University of Minnesota Nano Center Standard Operating Procedure

Equipment Name: Optical Particle Analyzer
Model: Microtrac SIA
Location: PAN 185
Badger Name: Not on Badger
Revision Number: 3
Revisionist: J. Marti
Last Revision Date: 4/9/2020

A. Introduction

1. **Tool Description.** The Microtrac SIA is a particle characterization tool that uses a camera and image analysis system to obtain both size and shape information on particles between 1 and 1000 micrometers in size. The system suspends the particles in a liquid and circulates the suspension through an optical cell, where a high speed camera obtains a digital image of each particle in the focus plane. These images are analyzed to obtain particle size parameters (diameter, area, percentiles) and a number of shape metrics, such as aspect ratio, sphericity, ellipticity, and concavity/convexity. Note that this technique is a single particle analysis, i.e., the size distribution is constructed from measurements on individual particles. Compare this approach to ensemble techniques like laser diffraction or dynamic light scattering, where size distributions are estimated based on as observations of the collective scattering by a large population of particles.

2. The SIA is mounted on the Bluewave laser diffraction tool (see Figure 1), and the two systems share a common liquid flow system, the sample delivery controller (SDC). This allows both laser diffraction and optical measurements to be obtained simultaneously on the same sample. An in-line ultrasonicator in the SDC can be used to break up particle agglomerates, and denser/larger particles can be prevented from settling by using faster liquid circulation speeds. Dry particles can be tested by pre-suspending them in a small amount of liquid, then adding them to the SDC flow system. Suspension liquids should be chosen so that they will not dissolve or swell the particles. Surfactants may be used to help suspend particles.

3. **Tool Components**. Figure 1 shows a front view of the SIA/Bluewave combination. Key components visible in this view are:
   a. Bluewave laser diffraction system
   b. Flow system/sample delivery controller (SDC)
   c. SIA optical particle shape analyzer

4. **Safety**. If you are using non-aqueous solvents for the suspension liquid, use solvent-resistant gloves and safety glasses when working with the system.

5. **Restrictions/requirements**. Particles smaller than 1 μm are too small to be imaged by the SIA optical 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.

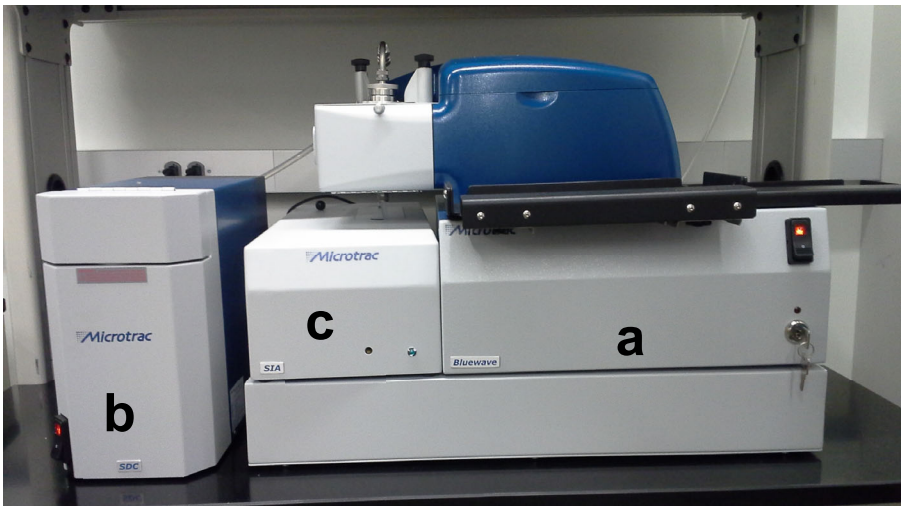

Figure 1. The Microtrac SIA (labeled c), mounted on the Bluewave tool (a).

