWING:

1) Make sure the unit is unplugged.
2) Unscrew the front panel; pull it out bottom first; set it face down in front of the unit.
3) You will see a slotted block on the lower left side of the unit with a T-handle screw sticking out of it. Remove the screw. You may need to use a pair of pliers to get additional torque to remove the screw.
4) Once the screw is removed, slide the washer on which it was mounted to the left side of the block, where a threaded hole is provided for storage of the shipping screw and washer. Now insert the screw through the washer into this hole and screw it in as far as it can go.
5) Replace the front panel, sliding the top in first.

TO INSTALL THE SHIPPING SCREW DO THE FOLLOWING:

1) With the unit plugged in press the HEAD button to retract the X-ray detector.
2) Now turn the unit OFF and unplug it from the mains.
3) Unscrew the shipping screw and move the washer on which it rests to the right. Align the washer with the locking hole provided in the slide and screw the shipping screw into this hole. Tighten the screw with a pair of pliers to get more torque.
4) Replace the front panel, sliding the top in first.

Appendix II: Removing and Replacing Discs

Follow these steps when removing a mounted disc:

1. If the detector head assembly is completely inside the body of the instrument proceed to the next step, otherwise turn on the power to the analyzer and press the yellow head button and wait until the head is completely retracted
2. Turn off the power to the analyzer.
3. Open the door on the right side of the analyzer.
4. By hand rotate the disc until the two locking screws on the hub are visible from the top.
5. Use the long, hexagonal cross section tool (Allen head) supplied with the XDC to loosen both screws. Hold the disc steady with one hand while using the other to loosen the screws.
6. Slowly and gently remove the disc. Notice the key and the keyway on the hub and shaft. Keep the key.

To replace a disc reverses the procedure above. Be sure that the key is fully seated in the keyways on the shaft and hub. Make sure that the disc is pushed back as far as it can go before you tighten the setscrews. Turn the power to the XDC back on. Leave the door open. Press the yellow button to restore the head in front of the disc. As the head moves back into place make sure that it does not touch the disc itself. If it gets jammed it will stop moving and will automatically retract after 25 seconds. In any case if the front of the head is too close to the disc, the disc must be pushed further back onto the shaft.

NOTE: THE DETECTOR HEAD ASSEMBLY MUST BE REPOSITIOND EVERY TIME A DISC IS CHANGED. Please refer to Appendix III for details on repositioning the head.

Appendix III: Detector Alignment Procedure for the BI-XDC

Theory:

Exactly 6 ml of water is injected into the disc. The signal output from the X-ray source is monitored as the head is moved upward until the meniscus is detected. This indicates the precise location of the head in relation to the edge of the disc bowl. The head must be moved up by 32 steps, once the meniscus is detected, to take into account the finite thickness of the X-ray beam.

Procedure:

Inject 6 ml of water into the disc bowl.

Stop the centrifuge motor if it is running. Press the HEAD↑ button. This will bring up the head movement menu on the little screen.

Press 3 on the keypad, "Align head". Press 1 on the keypad, "Manual Align". At this point the head will move until it finds its home position. On the bottom line of the little screen is displayed the current position of the head and the X-ray signal strength. The home position is assigned the value zero. The head can only be moved upward from this point.

The head is raised by pressing the HEAD↑ button and lowered by pressing the HEAD↓ button. Keeping either button depressed will move the head at a faster rate. Individual clicks of these buttons will move the head in single steps. Each step moves the head by 4 μm.

Press the HEAD↑ button until the signal indicates that the solid portion of the disc has been passed and the beam is passing through the water. Continue raising the head until the signal starts rising rapidly. This indicates that the beam is somewhere in the air-water interface at the meniscus. Raise the head further until the signal saturates at 2.500 V to confirm that the detector is really at the air-water interface.

The top edge of the beam should now be located at the edge of this interface with greater precision. Press the HEAD↓ button until the beam passes once more through the water alone (as indicated by a drop in the signal). Now raise the head one step at a time until a slight increase in the signal strength (0.05 V more than the maximum reading observed with the beam passing through water alone) is observed. The top of the beam is now located at the meniscus.

Raise the head 32 steps more and press the MOTOR button to complete the alignment procedure.

NOTE: If you have changed discs you must change the disc calibration parameters in the Setup Menu of the XDC program. Use the following equation to calculate the Detector Radius:

$$R_{det} = [R^2_{disc} - Volume/(\pi * Width)]^{0.5}$$

Appendix IV: Concentration Definitions and Calculations

A reasonable concentration for measurements with any sedimentation device, including the BI-XDC, is 0.005 volume fraction, also designated as 0.5% by volume. While there are some cases where a concentration of this magnitude might cause particle-particle interactions and, as a result, distortions in the final answer, this is s good initial value. If in doubt, make measurements at .025% by volume to see if there has been a systematic shift in the results. (Occasionally, depending on the sample, you may be forced to work at higher concentrations in order to get any signal.)

