# University of Minnesota Nano Center Standard Operating Procedure

**Equipment Name:** Laser Diffraction Particle Analyzer
**Model:** Microtrac Bluewave
**Location:** PAN 185
**Badger Name:** Not on Badger
**Revision Number:** 1
**Revisionist**: J. Marti
**Last Revision Date:** 9/10/2014

## A. Introduction

1. **Tool Description.** The Microtrac Bluewave is a particle characterization tool that uses diffraction, also known as static light scattering, to derive size distributions for particles in the size range 100 nanometers to 2800 micrometers. The system suspends the particles in a liquid and circulates the suspension through an optical cell where lasers of three different wavelengths strike the sample. The light is diffracted by the particles in the sample, and these diffraction patterns are recorded by detectors. An inversion algorithm is used to calculate the particle size distribution based on these diffraction patterns.

2. For particles prone to agglomeration, the Bluewave offers an in-line ultrasonicator that can be used to break up agglomerates. Denser or larger particles can be prevented from settling by using faster circulation speeds. Particles in a dry state can be tested by suspending them in a liquid chosen so that it will not dissolve or swell the particles, along with the appropriate choice of surfactant.

3. **Tool Components**. Figure 1 shows a front view of the Bluewave. Key components visible in this view are:
    a. Main laser diffraction system
    b. Sample delivery controller (SDC)
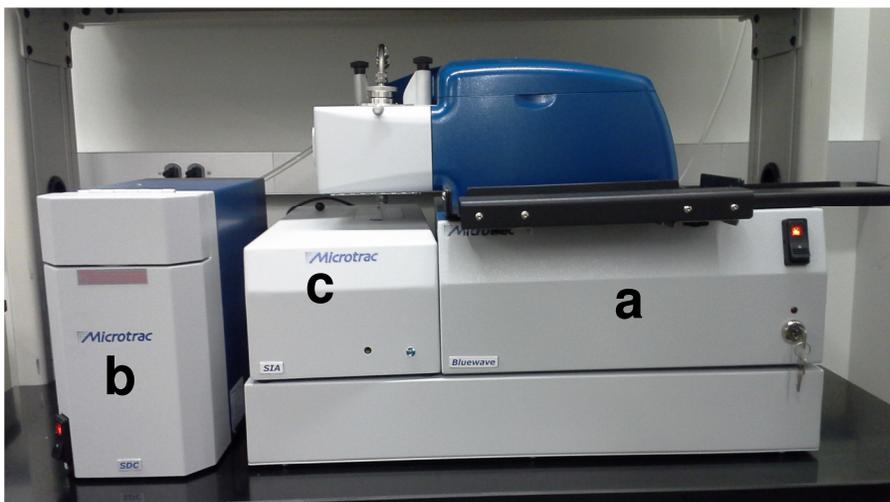    c. Optical particle shape analyzer



Figure 1. The Microtrac Bluewave tool.

4. **Safety**. If using non-aqueous solvents for the suspension liquid, use normal safety precautions when filling or draining the system, i.e., use solvent-resistant gloves and splash-proof goggles.

5. **Restrictions/requirements**. Particles smaller than 100 nm should be measured using the DLS rather than the laser diffraction system.

6. **Required Facilities.** A source reservoir for the suspending liquid and a reservoir for the liquid drain. Separate reservoirs must be provided for water and for solvents. In addition, 120V AC power and a connection to a Windows-based computer are required.

7. **Definitions.**
   a. Particle size distribution (PSD): a plot of particle concentration versus size. PSDs may be relative or absolute, and can be expressed in terms of particle count per size bin (number distribution), particle surface area per size bin (area distribution), or total particle volume pre size bin (volume distribution).
   b. Static light scattering is a technique to estimate the size distribution of particles between about 0.1 and 30000 microns in size. Particles are suspended in a liquid and circulated through an optical cell, where they are illuminated by a monochromatic light source, creating diffraction patterns. These patterns are recorded by multiple detectors. An inversion process is used to calculate a particle size distribution that created this set of patterns.

# B. Using the Bluewave

1. **Setup**
   a. Begin a new entry on the tool log sheet. List your name, the date, the materials you are working with (particle material(s) and suspending liquid), and any issues you encountered during your run.
   b. If not already on, turn on the Bluewave, then the computer. Log into the computer using your own X500 user name and password. This enables you to store files in your own My Documents folder, secure from other users.
   c. Open the operating software, Microtrac Flex. The software home screen will load (Figure 2). The screen contains controls for the laser diffraction analyzer and the liquid flow system, as shown in the figure.