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. Differential size distribution: a plot of particle concentration versus size that presents the differential number or fraction of particles at a given size.
   c. Cumulative size distribution: a plot of particle concentration versus size that presents the total number or fraction of particles that are at or below a given size.
   d. Aspect Ratio: a measure of particle shape defined as the particle's largest dimension divided by its smallest dimension. The aspect ratio of a circular or spherical particle is 1.0.
   e. Ferret diameter: the measure of an object's size along a specified direction. This is usually defined as the distance between the two parallel lines that are tangent to the object's edges and perpendicular to the specified direction. It is also called the *caliper diameter*. This is one of several different measures of particle size used by the SIA software.
   f. For a more complete set of particle shape metrics, consult the (reference).

B. Using the SIA

1. **Setup**
   a. Begin a new entry on the tool log sheet (use the Bluewave log sheet for SIA runs). List your name, the date, and the materials you are working with (particle material(s) and suspending liquid).
   b. If they are not already on, turn on the SIA, the Bluewave, the SDC, and 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, PartanSI, by double clicking on the desktop shortcut.

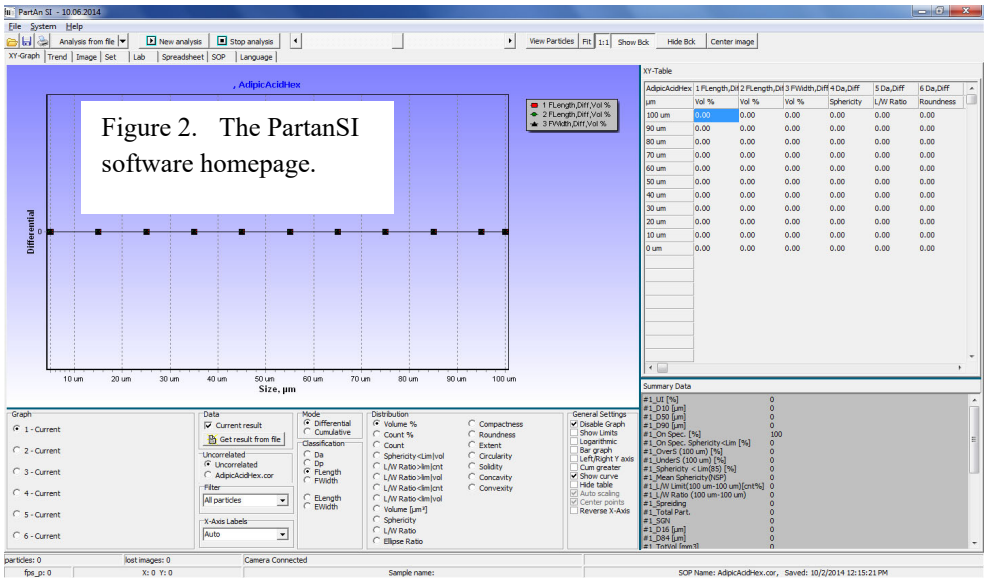

Figure 2. The PartanSI software homepage.

The software start screen (shown in Figure 2) will open.

Note: By default, the software opens in a limited user mode. To gain full access to all the features of the software, you must log in as a full user. See the lab manager for the password and method to do this.

d. Open the Microtrac Flex software. Figure 3 shows the Flex software start screen. The controls on the right side of the screen will be used to control the flow system. To operate the SIA simultaneously with laser diffraction, see the SOP for the Bluewave.

2. Sample preparation

a. Dry particles that are not water soluble or those that are already suspended in water can be directly added to the SDC circulation system. Particles that dissolve in water and must be suspended in organic solvents will require the draining of the SDC system, which is left filled with water by default, followed by refilling the system with a compatible solvent. **Arrange with the lab manager to perform this draining/refilling operation prior to your work session.**

- **i.** Available organic suspension liquids include methanol, isopropanol, hexane, heptane, and toluene.
- **ii.** Other specialty suspending liquids can be used if needed. Consult the lab manager to find a compatible liquid for your particles.

b. If you have dry particles that are insoluble in water, test their wetting properties prior to introducing them into the SDC. To do this, suspend the particles in a small vial of water and agitate them.