The volume fraction, ϕ, is a fundamental parameter for describing concentration. The cube root of ϕ is proportional to the ratio D/L, where L is the average center-to center inter particle distance and D is the particle size. When the volume fraction is high, the particles are close together, and concentration effects on the measured size are more likely to be important.

ϕ is defined as,

$$\phi = V_p/V_s = V_p /(V_p + V_l) \qquad \text{where}$$

V_p = the total volume of all particles,
V_l = the volume of the liquid,
V_s = the total volume of the suspension (particles + liquid).

For example, assume a suspension with 1 ml of particles and 49 ml of liquid. Then $\phi = 0.02$. The concentration is 2% by volume.

{Note for Chemists: Unless a solution is ideal, the total volume is not equal to the sum of the component volumes. This is due to molecular interactions. Suspensions are not solutions. Only the surface molecules around a stable particle interact significantly, if at all, with the liquid in which it is suspended. Therefore, it is a good approximation to assume that the volumes add. One need not be concerned with partial molar volumes. }

For low concentrations $V_p << V_l$ and $\phi \approx V_p/V_l$. For high concentrations, this approximation is no longer valid.

Another common definition for concentration is the mass fraction defined as the total mass of particles divided by the total mass of suspension. Like the volume fraction, the mass fraction is a true fraction: it has no units. Unlike the volume fraction, the mass fraction is not directly related to particle-particle interactions. In addition, it is not common practice to weigh the liquid used to make a suspension. Instead, the volume of the liquid is measured. This practice gives rise to the most common definition of concentration.

Let $C = M_p/V_l$, where M_p equals the total mass of the particles. For example, if 1 g of particles is added to 50 ml of liquid, then C = 0.02 g/ml.

This is – unfortunately and illogically – often referred to as a 2% solids concentration, or 2% by weight. Notice the mixed units, g/ml. A fraction and the corresponding percent have no units, as in the case of volume or mass fraction. A further problem arises when a concentration is given only as 2%. It is not clear whether this means volume or mass fraction or C. If in doubt, ask.

With the help of a little algebra, one can relate ϕ and C. Namely,

$$C = \phi \cdot \rho_p / (1 - \phi) \qquad \text{or} \qquad \phi = C/(C + \rho_p)$$

Where, ρ_p is the density of the particle.

For example, if a 30 g sample of particles with $\rho_p = 1.2$ g/cm^3 is suspended in 50 ml of liquid, C = (30 g)/(50 ml) = 0.6 g/ml, or 60% solids. Using the above equation to calculate ϕ results in 33.3% by volume.

For the low concentrations, $C \approx \phi \cdot \rho_p$. This is particularly useful approximation. For example, it was stated at the beginning that making measurements at $\phi = 0.5\%$ is a good starting point. For polyvinyl chloride, where $\rho_p = 1.38$ g/cm^3, C = 0.5% x 1.38 = 0.69%; and for TiO_2, where $\rho_p = 4.2$ g/cm^3, C= 0.5% x 4.2 = 2.1%.

In preparing samples for particle size analysis it is quite common to use wetting agents and surfactants. These should be used at very low concentrations. In addition, the density of these materials is typically close to 1g/cm^3. For these two reasons, it is normal practice to ignore completely the difference between ϕ and C.

Likewise, when preparing samples for use with the BI-90, BI-FOQELS, ZetaPlus, and the BI-200SM (all instruments based on dynamic light scattering), the concentrations are typically between 10^{-5} to 10^{-2} volume fraction. At such low concentrations the difference between suspension volume, V_s, and liquid volume, V_l, is insignificant, and the difference between ϕ and C is, again, often ignored.

Ignoring the difference between ϕ and C, at low concentrations, is very common and acceptable for rough, qualitative work. It is never acceptable for quantitative work. For example, if you are not sure if the current dilution is sufficient, halve the concentration, and repeat the measurement. It is not too important to know the exact concentration or its definition, if and only if the concentration is roughly in the right range. If, however, you want to plot values against concentration or fit results to an equation in concentration, then you must be quantitative when measuring, diluting and specifying the concentrations, especially the units.

Appendix V: ASCII Data File Format

This is the ASCII data file format created from **File/Database/Export Files**.