2. **Sample preparation**
   a. Particles that are suspended in water can be directly added to the Bluewave's circulation system. Particles that must be suspended in organic solvents require the draining of the Bluewave's circulation system (normally filled with water), followed by refilling with a compatible solvent. **Arrange with MNC staff to perform this draining/refilling operation prior to your work session.**
      i. Organic suspension liquids include methanol, isopropanol, and hepane.
      ii. Other specialty suspending liquids can be used if needed. Consult MNC staff to find a compatible liquid for your particles.

Figure 2. Flex software home screen

    **b.** Dry particles that are insoluble in water can be added directly to the Bluewave's circulation system. Consult MNC staff to determine if you suspect your material may dissolve or swell in the suspending liquid.

3. **Designing a Standard Operating Procedure (SOP)**. Before making a measurement, you must select an SOP (i.e., a run recipe) from a list of pre-defined procedures. The SOP defines the material properties of your sample (particle and liquid), how many times the measurement is to be carried and for how long, and several other required parameters. If none of the pre-defined SOPs meet your needs, you can edit an existing SOP and save it under a new name.

    a. In the Microtrac Flex home screen, click on the SOP button at the upper left. The "Measurement SOP" dialog box will appear. The top part of the box shows which SOP is currently loaded. The middle portion provides two drop-down menus; the upper menu allows you to select and load a pre-defined SOP. If you are using an SOP you've previously defined, select that SOP and then close the "Measurement SOP" box.

    b. If you need to define a new SOP, select any SOP in the upper drop-down menu, then type a name for your new SOP in the second drop-down menu.

    c. Click the "Options" button in the lower right. This brings up a series of screens with tabs that allow you to customize the SOP. The main tabs to modify are Timing and Analysis.

    d. Under the Timing tab, input the number of seconds to measure your sample; 10 seconds is the default value. Suspensions that have a wide range of particle sizes may require somewhat longer run times. It is a good idea to do at least three repeated measurements to determine repeatability and uncertainty.

    e. Under the Analysis tab, there are several sub-tabs. The first is Particle Information, where you enter particle parameters.

i. Check the Save/Recall drop down menu to see if your particle has a pre-defined parameter set; if so, select it with the drop-down menu. If not, enter the name of the particle material in the second drop down menu.

ii. Indicate if the material is expected to be absorbing or transparent. If the latter, provide an estimate of the refractive index of the material. For your convenience, refractive index values can be obtained for hundreds of materials by clicking on the "Particle Reference" folder icon.

iii. Indicate whether this material is expected to be spherical (usually the case for particles synthesized chemically) or irregular (the case for ground or milled particles).

iv. After setting these parameters, click the Save button to make this particle parameter set a permanent part of the program database.

f. The next sub-tab under Analysis is for entering information on the fluid suspension medium. Check the Save/Recall drop down menu to see if your suspension liquid has a pre-defined parameter set; if so, select it with the drop-down menu. If not, enter the name of the suspension liquid in the second drop down menu. The main numbers needed to define this material are index of refractive and liquid viscosity.

i. Refractive index values for most common suspension liquids can be obtained by clicking on the "Fluid Reference" folder icon.

ii. The same reference table gives information on viscosity vs. temperature for these liquids.

iii. Enter the information as required, then click the Save button to make this fluid parameter set a permanent part of the program database.

g. Do not alter the settings under "Advanced Analysis", "Analysis Options", and "Sample Loading".

h. The next box to update is "SDC", which set the parameters for the Sample Delivery Controller, namely:

i. Number of rinses: between samples the liquid circulation system should be flushed and rinsed. This control sets the number of rinsing cycles when the operator hits the Rinse key.

ii. De-aeration cycles: during rinse and fill operations, the Bluewave executes a waiting period to de-aerate the liquid and allow air bubbles to exit the chamber.

iii. Flow rate. Input a value between 5 and 100 to circulate the liquid at the desired speed. The default value is 60. Larger particles (> 100 um) may require more vigorous circulation to prevent settling.

iv. Ultrasound intensity and duration. Ultrasonic energy may be applied to the circulating suspension to break up agglomerates. These options let you set the ultrasound intensity (between 20 and 40 in arbitrary units) and duration (up to 999 sec).

v. All these values can be saved under a named SDC SOP.

i. The last box to update is the Sample ID information. The ID has four data fields to fill in. The first is for the sample name. Field 2 may be used for any sample label or descriptor you choose. Field 3 should be your last name. This allows for easier searching of data records after the measurements have been taken. The sample ID can be updated prior to each measurement.

j. After Timing, Analysis, SDC, and ID parameters have been set, click on Save. You

will be returned to the software home page. The SOP is now updated and loaded for running.