- **i.** If the particles suspend readily and do not stick together, they can be added directly to the SDC system.
- **ii.** If the particles stick together or do not suspend readily, you will need to develop a sample prep protocol to follow for all your measurements. Consult MNC staff for assistance in developing this protocol.

c. Dry particles that are soluble or swell in water must be run in an organic solvent and should be tested in the same fashion as in item (b) above. Pretest your dry particles in the organic liquid to determine how well they are wetted by the liquid, and select a suspending liquid based on which liquid gives optimum particle suspension under slight agitation.

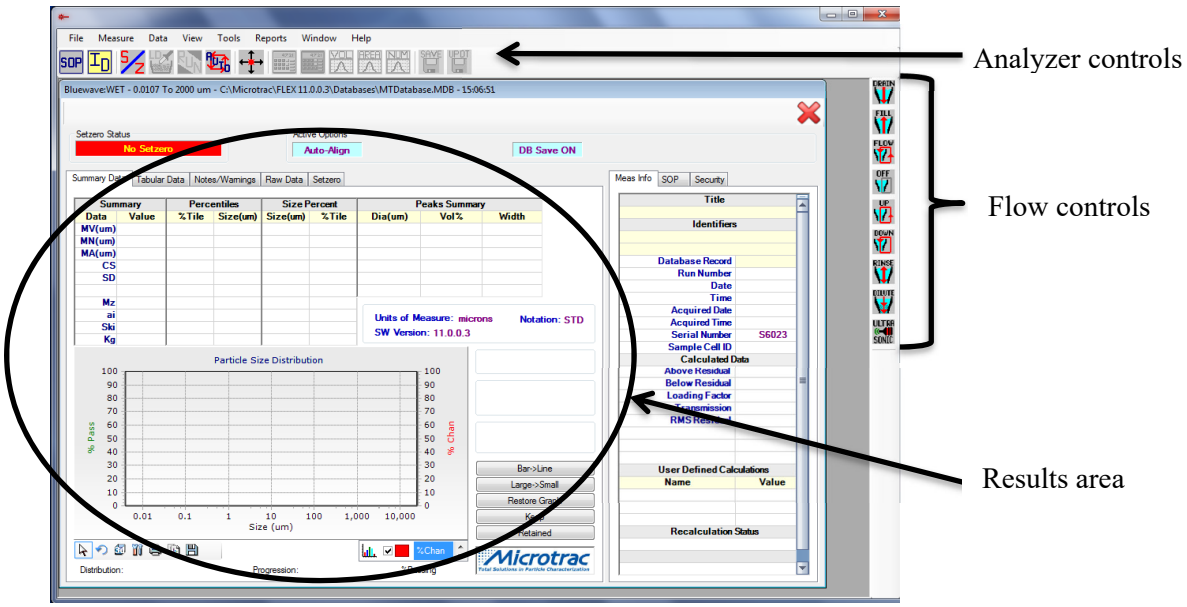

Figure 3. Flex software home screen

3. **Designing a Test.** Before making a measurement, you must define a Standard Operating Procedure (SOP), that is, a run recipe, to set the parameters for the run. The SOP defines how the measured particle sizes will be analyzed and displayed. 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 PartanSI home screen, click on the "New Analysis" button at the upper left. The "Load Correlation" box will appear. If you have previously defined an SOP, select the correlation file of the same name. Otherwise, select the "default.cor" file.
    b. You will then be directed to a screen with several tabs; you will edit only the "Lab" and "SOP" tabs.
    c. Under the Lab tab, enter information in the following fields. The "Ticket Number" field is not used.
        i. Sample name field: a short descriptive label for your sample
        ii. Field 1: any further info you care to add about the sample
        iii. Field 2: your last name.
    d. Under the SOP tab, review and edit the following items on the left side of the screen:
        i. Under "Logarithmic bins", set the upper and lower bounds of the measured size distribution. This should be broad enough to encompass all the particles sizes you expect may be in your sample. If unsure, select 1 to 1500 µm.
        ii. Determine the number of size bins in the distribution. These intervals can be defined as linear or logarithmic progression between the lower and upper size limits. A reasonable number is 20-40 size bins.
    e. On the right side of the SOP screen, review and edit the following items:
        i. Start/Stop control: set the desired measurement time for the run. If your sample is concentrated, 1-2 minutes will be enough to count a sufficient number of particles to get reasonably good statistics. If the sample is dilute, you may need 5 minutes or more. Larger particle counts generate more reliable results, so if your first measurement counts less than ~1000 particles,

increase the counting time.