Company Name
Sample I.D., User I.D., Notes
Run Number
Batch Number
Scan Mode
Disc Speed (rpm)
Scan Distance (cm), Scan Speed (mm/min) , Scan Time (m)
Scan Delay Time (s), Run Time (m)
Particle Density (g/cc)
Spin Fluid Name
Spin Fluid Density (g/cc), Viscosity (cP), and Temperature (°C)
Spin Fluid Volume (ml)
Sampling Interval (s)
Disc Cavity Width (cm), Disc Cavity Radius (cm), Detector Radius (cm)
Measurement Date (mm/dd/yy), Time (hh:mm:ss)
Upper Baseline (volt), Lower Baseline (volt)
Number of Sample Points
Diameter 1,Cumulative Mass 1,Differential Mass 1,Log Differential Mass 1
Diameter 2,Cumulative Mass 2,Differential Mass 2,Log Differential Mass 2
.
Diameter N, Cumulative Mass N, Differential Mass N, Log Differential Mass N
Number of Results
d_{10}
d_{16}
d_{50}
d_{84}
d_{90}
d_{50}/d_{16}
d_{84}/d_{50}
span = $(d_{90} - d_{10})d_{50}$
mode
delta d_{50}
delta d_{50}/mode
mean
standard deviation
geometric mean
geometric standard deviation
specific surface
Number of raw data points
Raw Signal Values (volts)
.
.

Appendix VI: Distribution Statistics, Definitions and Calculations

Following are the definitions of the various distribution statistics that are calculated in the "Detailed Results" window.

d_{xx} is the diameter at the XX percentile of the cumulative undersize distribution. (For example, d_{50} is the 50^{th} percentile, also known as the median. And d_{25}, d_{50}, and d_{75} are the 25^{th}, 50^{th}, and 75^{th} percentiles, also known as the quartiles.)

Span is one measure of distribution width. We define span as $(d_{90} - d_{10})/d_{50}$. Our definition of span is relative.

d_{84}, d_{16} are used to compare a monomodal distribution to a lognormal distribution. When $d_{84}/d_{50} = d_{50}/d_{16}$, the measured distribution, if monomodal, approximates a lognormal distribution, at least over the range from 84% to 16%.

The mean diameter is calculated from the differential distribution.

$$\text{Mean} = \frac{\sum_{i=1}^{n} d_i V_i}{\sum_{i=1}^{n} V_i} = \bar{d}_v$$

Where, V_i is the volume of the particle, with diameter d_i, and n is the number of size classes. (The number of size classes, except for merged file, is 98, independent of the runtime. In other words all data points are smoothed and then redistributed linearly over 98 size classes.) Analogous definitions for distributions weighted by Number and Surface Area apply.

The Std. Deviation, also calculated from the differential distribution, is another measure of distribution width, and is defined as follows:

$$\text{Std. Deviation} = \left(\overline{d_v^2} - \overline{d_v}^2 \right)^{\frac{1}{2}}$$

Where,

$$\overline{d_v^2} = \frac{\sum_{i=1}^{n} d_i^2 V_i}{\sum_{i=1}^{n} V_i}$$

The mode in the differential distribution is the diameter of the highest value and is most important in a monomodal distribution. For multimode distributions use the cursor to determine the diameters at the remaining modes.

FWHM is the full width at half maximum of the highest peek in the differential distribution. It is most useful for a monomodal distribution. It is another measure of distribution width.

FWHM/Mode is a unit less ratio. It is a relative measure of the distribution width.

Geometric Mean is calculated as follows:

$$\text{Geometric Mean} = \sqrt[\frac{1}{\phi}]{\prod_{i=1}^{n} d_i^{V_i}}$$

Where , $\phi = \sum V_i$.

For a monomodal distribution that is uniquly described by the lognormal function, the modal diameter equals the geometric mean diameter.

Geometric Std. Dev. is calculated as follows:

$$\text{Geometric Std.Dev.} = \sqrt{\frac{d_{84}}{d_{50}} \times \frac{d_{50}}{d_{16}}}$$

When the Geometeric Std. Deviation = 1, the distribution is monodisperse. In this case all d_{xx} values are equal, FWHM = 0, and all mean values are equal and equal to the modal diameter.

Note: This simple method of estimating geometric standard deviation is only valid when the actual distribution is accurately described by a lognormal function. When this is true $d_{84}/d_{50} \approx d_{50}/d_{16}$, values of which are presented in the “Detailed Results” window. A more complete definition using all the data points is given in William C. Hinds[1], chapter 4, pg.85, equation 4.40.

[1] William C. Hinds, Aerosol Technology Properties, Behavior, and Measurement of Airborne Particles; Wiley-Interscience Publications: New York, 1982.