4. **Setting zero.** The detectors on the Bluewave have some intrinsic electronic noise that must be subtracted from the signal. This is done via the Set Zero function.
   a. Ensure the SDC is filled with clean, particle free suspension liquid.
   b. Start the liquid circulating by clicking on the "Flow" icon on the right side of the Flex home screen.
   c. At the top of the Flex home screen, click on "SZ". You will go to the Setzero screen.
   d. The system will automatically collect a signal blank, and calculate a correction factor. After about 10 sec, the system will announce whether it has acquired a sufficient set zero value.
   e. If the zero value was not set successfully, it may be that the suspension liquid was not clean and particle free due to carry-over of particles from a previous measurement. Drain the liquid using the "Drain" button on the right side of the Flex home screen, then click on "Rinse" This will fill and drain the system multiple times to purge any contaminant. When rinsing is done, refill the SDC, start the circulation flow, and repeat the set zero procedure.

5. **Loading the sample.** Once the SOP has been selected and loaded, you are ready to test your sample. The Bluewave's detectors require a certain range of signal in order to get a reliable measurement, which depends on particle concentration. This loading range is determined using the LD (Load) screen.

   First, click on the Flow icon on the Flex home screen to begin the liquid circulating. Click on the "LD" button on the top of the screen. You will see a vertical scale indicating reflected laser power, with the optimum range indicated in green. Begin adding small amounts of your sample to the sample port in the liquid circulation system. Watch the loading scale and add sample until the scale is in the green range. This indicates a sufficient concentration for a reliable measurement.

   Ensure that the loading does not exceed the maximum level. If it does, you may use the "Dilute" function on the right side of the home screen to partially drain the liquid and add fresh liquid.

6. **Running the sample.** Once the correct loading has been achieved, click on the Run button. You will be prompted to review or update the sample ID info. When you hit okay, the system will run a measurement for the time you selected in the SOP.

   The results will be plotted on the screen as a distribution plot of the percentage of the particles in the sample that lie in each narrow size range, referred to as a size channel. The full particle size range is 0.01 to 2800 micrometers. You have several options on how to plot this data.
   a. Number distribution. This gives the percentage of all particles in the sample that lie in each size channel.
   b. Volume (or mass) distribution gives the percentage of the total particle volume in the sample that lies in each size channel. Since large particles in a sample represent

most of the total particulate volume, the volume distribution is dominated by the larger particles.

    c. Size distributions can also be given in terms of particle surface area.

Which measure of size distribution to use will depend on what you are looking to find out about your sample. Consult with MNC staff to discuss the choice in detail. Once the data has been collected, the Microtrac software can switch between number, area, and volume distributions without having to re-test the sample.

7. **Running multiple samples.** After you have run a sample, you need to drain, rinse and refill the circulation system to remove the old material.
    a. Click on the Drain icon. Fluid will drain in a few seconds.
    b. Click on the Rinse icon. The system will be rinsed with fresh liquid. The number of rinses can be set in the SOP.
    c. Click on the Fill icon to replenish the suspension liquid.
    d. You may now turn on the circulation flow and proceed to your next sample. Repeat this procedure for each new sample or material. It is not necessary to establish a new Set Zero value unless you are switching the type of suspension liquid.

8. **Exporting your data.** After the data is displayed on the screen, you can right click on the plot to either print the graph in room 185 or export the data digitally. The data may be exported as comma separated variable (CSV) format, which makes it easy to export into spreadsheet or scientific graphing programs. The graph may be exported as a PDF file, if you would like to insert the graph into a word processing or presentation file.

9. **Using the database.** All measurements taken with the Bluewave are recorded in a database named MTData. To access a data record, click on the "DB Retrieval" button on the main interface (go to the File tab on the main interface select the "Open Database" command?). You will be given a database query form which enables you to select the data sets from previous measurements (collectively known as "records"). You may search by record date range, or by any value in any of the record label fields. Click the "Enable" box to allow searching by data field. Note that since the third data field in the sample ID should consist of the last name of the operator, you should be able to search for all your records at once by using this field.

After you have entered the data on the query form, clicking on the next tab will perform the search and bring up the records matching your queries. If no search fields are used, or if you forget to click the "Enable" box, all records in the database will be returned, sorted by date.

Select the records that you wish to view or export using the mouse, then click "Retrieve". The records will be plotted using the same format as that of live measurements, except that a few new buttons are enabled. If you selected more than one record, you can scan through them using the "Next" or "Prev" buttons. The recalled data may be used to recalculate a distribution (for example, from volume to number distribution), exported to another data format, or printed using the local printer.

## C. Tool Shutdown

When your work is complete:
1. If working in water as the suspending liquid, perform a final drain, rinse, and refill of the system.
2. If working in an organic solvent, contact MNC staff to let them know you are finished. They will handle solvent disposal.
3. Clean the work area and place used beakers near the sink for cleaning.
4. Log out of the computer.