ii. Summary Data: On the table marked "visible summary data", select which parameters you want plotted in the X-Y data graph.

4. **Loading the sample.** Once the measurement SOP has been designed in the PartanSI software, you must start the SDC flow system. Go to the Flex software home screen (Figure 3) and use the flow controls on the right side of the screen.

   If this is the first time you have run the system today, select "rinse" and let the system drain, rinse, and refill with fresh liquid. When the rinsing is complete, select "Flow" to set up the liquid circulation. The flow rate may be increased or decreased by clicking on "Up" or "Down" respectively. Particles larger than 500 μm generally require higher flow rates (above 60% of full scale) to prevent settling.

   To acquire a reasonable number of data points, you need to add enough sample to count at least 1000 particles. Monitor the particle loading as follows.

   a. After starting the flow system, go to the Lab tab in the Partan home screen and select "Start analysis".
   b. The program will open a camera view window and suspended particles will be visible. A live particle counter will be active in the lower right of the window.
   c. Add sample until a reasonable number of particles are visible in the flow and the desired particle count rate is achieved.
   d. Once the correct loading has been achieved, click on "Stop Analysis". You will prompted to save the data, but since this was just a loading run, click "Cancel" in the Save dialog.

5. **Running the sample.** While keeping the flow system running, click on "New Analysis". You will be asked to open an SOP file. Select the one you defined under step 3 above. Then in the "Lab" tab, click on "Start Analysis". The measurement will now proceed for the time period you set in your SOP. Measurement progress can be monitored by clicking on the "Image" and "X-Y Graph" tabs.

   At the end of the run, you will prompted to save your data with a filename based on the name you gave your sample. The default file storage location is on the C:\ drive under the "SI Storage" folder. You can change both the file name and the location if you choose.

   After you have run a sample, you need to drain, rinse and refill the circulation system to remove the old material.

   a. ON the Flex home screen, click on the Drain icon. Fluid will drain out of the circulator 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.

6. **Analyzing your data.** After the data run is complete, the Partan SI software offers several ways to examine your data.

a. X-Y Graph. This plots the curves you selected when setting up the SOP. The differential volume distribution ("Vol%") and number distribution ("Count%") are most often used. The selections under "Mode", "Distribution", and "General settings" can be used to customize the data graph.
b. Tabular data. A summary of the particle distribution measurements can be found under the "Spreadsheet" tab.
c. Particle images. By clicking "View Particles" in the upper right menu, you can get a photo record of each particle recorded and measured during your run. The images can be sorted by the value of any shape or size parameter available within the Partan software by clicking on "Sort Value" and using the dropdown menu. The parameter values can be attached to each particle image by selecting "Show Value".

7. **Exporting and Reporting Data.** There are several ways to export your data from the Partan software.
    a. Under "File", selecting "Save Raw Data" will save a file in Excel format that includes all particle count information. This is the most comprehensive way to export data for further processing or graphing with a scientific plotting program.
    b. To print a pre-formatted report that includes summary data and the size distribution graph, select either "Print" (outputs to the local printer) or "Export to PDF" (creates a PDF file with the same format).
    c. The images of individual particles can be printed by selecting "View Particles", then right clicking on the particle images. You will have a choice of printing a report that includes the images of all particles, saving the images as a PDF file, or saving the image of each particle as a BMP file.

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.
5. Update the Bluewave log sheet any issues you may have encountered during your run.